

FIG. 24

BEST AVAILABLE COPY



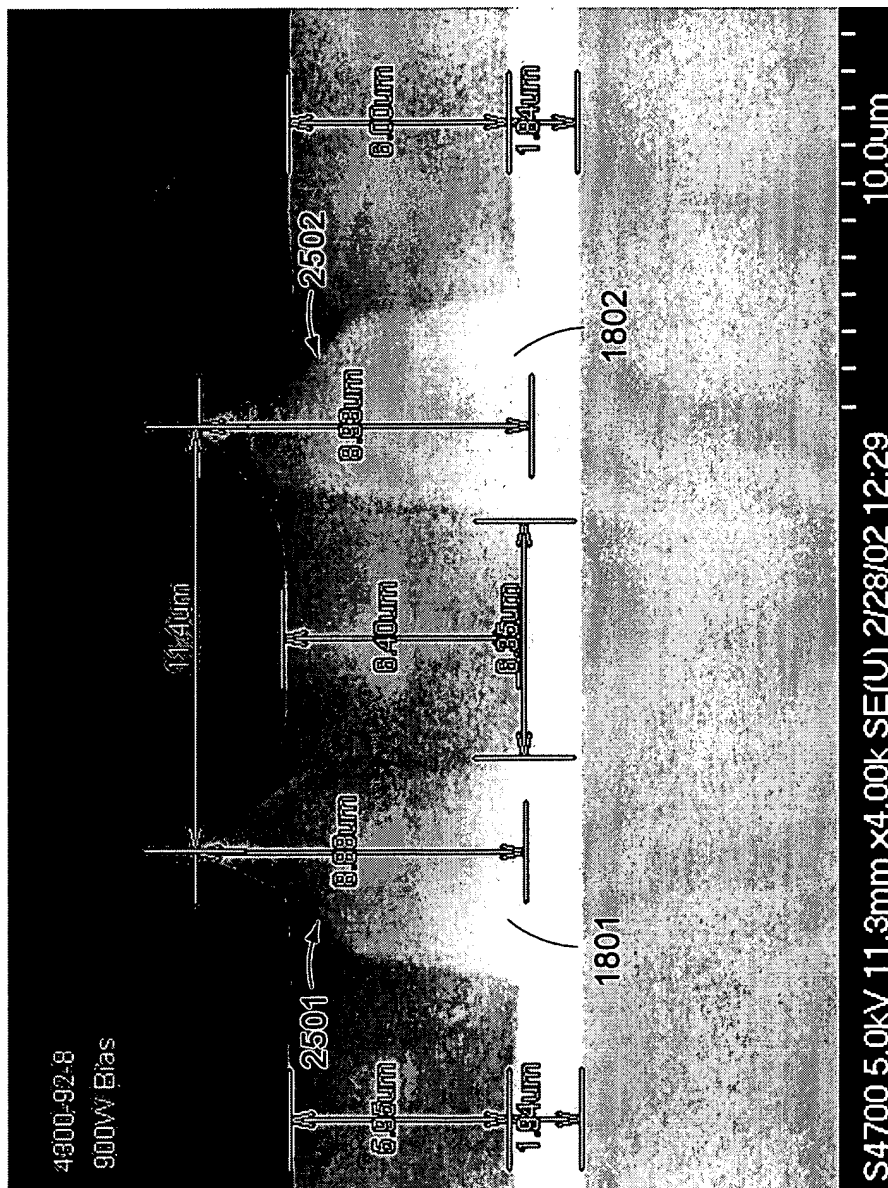


FIG. 25



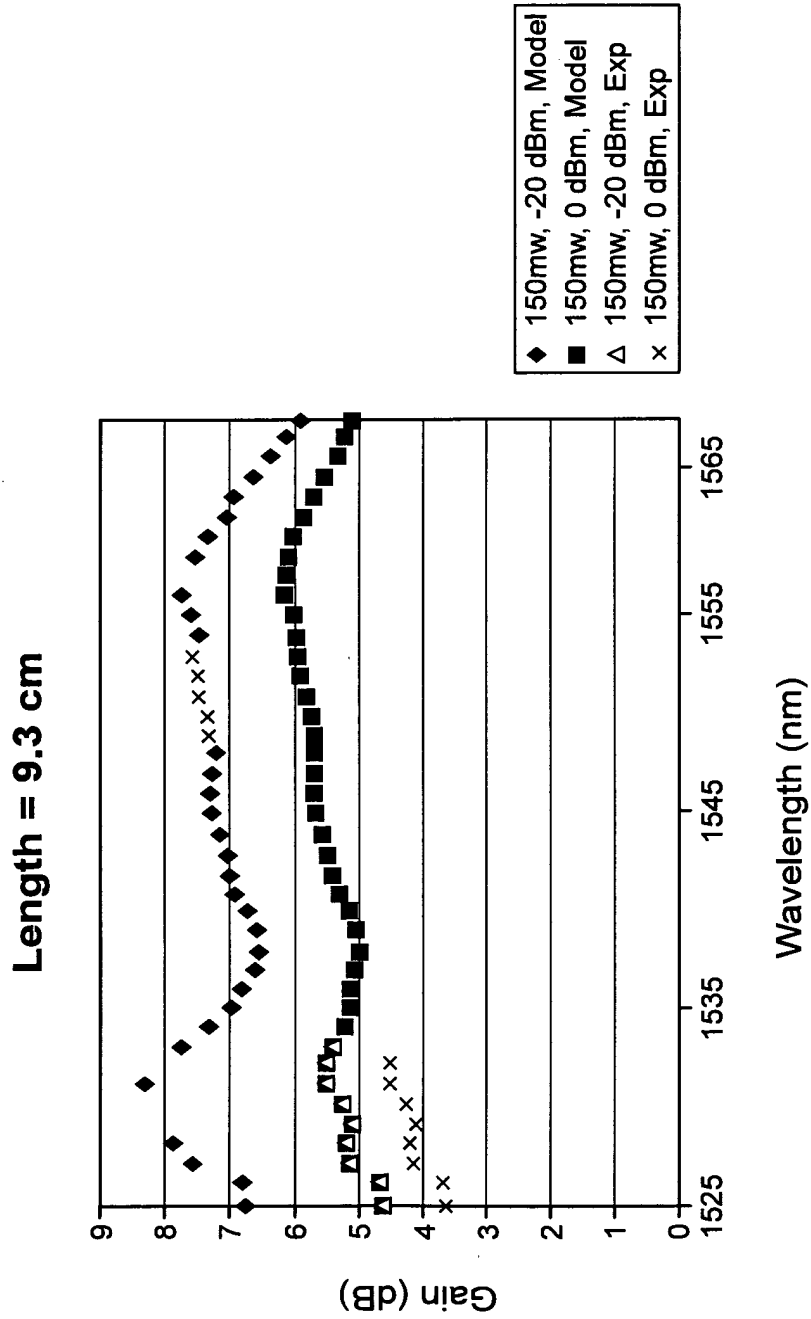


FIG. 26



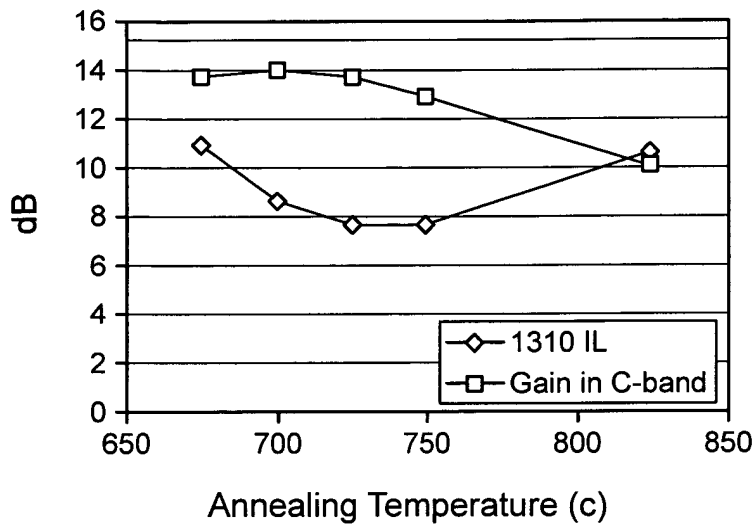


FIG. 27

Life Time and Up-conversion vs. Annealing Temperature

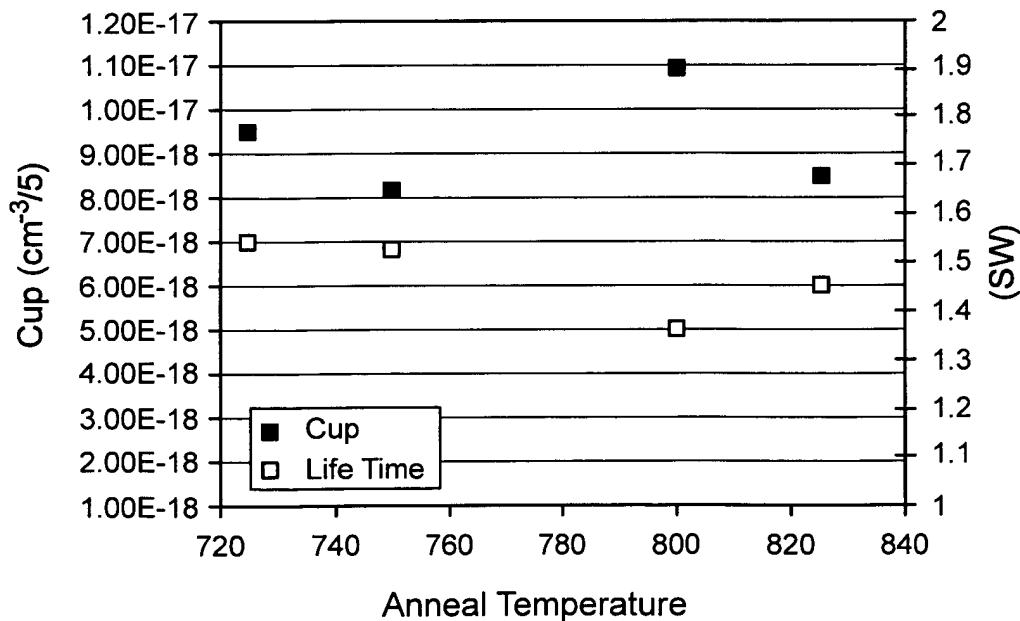


FIG. 28



Index and Thickness

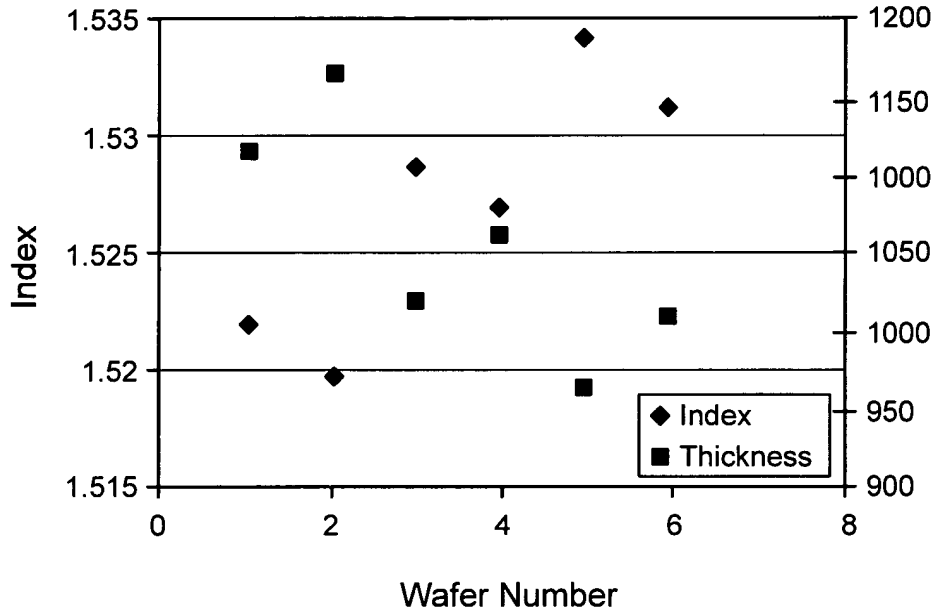


FIG. 29

532nm PL/um

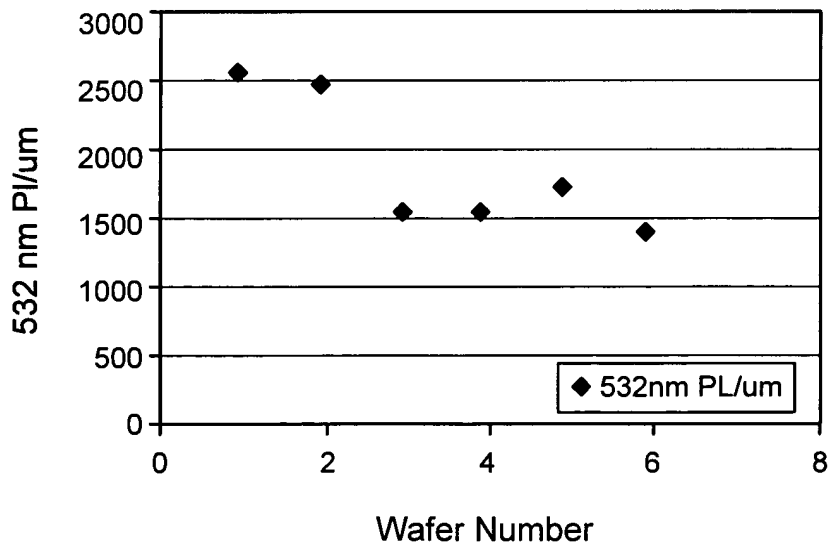


FIG. 30



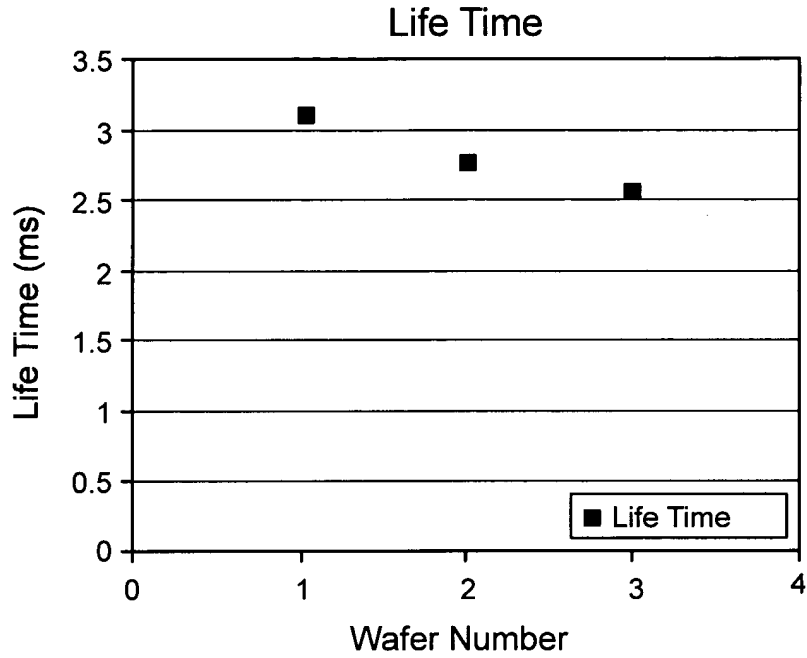


FIG. 31

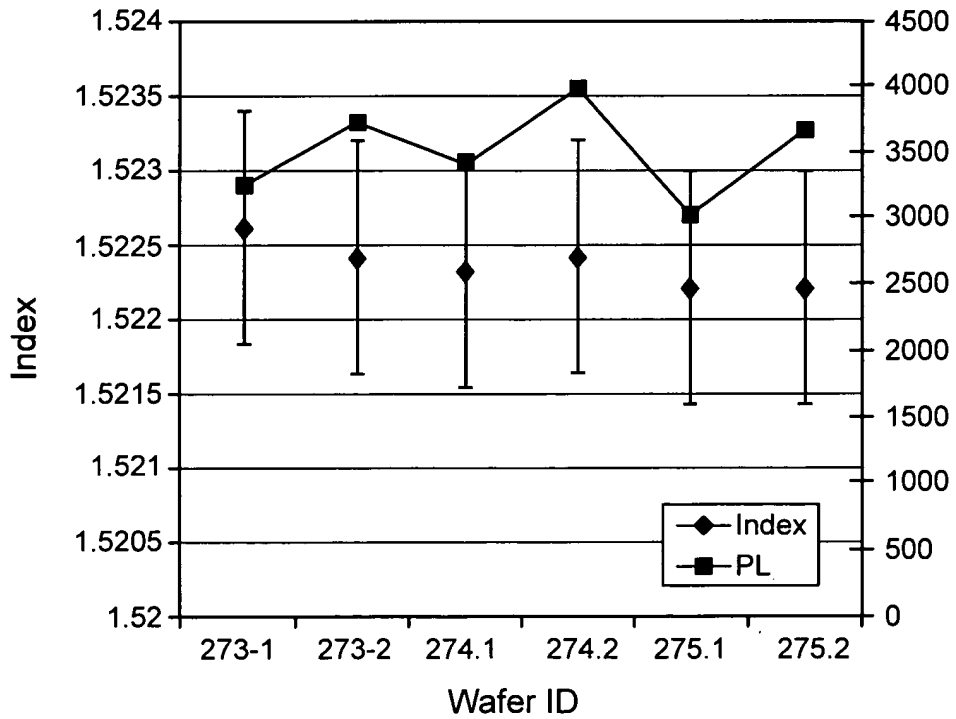


FIG. 32



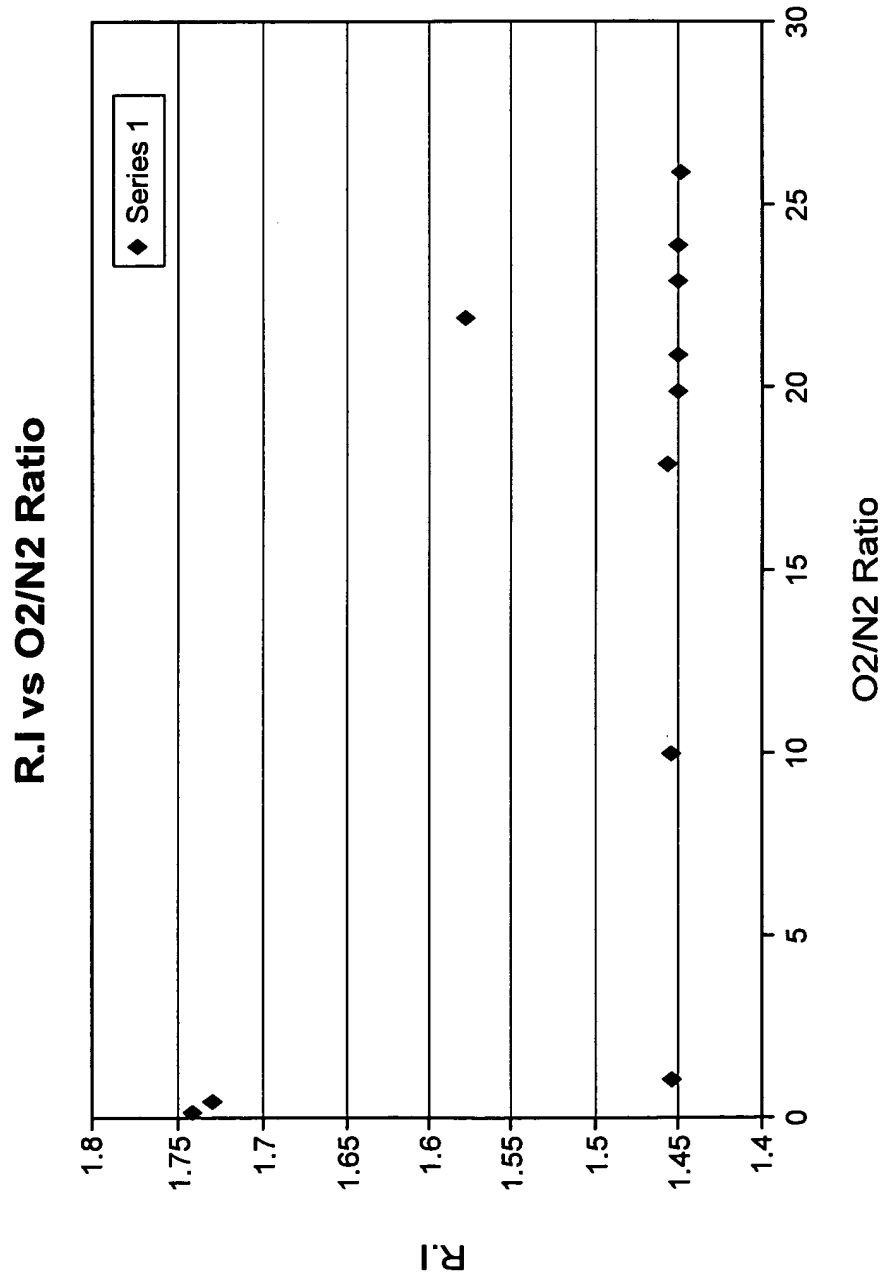


FIG. 33





PATENT
Customer No. 22,852
Attorney Docket No. 9140.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
)	
ZHANG, Hongmei et al.)	Group Art Unit: 2823
)	
Application No.: 10/954,182)	Examiner: ESTRADA, Michelle
)	
Filed: October 1, 2004)	
)	Confirmation No.: 9873
For: BIASED PULSE DC REACTIVE)	
SPUTTERING OF OXIDE FILMS)	

MAIL STOP AMENDMENT
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

THIRD SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT
UNDER 37 C.F.R. § 1.97(c)

Pursuant to 37 C.F.R. §§ 1.56 and 1.97(c), Applicants bring to the attention of the Examiner the documents on the attached listing. This Information Disclosure Statement is being filed after the events recited in Section 1.97(b) but, to the undersigned's knowledge, before the mailing date of either a Final action, Quayle action, or a Notice of Allowance. Under the provisions of 37 C.F.R. § 1.97(c), the Commissioner is hereby authorized to charge the fee of \$180.00 to Deposit Account No. 06-0916 as specified by Section 1.17(p).

Copies of the listed non-patent literature documents are attached. Copies of the U.S. patents and patent publications are not enclosed.

Applicants respectfully request that the Examiner consider the listed documents and indicate that they were considered by making appropriate notations on the attached form.

12/28/2005 SSITHIB1 00000001 060916 10954182
01 FC:1806 180.00 DA

This submission does not represent that a search has been made or that no better art exists and does not constitute an admission that each or all of the listed documents are material or constitute "prior art." If the Examiner applies any of the documents as prior art against any claims in the application and Applicants determine that the cited documents do not constitute "prior art" under United States law, Applicants reserve the right to present to the office the relevant facts and law regarding the appropriate status of such documents.

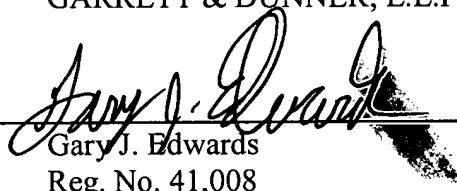
Applicants further reserve the right to take appropriate action to establish the patentability of the disclosed invention over the listed documents, should one or more of the documents be applied against the claims of the present application.

If there is any fee due in connection with the filing of this Statement, please charge the fee to our Deposit Account No. 06-0916.

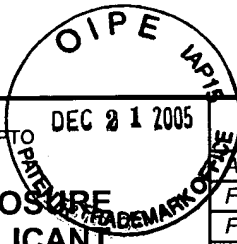
Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Dated: December 21, 2005

By: 
Gary J. Edwards
Reg. No. 41,008

**Express Mail Label No.
EV 758329240 US**



IDS Form PTO/SB/08: Substitute for form 1449A/PTO

Complete if Known

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet	1	of	1	Application Number	10/954,182
				Filing Date	October 1, 2004
				First Named Inventor	Hongmei ZHANG
				Art Unit	2823
				Examiner Name	ESTRADA, Michelle
				Attorney Docket Number	9140.0016-01

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS

Examiner Initials ⁷	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
		US 2002/0076133 A1	06-20-2002	Li et al.	
		US 5,478,456	12-26-1995	Humpal et al.	
		US 6,846,765 B2	01-25-2005	Imamura et al.	

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

FOREIGN PATENT DOCUMENTS

Examiner Initials ⁷	Cite No. ¹	Foreign Patent Document	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁶
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)				

NON PATENT LITERATURE DOCUMENTS

Examiner Initials ⁷	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
		DOREY, R.A., "Low temperature micromoulding of functional ceramic devices," Grant summary for GR/S84156/01 for the UK Engineering and Physical Sciences Research Council, 2 pages (2004).	
		HOWSON, R.P., "The reactive sputtering of oxides and nitrides," <i>Pure & Appl. Chem.</i> 66(6):1311-1318 (1994).	
		Office Action issued September 21, 2005 in U.S. Application No. 11/100,856 (Attorney Docket No. 09140.0015-01).	
		Office Action issued on August 4, 2005, in U.S. Application No. 10/101,863 (Attorney Docket No. 09140.0016-00).	
		Office Action issued on August 8, 2005 in U.S. Application No. 10/101,341 (Attorney Docket No. 09140-0017-00).	
		Office Action issued on October 3, 2005 in U.S. Application No. 10/650,461 (Attorney Docket No. 09140-0025-00).	
		Office Action issued on March 24, 2005 in U.S. Application No. 10/851,542 (Attorney Docket No. 09140-0033-00).	
		Response to Office Action filed July 25, 2005 in U.S. Application No. 10/851,542 (Attorney Docket No. 09140-0033-00).	
		Office Action issued on October 19, 2005 in U.S. Application No. 10/851,542 (Attorney Docket No. 09140.0033-00).	

Examiner Signature		Date Considered	
--------------------	--	-----------------	--

EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

ZHANG, Hongmei et al.

Application No.: 10/954,182

Filed: October 1, 2004

For: BIASED PULSE DC REACTIVE
SPUTTERING OF OXIDE FILMS



Group Art Unit: 2823

Examiner: ESTRADA, Michelle

Confirmation No.: 9873

MAIL STOP AMENDMENT

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

SUBMISSION OF REPLACEMENT DRAWINGS

Subject to the approval of the Examiner, please replace the drawings in the above-identified application with the twenty-seven (27) sheets of drawings filed herewith (Figures 1A-1B, 2-15, 16A-16D, and 17-33). If the replacement drawings for any reason are not in full compliance with the pertinent statutes and regulations, please so advise the undersigned.

If any fees are necessary for the submission of these formal drawings, please charge our Deposit Account No. 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Date: December 21, 2005

By: 

Gary J. Edwards
Reg. No. 41,008

**EXPRESS MAIL LABEL NO.
EV 758329240 US**

PATENT APPLICATION FEE DETERMINATION RECORD
Effective December 8, 2004

10/954182

CLAIMS AS FILED - PART I

	(Column 1)	(Column 2)
TOTAL CLAIMS		
FOR	NUMBER FILED	NUMBER EXTRA
TOTAL CHARGEABLE CLAIMS	minus 20= *	
INDEPENDENT CLAIMS	minus 3 = *	
MULTIPLE DEPENDENT CLAIM PRESENT <input type="checkbox"/>		

* If the difference in column 1 is less than zero, enter "0" in column 2

CLAIMS AS AMENDED - PART II

	(Column 1)	(Column 2)	(Column 3)
AMENDMENT A	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
	12-21-05		
Total	* 31	Minus ** 61	= /
Independent	* 4	Minus *** 9	= /
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

	(Column 1)	(Column 2)	(Column 3)
AMENDMENT B	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
Total	*	Minus **	=
Independent	*	Minus ***	=
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

	(Column 1)	(Column 2)	(Column 3)
AMENDMENT C	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
Total	*	Minus **	=
Independent	*	Minus ***	=
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

SMALL ENTITY TYPE

OR OTHER THAN SMALL ENTITY

RATE	FEE	OR	RATE	FEE
BASIC FEE	150.00	OR	BASIC FEE	300.00
X\$ 25=		OR	X\$50=	
X100=		OR	X200=	
+180=		OR	+360=	
TOTAL		OR	TOTAL	

SMALL ENTITY OR OTHER THAN SMALL ENTITY

RATE	ADDITIONAL FEE	OR	RATE	ADDITIONAL FEE
X\$ 25=	/	OR	X\$50=	/
X100=	/	OR	X200=	/
+180=	/	OR	+360=	/
TOTAL ADDIT. FEE		OR	TOTAL ADDIT. FEE	

RATE	ADDITIONAL FEE	OR	RATE	ADDITIONAL FEE
X\$ 25=		OR	X\$50=	
X100=		OR	X200=	
+180=		OR	+360=	
TOTAL ADDIT. FEE		OR	TOTAL ADDIT. FEE	

RATE	ADDITIONAL FEE	OR	RATE	ADDITIONAL FEE
X\$ 25=		OR	X\$50=	
X100=		OR	X200=	
+180=		OR	+360=	

CLAIMS ONLY

Application Number 109,541,82 Filing Date

Applicant(s)

* May be used for additional claims or amendments

CLAIMS	AS FILED		AFTER FIRST AMENDMENT		AFTER SECOND AMENDMENT	
	Indep	Depend	Indep	Depend	Indep	Depend
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Total Indep						
Total Depend						
Total Claims						

03-06-06

IFW



PATENT
Customer No. 22,852
Attorney Docket No. 9140.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
)	
ZHANG, Hongmei et al.)	Group Art Unit: 2823
)	
Application No.: 10/954,182)	Examiner: ESTRADA, Michelle
)	
Filed: October 1, 2004)	
)	Confirmation No.: 9873
For: BIASED PULSE DC REACTIVE)	
SPUTTERING OF OXIDE FILMS)	

MAIL STOP AMENDMENT
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

FOURTH SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT
UNDER 37 C.F.R. § 1.97(c)

Pursuant to 37 C.F.R. §§ 1.56 and 1.97(c), Applicants bring to the attention of the Examiner the documents on the attached listing. This Information Disclosure Statement is being filed after the events recited in Section 1.97(b) but, to the undersigned's knowledge, before the mailing date of either a Final action, Quayle action, or a Notice of Allowance. Under the provisions of 37 C.F.R. § 1.97(c), the Commissioner is hereby authorized to charge the fee of \$180.00 to Deposit Account No. 06-0916 as specified by Section 1.17(p).

Copies of the listed non-patent literature documents are attached. Copies of the U.S. patents and patent publications are not enclosed.

Applicants respectfully request that the Examiner consider the listed documents and indicate that they were considered by making appropriate notations on the attached form.

This submission does not represent that a search has been made or that no better art exists and does not constitute an admission that each or all of the listed documents are material or constitute "prior art." If the Examiner applies any of the documents as prior art against any claims in the application and Applicants determine that the cited documents do not constitute "prior art" under United States law, Applicants reserve the right to present to the office the relevant facts and law regarding the appropriate status of such documents.

Applicants further reserve the right to take appropriate action to establish the patentability of the disclosed invention over the listed documents, should one or more of the documents be applied against the claims of the present application.

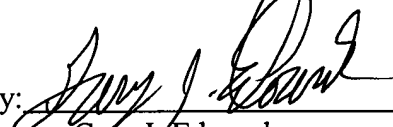
If there is any fee due in connection with the filing of this Statement, please charge the fee to our Deposit Account No. 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

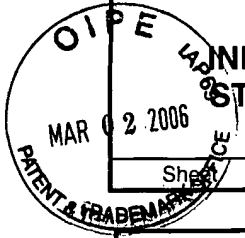
Dated: March 2, 2006

By:



Gary L. Edwards
Reg. No. 41,008

**Express Mail Label No.
EV 860819695 US**



IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>			Application Number	10/954,182
			Filing Date	October 1, 2004
			First Named Inventor	Hongmei ZHANG
			Art Unit	2823
			Examiner Name	ESTRADA, Michelle
			Attorney Docket Number	9140.0016-01
Sheet	1	of	3	

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner Initials*	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
		US 3,309,302	03-14-1967	Heil	
		US 5,338,625	08-16-1994	Bates et al.	
		US 5,561,004	10-01-1996	Bates et al.	
		US 5,909,346	06-01-1999	Malhotra et al.	
		US 5,930,046	07-27-1999	Solberg et al.	
		US 6,000,603	12-14-1999	Koskenmaki et al.	
		US 6,133,670	10-17-2000	Rodgers et al.	
		US 6,242,129 B1	06-05-2001	Johnson	
		US 6,280,875 B1	08-28-2001	Kwak et al.	
		US 6,290,821 B1	09-18-2001	McLeod	
		US 6,356,694 B1	03-12-2002	Weber	
		US 6,376,027 B1	04-23-2002	Lee et al.	
		US 6,632,563 B1	10-14-2003	Krasnov et al.	
		US 6,768,855 B1	07-27-2004	Bakke et al.	
		US 6,683,244 B2	01-27-2004	Fujimori et al.	
		US 6,683,749 B2	01-27-2004	Daby et al.	
		US 2002/0001746 A1	01-03-2002	Jenson	
		US 2002/0076133 A1	06-20-2002	Li et al.	
		US 2002/0140103 A1	10-03-2002	Kloster et al.	

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

FOREIGN PATENT DOCUMENTS						
Examiner Initials*	Cite No. ¹	Foreign Patent Document	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁶
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)				
		WO 2004/106581 A2	12-09-2004	Symmorphix, Inc.		
		WO 2004/106582 A2	12-09-2004	Symmorphix, Inc.		

NON PATENT LITERATURE DOCUMENTS					
Examiner Initials*	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or			Translation ⁶

Examiner Signature		Date Considered	
--------------------	--	-----------------	--

EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

IDS Form PTO/SB/08: Substitute for form 1449A/PTO INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Use as many sheets as necessary)				Complete if Known	
				<i>Application Number</i>	10/954,182
				<i>Filing Date</i>	October 1, 2004
				<i>First Named Inventor</i>	Hongmei ZHANG
				<i>Art Unit</i>	2823
				<i>Examiner Name</i>	ESTRADA, Michelle
Sheet	2	of	3	<i>Attorney Docket Number</i>	9140.0016-01

NON PATENT LITERATURE DOCUMENTS		
		country where published.
	KIM, H-K. and YOON, Y., "Characteristics of rapid-thermal-annealed LiCoO ₂ cathode film for an all-solid-state thin film microbattery," <i>J. Vac. Sci. Technol. A</i> 22(4):1182-1187 (2004).	
	Response to Office Action filed on October 17, 2005 in U.S. Application No. 10/291,179 (Attorney Docket No. 09140-0001-00).	
	Final Office Action issued on December 14, 2005 in U.S. Application No. 10/291,179 (Attorney Docket No. 09140-0001-00).	
	PCT Invitation to Pay Additional Fees for PCT/US01/22750, dated March 13, 2002 (Attorney Docket No. 09140.0002-00304).	
	PCT International Search Report for PCT/US01/22750, dated July 19, 2002 (Attorney Docket No. 09140.0002-00304).	
	PCT Written Opinion for PCT/US01/22750, dated July 23, 2002 (Attorney Docket No. 09140.0002-00304).	
	PCT International Preliminary Examination Report for PCT/US01/22750, dated October 8, 2002 (Attorney Docket No. 09140.0002-00304).	
	Amendment/RCE filed on March 10, 2005 in U.S. Application No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
	Office Action issued on November 28, 2005 in U.S. Application No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
	Response to Office Action filed February 17, 2006 in U.S. Application No. 11/100,856 (Attorney Docket No. 09140.0015-01).	
	Response to Office Action filed December 5, 2005, in U.S. Application No. 10/101,863 (Attorney Docket No. 09140.0016-00).	
	Final Office Action issued on February 14, 2006, in U.S. Application No. 10/101,863 (Attorney Docket No. 09140.0016-00).	
	Response to Office Action filed February 24, 2006, in U.S. Application No. 10/101,863 (Attorney Docket No. 09140.0016-00).	
	Response to Office Action filed on November 8, 2005, in U.S. Application No. 10/101,341 (Attorney Docket No. 09140-0017-00).	
	Office Action issued on February 13, 2006, in U.S. Application No. 10/101,341 (Attorney Docket No. 09140-0017-00).	
	Response to Office Action filed on January 3, 2006 in U.S. Application No. 10/650,461 (Attorney Docket No. 09140-0025-00).	
	International Preliminary Examination Report mailed on April 15, 2004 in PCT/US03/24809 (Attorney Docket No. 09140-0025-00304).	
	Office Action issued on December 2, 2005 in U.S. Application No. 10/789,953 (Attorney Docket No. 09140.0030-00).	
	Specification and Preliminary Amendment as filed for U.S. Appl. No. 11/297,057 (Attorney Docket No. 09140.0030-01).	
	Response to Office Action filed January 19, 2006 in U.S. Application No. 10/851,542 (Attorney Docket No. 09140.0033-00).	

Examiner Signature		Date Considered	
--------------------	--	-----------------	--

EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

IDS Form PTO/SB/08: Substitute for form 1449A/PTO INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Use as many sheets as necessary)				Complete if Known	
				<i>Application Number</i>	10/954,182
				<i>Filing Date</i>	October 1, 2004
				<i>First Named Inventor</i>	Hongmei ZHANG
				<i>Art Unit</i>	2823
				<i>Examiner Name</i>	ESTRADA, Michelle
Sheet	3	of	3	<i>Attorney Docket Number</i>	9140.0016-01

NON PATENT LITERATURE DOCUMENTS			
		PCT International Search Report and Written Opinion for Application No. PCT/US2004/014524 dated March 2, 2005 (Attorney Docket No. 09140.0033-00304).	
		PCT International Preliminary Report on Patentability for Application No. PCT/US2004/014524, dated December 8, 2005 (Attorney Docket No. 09140.0033-00304).	
		PCT International Search Report for Application No. PCT/US2004/014523 dated January 17, 2005 (Attorney Docket No. 09140.0034-00304).	
		PCT Written Opinion for Application No. PCT/US2004/014523 dated January 17, 2005 (Attorney Docket No. 09140.0034-00304).	
		PCT International Preliminary Report on Patentability for Application No. PCT/US2004/014523, dated December 8, 2005 (Attorney Docket No. 09140.0034-00304).	
		Specification as filed for U.S. Appl. No. 11/297,057 (Attorney Docket No. 09140.0042-00).	

Examiner Signature		Date Considered	
--------------------	--	-----------------	--

EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property
Organization
International Bureau



(43) International Publication Date
9 December 2004 (09.12.2004)

PCT

(10) International Publication Number
WO 2004/106581 A2

(51) International Patent Classification: C23C 14/08,
14/34

(21) International Application Number:
PCT/US2004/014523

(22) International Filing Date: 21 May 2004 (21.05.2004)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
60/473,379 23 May 2003 (23.05.2003) US

(71) Applicant (for all designated States except US): SYM-
MORPHIX, INC. [US/US]; 1278 Reamwood Avenue,
Sunnyvale, CA 94089-2233 (US).

(72) Inventors; and

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(81) Designated States (unless otherwise indicated, for every
kind of national protection available): AE, AG, AL, AM,
AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN,
CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI,
GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE,
KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD,
MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG,
PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM,
TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM,
ZW.

(84) Designated States (unless otherwise indicated, for every
kind of regional protection available): ARIPO (BW, GH,
GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM,
ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM),
European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI,
FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI,
SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ,
GW, ML, MR, NE, SN, TD, TG).

Published:

— without international search report and to be republished
upon receipt of that report

For two-letter codes and other abbreviations, refer to the "Guid-
ance Notes on Codes and Abbreviations" appearing at the begin-
ning of each regular issue of the PCT Gazette.



WO 2004/106581 A2

(54) Title: TRANSPARENT CONDUCTIVE OXIDES

(57) Abstract: A method of deposition of a transparent conductive film from a metallic target is presented. A method of forming a transparent conductive oxide film according to embodiments of the present invention include depositing the transparent conductive oxide film in a pulsed DC reactive ion process with substrate bias, and controlling at least one process parameter to affect at least one characteristic of the conductive oxide film. The resulting transparent oxide film, which in some embodiments can be an indium-tin oxide film, can exhibit a wide range of material properties depending on variations in process parameters. For example, varying the process parameters can result in a film with a wide range of resistive properties and surface smoothness of the film.

Transparent Conductive Oxides

Related Applications

[0001] The present application claims priority to U.S. Provisional Application 60/473,379, "Transparent Conductive Oxides from a Metallic Target," by R. Ernest Demaray and Mukundan Narasimhan, filed on May 23, 2003, herein incorporated by reference in its entirety.

Background

1. Field of the Invention

[0002] The present invention is related to deposition of oxides on a substrate and, in particular, deposition of transparent conductive oxides.

2. Discussion of Related Art

[0003] Transparent conductive oxides have a wide variety of uses, including applications to solar cells, organic light emitting diodes (OLEDs), electric field devices, current devices (i.e. touch screens), energy efficient windows, conductive anti-reflective devices, electromagnetic interference shields, heaters, transparent electrodes, coatings for cathode ray tube (CRT) displays, to name only a few. Another important application is for touch sensitive MEMS devices, such as those used, for example, in fingerprint sensors and such. In many cases, the electrical properties of the conducting film is of great importance.

[0004] Specifically, for OLED applications, films deposited with current technologies are generally rough, resulting in stress risers and field concentration issues, that can cause leakage. Further, asperities in the resulting film can induce lifetime dependent defects in nearest neighbor films that can shorten device lifetimes. Additionally, the brightness of the emergent light from the OLED can be reduced.

[0005] Transparent conductive oxides have been deposited from ceramic targets by RF magnetron sputtering. However, the surface of properties of the resulting films often include nodules or asperities which can cause arcing, defects, surface roughness, and other deleterious effects in the resulting film. Additionally, ceramic targets tend to be more expensive to produce than metallic targets.

[0006] Previous attempts at deposition of transparent conductive oxides, for example indium tin oxide (ITO), with metallic targets have presented numerous

problems, including small process windows, problems in process controllability, a disappearing anode effect, and particle deposition on the film. Such attempts have been abandoned. Deposition with ceramic targets has also been difficult, including problems with particles, nodule formation, and arching during deposition. In both cases, film smoothness has presented major difficulties. Additionally, control of film parameters such as, for example, resistivity and transparency has been difficult.

[0007] Therefore, there is need for cost effective deposition of smoother layers of transparent conductive oxides with greater control over layer properties such as resistivity and transparency.

Summary

[0008] In accordance with the present invention, a method of depositing of a transparent conductive film from a metallic target is presented. A method of forming a transparent conductive oxide film according to embodiments of the present invention includes depositing the transparent conductive oxide film in a pulsed DC reactive ion process with substrate bias, and controlling at least one process parameter to provide at least one characteristic of the conductive oxide film at a particular value.

[0009] A method of depositing a transparent conductive oxide film on a substrate according to some embodiments of the invention, then, includes placing the substrate in a reaction chamber, adjusting power to a pulsed DC power supply coupled to a target in the reaction chamber, adjusting an RF bias power coupled to the substrate, adjusting gas flow into the reaction chamber, and providing a magnetic field at the target in order to direct deposition of the transparent conductive oxide film on the substrate in a pulsed-dc biased reactive-ion deposition process, wherein the transparent conductive oxide film has a particular characteristic.

[0010] The resulting transparent oxide film, which can be deposited according to some embodiments of the present invention, can be an indium-tin oxide (ITO) film. An ITO film can have a wide range of material properties depending on variations in process parameters. For example, varying the process parameters according to some embodiments of the present invention can result in a wide range of resistive properties and surface smoothness of the film.

[0011] These and other embodiments of the invention are further discussed below

with reference to the following figures.

Short Description of the Figures

- [0012] Figures 1A and 1B illustrate a pulsed-DC biased reactive ion deposition apparatus that can be utilized in the methods of depositing according to the present invention.
- [0013] Figure 2 shows an example of a target that can be utilized in the reactor illustrated in Figures 1A and 1B
- [0014] Figure 3A shows an Atomic Force Microscopy (AFM) image of an indium-tin-oxide (ITO) process according to some embodiments of the present invention.
- [0015] Figure 3B shows an Atomic Force Microscopy (AFM) image of another ITO process deposited using a process according to some embodiments of the present invention.
- [0016] Figure 4 shows the variation of bulk resistivity of an ITO layer according to some embodiments of the present invention as a function of the oxygen flow for two different target powers before and after a 250 °C anneal in vacuum.
- [0017] Figure 5 shows the variation of the sheet resistance of an ITO layer according to some embodiments of the present invention as a function of the oxygen flow used for two different target powers before and after a 250 °C anneal in vacuum.
- [0018] Figure 6 shows the target current and voltage (min and max) as a function of oxygen flow.
- [0019] Figure 7 shows the thickness change in layers of ITO according to embodiments of the present invention as a function of oxygen flow.
- [0020] Figure 8 illustrates the relationship between oxygen flow and oxygen partial pressure for a metallic target.
- [0021] Figures 9A-9D illustrate the smoothness of transparent conductive oxides deposited with ceramic targets according to the present invention.
- [0022] In the figures, elements having the same designation have the same or similar function.

Detailed Description

[0023] Deposition of materials by pulsed-DC biased reactive ion deposition is described in U.S. Patent Application Serial No. 10/101863, entitled "Biased Pulse DC Reactive Sputtering of Oxide Films," to Hongmei Zhang, et al., filed on March 16, 2002. Preparation of targets is described in U.S. Patent Application Serial No. 10/101,341, entitled "Rare-Earth Pre-Alloyed PVD Targets for Dielectric Planar Applications," to Vassiliki Milonopoulou, et al., filed on March 16, 2002. U.S. Patent Application Serial No. 10/101863 and U.S. Patent Application Serial No. 10/101,341 are each assigned to the same assignee as is the present disclosure and each is incorporated herein in their entirety. Deposition of oxide materials has also been described in U.S. Patent No. 6,506,289, which is also herein incorporated by reference in its entirety. Transparent oxide films are deposited utilizing processes similar to those specifically described in U.S. Patent No. 6,506,289 and U.S. Application Serial No. 10/101863.

[0024] Figure 1A shows a schematic of a reactor apparatus 10 for sputtering material from a target 12 according to the present invention. In some embodiments, apparatus 10 may, for example, be adapted from an AKT-1600 PVD (400 X 500 mm substrate size) system from Applied Komatsu or an AKT-4300 (600 X 720 mm substrate size) system from Applied Komatsu, Santa Clara, CA. The AKT-1600 reactor, for example, has three deposition chambers connected by a vacuum transport chamber. These Komatsu reactors can be modified such that pulsed DC power is supplied to the target and RF power is supplied to the substrate during deposition of a material film.

[0025] Apparatus 10 includes target 12 which is electrically coupled through a filter 15 to a pulsed DC power supply 14. In some embodiments, target 12 is a wide area sputter source target, which provides material to be deposited on a substrate 16. Substrate 16 is positioned parallel to and opposite target 12. Target 12 functions as a cathode when power is applied to it and is equivalently termed a cathode. Application of power to target 12 creates a plasma 53. Substrate 16 is capacitively coupled to an electrode 17 through an insulator 54. Electrode 17 can be coupled to an RF power supply 18. A magnet 20 is scanned across the top of target 12.

[0026] For pulsed reactive dc magnetron sputtering, as performed by apparatus 10,

the polarity of the power supplied to target 12 by power supply 14 oscillates between negative and positive potentials. During the positive period, the insulating layer on the surface of target 12 is discharged and arcing is prevented. To obtain arc free deposition, the pulsing frequency exceeds a critical frequency that can depend on target material, cathode current and reverse time. High quality oxide films can be made using reactive pulse DC magnetron sputtering as shown in apparatus 10.

[0027] Pulsed DC power supply 14 can be any pulsed DC power supply, for example an AE Pinnacle plus 10K by Advanced Energy, Inc. With this DC power supply, up to 10 kW of pulsed DC power can be supplied at a frequency of between 0 and 350 KHz. The reverse voltage can be 10% of the negative target voltage. Utilization of other power supplies can lead to different power characteristics, frequency characteristics and reverse voltage percentages. The reverse time on this embodiment of power supply 14 can be adjusted between 0 and 5 μ s.

[0028] Filter 15 prevents the bias power from power supply 18 from coupling into pulsed DC power supply 14. In some embodiments, power supply 18 can be a 2 MHz RF power supply, for example a Nova-25 power supply made by ENI, Colorado Springs, Co.

[0029] In some embodiments, filter 15 can be a 2 MHz sinusoidal band rejection filter. In some embodiments, the band width of the filter can be approximately 100 kHz. Filter 15, therefore, prevents the 2 MHz power from the bias to substrate 16 from damaging power supply 18.

[0030] However, both RF and pulsed DC deposited films are not fully dense and may have columnar structures. Columnar structures can be detrimental to thin film applications. By applying a RF bias on wafer 16 during deposition, the deposited film can be densified by energetic ion bombardment and the columnar structure can be substantially eliminated.

[0031] In the AKT-1600 based system, for example, target 12 can have an active size of about 675.70 X 582.48 by 4 mm in order to deposit films on substrate 16 that have dimension about 400 X 500 mm. The temperature of substrate 16 can be held at between -50 °C and 500 °C. The distance between target 12 and substrate 16 can be between about 3 and about 9 cm. Process gas can be inserted into the chamber of

apparatus 10 at a rate up to about 200 sccm while the pressure in the chamber of apparatus 10 can be held at between about .7 and 6 millitorr. Magnet 20 provides a magnetic field of strength between about 400 and about 600 Gauss directed in the plane of target 12 and is moved across target 12 at a rate of less than about 20-30 ' sec/scan. In some embodiments utilizing the AKT 1600 reactor, magnet 20 can be a race-track shaped magnet with dimensions about 150 mm by 600 mm.

[0032] Figure 2 illustrates an example of target 12. A film deposited on a substrate positioned on carrier sheet 17 directly opposed to region 52 of target 12 has good thickness uniformity. Region 52 is the region shown in Figure 1B that is exposed to a uniform plasma condition. In some implementations, carrier 17 can be coextensive with region 52. Region 24 shown in Figure 2 indicates the area below which both physically and chemically uniform deposition can be achieved, for example where physical and chemical uniformity provide refractive index uniformity. Figure 2 indicates region 52 of target 12 that provides thickness uniformity is, in general, larger than region 24 of target 12 providing thickness and chemical uniformity. In optimized processes, however, regions 52 and 24 may be coextensive.

[0033] In some embodiments, magnet 20 extends beyond area 52 in one direction, for example the Y direction in Figure 2, so that scanning is necessary in only one direction, for example the X direction, to provide a time averaged uniform magnetic field. As shown in Figures 1A and 1B, magnet 20 can be scanned over the entire extent of target 12, which is larger than region 52 of uniform sputter erosion. Magnet 20 is moved in a plane parallel to the plane of target 12.

[0034] The combination of a uniform target 12 with a target area 52 larger than the area of substrate 16 can provide films of highly uniform thickness. Further, the material properties of the film deposited can be highly uniform. The conditions of sputtering at the target surface, such as the uniformity of erosion, the average temperature of the plasma at the target surface and the equilibration of the target surface with the gas phase ambient of the process are uniform over a region which is greater than or equal to the region to be coated with a uniform film thickness. In addition, the region of uniform film thickness is greater than or equal to the region of the film which is to have highly uniform optical properties such as index of refraction,

density, transmission or absorptivity.

[0035] Target 12 can be formed of any materials. Typically metallic materials, for example, include combinations of In and Sn. Therefore, in some embodiments, target 12 includes a metallic target material formed from intermetallic compounds of optical elements such as Si, Al, Er and Yb. Additionally, target 12 can be formed, for example, from materials such as La, Yt, Ag, Au, and Eu. To form optically active films on substrate 16, target 12 can include rare-earth ions. In some embodiments of target 12 with rare earth ions, the rare earth ions can be pre-alloyed with the metallic host components to form intermetallics. See U.S. Application Serial No. 10/101,341. Typical ceramic target materials include alumina, silica, alumina silicates, and other such materials.

[0036] In some embodiments of the invention, material tiles are formed. These tiles can be mounted on a backing plate to form a target for apparatus 10. A wide area sputter cathode target can be formed from a close packed array of smaller tiles. Target 12, therefore, may include any number of tiles, for example between 2 to 20 individual tiles. Tiles can be finished to a size so as to provide a margin of non-contact, tile to tile, less than about 0.010" to about 0.020" or less than half a millimeter so as to eliminate plasma processes that may occur between adjacent ones of tiles 30. The distance between tiles of target 12 and the dark space anode or ground shield 19 in Figure 1B can be somewhat larger so as to provide non contact assembly or to provide for thermal expansion tolerance during process chamber conditioning or operation.

[0037] As shown in Figure 1B, a uniform plasma condition can be created in the region between target 12 and substrate 16 in a region overlying substrate 16. A plasma 53 can be created in region 51, which extends under the entire target 12. A central region 52 of target 12 can experience a condition of uniform sputter erosion. As discussed further below, a layer deposited on a substrate placed anywhere below central region 52 can then be uniform in thickness and other properties (i.e., dielectric, optical index, or material concentrations). In addition, region 52 in which deposition provides uniformity of deposited film can be larger than the area in which the deposition provides a film with uniform physical or optical properties such as

chemical composition or index of refraction. In some embodiments, target 12 is substantially planar in order to provide uniformity in the film deposited on substrate 16. In practice, planarity of target 12 can mean that all portions of the target surface in region 52 are within a few millimeters of a planar surface, and can be typically within 0.5 mm of a planar surface.

[0038] Reactive gases that provide a constant supply of ionic oxygen to keep the target surface oxidized can be provided to expand the process window. Some examples of the gases that can be utilized for controlling surface oxidation are CO₂, water vapor, hydrogen, N₂O, fluorine, helium, and cesium. Additionally, a feedback control system can be incorporated to control the oxygen partial pressure in the reactive chamber. Therefore, a wide range of oxygen flow rates can be controlled to keep a steady oxygen partial pressure in the resulting plasma. Other types of control systems such as target voltage control and optical plasma emission control systems can also be utilized to control the surface oxidation of the target. As shown in Figure 1A, power to target 12 can be controlled in a feedback loop at supply 14. Further, oxygen partial pressure controller 20 can control either oxygen or argon partial pressures in plasma 53.

[0039] In some embodiments, transparent conductive oxides can be deposited on various substrates utilizing an indium-tin (In/Sn) metallic target. A series of depositions on glass in accordance with the present invention is illustrated in Table I. The parameters in the process column of Table I are in the format (pulsed DC power/RF bias power/pulsing frequency/reverse time/deposition time/Ar flow (sccms)/O₂ flow (sccms)). An indium-tin (In/Sn: 90%/10% by weight) target using a reactive-pulsed DC (RPDC) process such as that described in U.S. Application Serial No. 10/101,863 was utilized. A power supply with 2MHz RF bias applied to substrate 16 was utilized in the process. Along with the process parameters for each of the separate depositions, each defined by a "Slot" number in the first column, the target voltage, and target current ranges for each of the depositions is also listed.

[0040] Table 2 shows the results obtained by using the process parameters in Table 1. The results include the sheet resistance, thickness, bulk resistivity, and refractive indices of the resulting films. Again, the first column indicates the slot

number of the deposition. The process for each slot number is reiterated in column 2 of Table 2. The sheet resistance of selected ones of the films resulting from the deposition is listed in the third column and the uniformity of the sheet resistance is indicated in the fourth column. The thickness of the film and its uniformity of each of the films deposited by the indicated process is indicated in the fifth and sixth columns. The bulk resistance of selected ones of the films, ρ , is also indicated. Additionally, the refractive index taken at 632 nm is indicated along with the film uniformity of that index. The comments section of Table 2 indicates whether the resulting film is transparent, translucent, or metallic in character.

[0041] Figure 3A shows the Atomic Force Microscopy (AFM) image of an ITO film produced by the process identified in slot #5 in tables 1 and 2. That process, with particularly low oxygen flow rates (24 sccm), produced a rough film with an Ra of about 70 Å and an Rms of about 90 Å. The film also appears to be metallic with this particular oxygen flow and the film roughness is high. Such a film could be applicable to large surface area requirements, for example solar cell applications. While not being limited by any particular theory, it is suspected that the roughness of this film reflects the sub-stoichiometric nature of the film caused by insufficient oxygen flow in the plasma. As can be seen in Figure 3B, where the oxygen flow during deposition has been significantly increased to about 36 sccm, the film is smooth.

[0042] Figure 3B shows an Atomic Force Microscopy (AFM) image of an ITO film deposited using the process described in slot #19 of Tables 1 and 2. In that process, the oxygen flow rate is increased to 36 sccm. The film appears to be transparent and conductive and the surface roughness is ~6Å Ra and Rms of about 13 Å, which is acceptable for OLED requirements. As can be seen from Figures 3A and 3B, variation in oxygen partial pressure (as indicated by increased flow rate) has a large influence on the characteristics of the resulting deposited film.

[0043] The resistivity of the film layer and the smoothness of the film layer can be related. In general, the higher the resistivity of the film layer, the smoother the film layer. Figure 4 shows the variation of bulk resistivity of the ITO as a function of the oxygen flow rate used for two different target powers before and after a 250 °C anneal

in vacuum. The bulk resistivity of the film exhibits a sudden transition downward as the oxygen flow rate is lowered. This transition occurs when the target surface becomes metallic from being poisoned with oxygen. The data utilized to form the graph shown in Figure 4 has been taken from Tables 1 and 2.

[0044] Figure 5 shows the variation of the sheet resistance of an ITO film as function of the O₂ flow used for two different target powers before and after a 250C anneal in vacuum. As shown in Figure 5, the sheet resistance follows similar trends as the bulk resistivity of the film.

[0045] Figure 6 shows the target current and voltage (min and max) as a function of the oxygen flow rate. The target voltage increases as the oxygen flow rate is lowered. It could be seen here that at a 40 sccm oxygen flow rate through repeated depositions, the target voltage is not constant. This illustrates the utility of a target voltage feedback control system that adjusts the power supplied to target 12 to hold the target voltage constant. Therefore, as shown in Figure 1A, PDC power 14 can include feedback loop to control the voltage on target 12.

[0046] Figure 7 shows the thickness change of a resulting film as a function of oxygen flow rate in sccm. The thickness of the film increases as the oxygen flow decreases but this could make opaque metallic films and so choosing the correct oxygen flow and utilizing an oxygen flow feedback control system to control material characteristics such as, for example, transparency or conductivity can be desirable.

[0047] In some embodiments, instead of oxygen flow rate, oxygen partial pressure can be controlled with a feedback system 20 (see Figure 1A). Controlling the oxygen partial pressure can provide better control over the oxygen content of the plasma, and therefore the oxygen content of the resulting films, and allows better control over the film characteristics. Figure 8 illustrates the relationship between the flow rate and partial pressure. As can be seen from Figure 8, in order to reach the saturated region (e.g., when target 12 is completely poisoned with oxygen), no increase in flow rate is required. In some embodiments, reactor 10 can include a partial pressure feedback loop controller 20 that controls the oxygen flow in order to maintain a desired partial pressure of oxygen in the plasma. Such a controller can be the IRESS system, that can be purchased from Advanced Energy, Inc., Ft. Collins, Colorado. It has been

found that film parameters such as resistivity, smoothness, and transparency can be highly dependent on oxygen partial pressures, and therefore these characteristics of the resulting deposited layer can be controlled by adjusting the oxygen partial pressures.

[0048] Some embodiments of the present invention can be deposited with ceramic targets. An example target is an ITO (In/Sn 90/10) ceramic target can be utilized. Table 3 illustrates some example processes for deposition of ITO utilizing a ceramic target according to the present invention. Bulk resistivity, sheet resistance, resistance, thicknesses, deposition rates, and index of refraction of the resulting films are shown along with the process parameters utilized in the deposition. Figure 9A shows an AFM depiction of a transparent conductive oxide film corresponding to run #10 in Table 3. Figure 9B shows an AFM depiction of a transparent conductive oxide film corresponding to run #14 in Table 3. Figure 9C shows an AFM depiction of a transparent conductive oxide film corresponding to run #16 in Table 3. Figure 9D shows an AFM depiction of a transparent conductive oxide film layer corresponding to run #6 in Table 3.

[0049] Figures 9A through 9D illustrate the roughnesses of selective depositions of ITO deposited utilizing the ceramic target. In Figure 9A, the roughest surface shown, the film was deposited using 3kW RF power, 100W bias, 3 sccm O₂ and 60 sccm Ar at a temperature of 280 °C. The layer grew to a thickness of 1200 Å in 100 seconds of deposition time and exhibited a sheet resistance of 51 ohms/sq. The roughness illustrated in Figure 9A is characterized by an Ra=2.3 nm and R_{MS} of 21 nm.

[0050] The ITO film shown in Figure 9B was deposited using 3 kW RF power, 300 W bias, 3 sccm O₂ and 60 sccm Ar at a temperature of 280 °C. The layer illustrated in Figure 9B grew to a thickness of 1199 Å in 100 sec. The layer in Figure 9B exhibited a sheet resistance of 39 ohms/sq. The roughness illustrated in Figure 9B is characterized by an Ra=1.1 nm and R_{max} of 13 nm.

[0051] The ITO film shown in Figure 9C was deposited using 3 kW RF power, 300 W bias, 3 sccm O₂, 30 sccm Ar at a temperature of 280 °C. The layer grew to a thickness of 1227 Å in 100 seconds of deposition time and exhibited a sheet resistance

of 57 ohms/sq. The roughness illustrated in Figure 9C can be characterized by an $R_a=0.88$ nm and a R_{max} of 19.8 nm.

[0052] Figure 9D was deposited using 1.5 kW RF power, 300 W bias, 0 sccm O_2 , 30 sccm Ar at a temperature of 280 C. The layer grew to a thickness of 580 Å in 100 seconds of deposition time and exhibited a sheet resistance of 106 ohms/sq. The roughness illustrated in Figure 9C can be characterized by an $R_a=0.45$ nm and an R_{max} of 4.6 nm.

[0053] Utilizing the example depositions described herein, the roughness and resistivity of a transparent oxide film can be tuned to particular applications. In general, particularly high resistivities can be obtained, which are useful for touch sensitive devices. As shown in Table 3, the sheet resistance ranged from about 39 Ω /sq for trial # 14 to a high of 12,284 Ω /sq for trial #1. Careful variation of the process parameters, therefore, allow control of sheet resistance over an extremely broad range. Low resistivities can be obtained by adjusting the process parameters for uses in devices such as OLEDs and MEMS display devices. As is illustrated in Table 3, the bulk resistivity can be controlled to be between about $2E-4$ micro-ohms-cm to about 0.1 micro-ohms-cm. Additionally, other parameters such as refractive index and transparency of the film can be controlled.

[0054] Further, deposition of transparent conductive oxide layers, for example ITO, can be doped with rare-earth ions, for example erbium or cerium, can be utilized to form color-conversion layers and light-emission sources. In some embodiments, a rare-earth doped target can be made in a single piece to insure uniformity of doping. Co-doping can be accomplished in the target.

[0055] Similar processes for other metallic conductive oxides can also be developed. For example, deposition of zinc oxide films. Further, as can be seen in the examples shown in Table 3, low temperature depositions can be performed. For example, transparent conductive oxides according to the present invention can be deposited at temperatures as low as about 100 °C. Such low temperature depositions can be important for depositions on temperature sensitive materials such as plastics.

[0056] Other thin film layers according to the present invention include deposition of other metal oxides to form conducting and semi-conducting films. Thin films

formed according to the present invention can be utilized in many devices, including, but not limited to, displays, photovoltaics, photosensors, touchscreens, and EMI shielding.

[0057] Embodiments of the invention disclosed here are examples only and are not intended to be limiting. Further, one skilled in the art will recognize variations in the embodiments of the invention described herein which are intended to be included within the scope and spirit of the present disclosure. As such, the invention is limited only by the following claims.

Table I

Slot #	Process	Target Voltage (V)		Target Current (Amps)	
		Min	Max	Mix	Max
14	1.5kw/100w/200khz/2.2µs/300s/20Ar/80O ₂	244	252	5.94	6.14
15	1.5kw/100w/200khz/2.2µs/300s/20Ar/40O ₂	254	263	5.7	5.9
17	1.5kw/100w/200khz/2.2µs/300s/20Ar/40O ₂	252	260	5.76	5.96
19	1.5kw/100w/200khz/2.2µs/300s/20Ar/36O ₂	254	263	5.72	5.92
21	1.5kw/100w/200khz/2.2µs/300s/20Ar/30O ₂	255	268	5.76	5.9
1	1kw/100w/200khz/2.2µs/300s/20Ar/ 80O ₂	224	233	4.32	4.5
2	1kw/100w/200khz/2.2µs/300s/20Ar/ 36O ₂	231	243	4.12	4.3
3	1kw/100w/200khz/2.2µs/300s/20Ar/ 32O ₂	232	242	4.12	4.28
4	1kw/100w/200khz/2.2µs/300s/20Ar/ 28O ₂	237	243	4.1	4.22
5	1kw/100w/200khz/2.2µs/300s/20Ar/ 24O ₂	233	243	4.1	4.34
6	1kw/100w/200khz/2.2µs/300s/20Ar/ 28O ₂	231	245	4.12	4.3

Table II

Slot #	Process	Rs (Ohms/Sq)	Rs unif %	Th (nm)	Th std 1sig	Bulk Rho ($\mu\text{Ohm-cm}$)	R.I (@632nm)	R.I Unif (%)	Comments
14	1.5kw/100w/200khz/2.2 μs / 300s/20Ar/80O2			38.59	0.16		1.980758	0.000005	transparent
15	1.5kw/100w/200khz/2.2 μs / 300s/20Ar/40O2	94112	2	57.28	0.51	539073.5	1.951452	0.029342	translucent
17	1.5kw/100w/200khz/2.2 μs / 300s/20Ar/40O2	33927	60.282	58.48	1.37	198405.1	1.936166	0.040957	translucent
19	1.5kw/100w/200khz/2.2 μs / 300s/20Ar/36O2	7335.32	72.49	67.75	1.03	49696.8	1.980746	0.000018	translucent
21	1.5kw/100w/200khz/2.2 μs / 300s/20Ar/30O2	22.3507	2.995	80		178.8			metallic
1	1kw/100w/200khz/2.2 μs / 300s/20Ar/80O2			26.69	0.32		1.980326	0.00096	transparent
2	1kw/100w/200khz/2.2 μs / 300s/20Ar/36O2			36.4	0.13		1.980756	0.000003	transparent
3	1kw/100w/200khz/2.2 μs / 300s/20Ar/32O2			39.3	0.15		1.980761	0	transparent
4	1kw/100w/200khz/2.2 μs / 300s/20Ar/28O2			44.02	0.24		1.98076	0.000001	transparent
5	1kw/100w/200khz/2.2 μs / 300s/20Ar/24O2	58.1031	7.467	50		290.5			metallic
6	1kw/100w/200khz/2.2 μs / 300s/20Ar/28O2	58.0992	10.566	45		261.4			metallic

Table III

Trial	Run- (sec)	Target Power (kW)	Bias/ W	O ₂	Ar	T. (°C)	Rs (Ohms/Sq)	Rs (non- unif)	Bulk Rho (uOhmcm)	Thick- ness (Å)	n	DepRa- te (A/sec)	Target N	Target/I
14	100	3	300	3	60	280	38.69	4.07%	4.64E-04	1200	1.864	12		
16	100	3	300	3	30	280	56.90	7.94%	6.98E-04	1227	1.888	12.27	288- 308	9.86- 10.42
10	100	3	100	3	60	280	50.98	11.89%	6.25E-04	1225	1.933	12.25	265- 275	10.92- 11.36
4	100	1.5	100	3	30	280	383.62	21.72%	2.09E-03	543.	2.016	5.439	238- 251	5.98-6.32
8	100	1.5	300	3	30	280	504.02	7.23%	2.44E-03	483.	2.082	4.835	239- 250	5.98-6.33
2	100	1.5	100	3	30	280	402.52	26.80%	2.10E-03	520.	2.056	5.207	225- 239	6.46-6.68
6	100	1.5	300	0	30	280	106.21	6.12%	6.17E-04	580.	1.945	5.805	237- 250	5.98-6.38
12	100	3	100	4	30	280	374.34	19.43%	4.18E-03	1116	1.917	11.16	285- 300	9.98- 10.52
15	100	3	300	4	30	100	6264.69	58.18%	6.81E-02	1087	1.897	10.87	282- 304	10.00- 10.62
7	100	1.5	200	4	30	100	7509.45	44.14%	2.95E-02	392.	2.149	3.923	237- 250	6.02-632
1	100	1.5	100	4	30	100	12284.82	112.55%	4.78E-02	389.	2.236	3.891	238- 250	6.04-632

Table III (Cont.)

11	100	3	100	3	60	100	631.77	49.40%	7.30E-03	1155	1.958	11.55	266-273	10.96-11.38
9	100	3	100	0	30	100	43.78	7.47%	5.55E-04	1268	1.945	12.68	288-307	9.78-10.42
5	100	1.5	200	3	60	100	1293.53	14.82%	5.88E-03	454.8	2.149	4.548	225-235	6.46-6.68
3	100	1.5	100	4	60	100	4154.43	28.25%	1.78E-02	428.8	2.211	4.288	226-235	6.44-6.64
13	100	3	200	0	60	100	49.05	7.24%	6.16E-04	1256	1.913	12.56	264-275	10.96-11.38
18	100	2.25	100	3	30	100	1476.79	21.54%	1.10E-02	744.5	2.044	7.445	263-277	8.08-8.56
17	100	1.5	150	0	60	100	157.23	8.83%	9.91E-04	630.5	1.931	6.305	225-231	6.48-6.74
19	100	2.25	150	3	60	100	526.72	13.01%	4.29E-03	814.2	2.021	8.142	247-255	8.78-9.14

Claims

We claim:

1. A method of forming a transparent conductive oxide film, comprising:
depositing the transparent conductive oxide film in a pulsed DC reactive ion process with substrate bias; and
controlling at least one process parameter to provide at least one characteristic of the conductive oxide film at a particular value.
2. The method of claim 1, wherein controlling at least one process parameter includes controlling the oxygen partial pressure.
3. The method of claim 1, wherein the transparent conductive oxide film includes indium-tin oxide.
4. The method of claim 1, wherein the at least one characteristic includes sheet resistance.
5. The method of claim 1, wherein the at least one characteristic includes film roughness.
6. The method of claim 5, wherein the transparent conductive oxide film includes an indium-tin oxide film and the film roughness is characterized by R_s , less than about 10 nm with R_{ms} of less than about 20 nm.
7. The method of claim 4, wherein the bulk resistance can be varied between about 2×10^{-4} micro-ohms-cm to about 0.1 micro-ohms-cm.
8. The method of claim 1, wherein the at least one process parameter includes a power supplied to a target.
9. The method of claim 1, wherein the at least one process parameter includes an oxygen partial pressure.
10. The method of claim 1, wherein the at least one process parameter includes bias power.
11. The method of claim 1, wherein the at least one process parameter includes deposition temperature.
12. The method of claim 1, wherein the at least one process parameter includes an argon partial pressure.
13. The method of claim 1, further including supplying a metallic target.

14. The method of claim 1, further including supplying a ceramic target.
15. The method of claim 1, wherein the transparent conductive oxide film is doped with at least one rare-earth ions.
16. The method of claim 15, wherein the at least one rare-earth ions includes erbium.
17. The method of claim 15, wherein the at least one rare-earth ions includes cerium.
18. A method of depositing a transparent conductive oxide film on a substrate, comprising:
 - placing the substrate in a reaction chamber;
 - adjusting power to a pulsed DC power supply coupled to a target in the reaction chamber;
 - adjusting an RF bias power coupled to the substrate;
 - adjusting gas flow into the reaction chamber; and
 - providing a magnetic field at the target in order to direct deposition of the transparent conductive oxide film on the substrate in a pulsed-dc biased reactive-ion deposition process, wherein the transparent conductive oxide film exhibits at least one particular property.
19. The method of claim 18, wherein at least one particular property of the transparent conductive oxide film is determined by parameters of the pulsed-dc biased reactive ion deposition process.
20. The method of claim 19, wherein the at least one particular property includes resistivity of the transparent conductive oxide film.
21. The method of claim 19, wherein the transparent conductive oxide film includes an indium-tin oxide film.
22. The method of claim 19, wherein the parameters include oxygen partial pressure.
23. The method of claim 19, wherein the parameters include bias power.
24. The method of claim 18, wherein the target can include at least one rare-earth ions.
25. The method of claim 24, wherein the at least one rare-earth ions includes erbium.
26. The method of claim 24, wherein the at least one rare-earth ion includes cerium.

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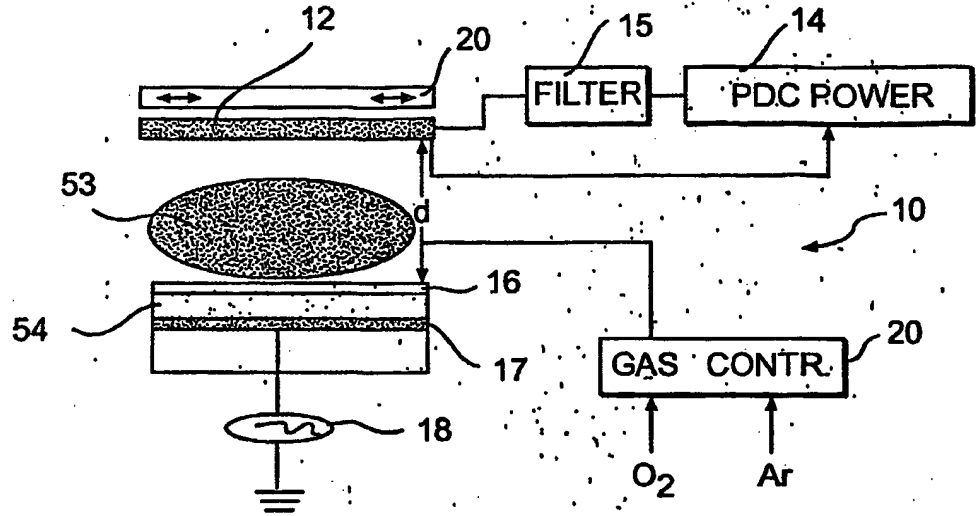


FIG. 1A

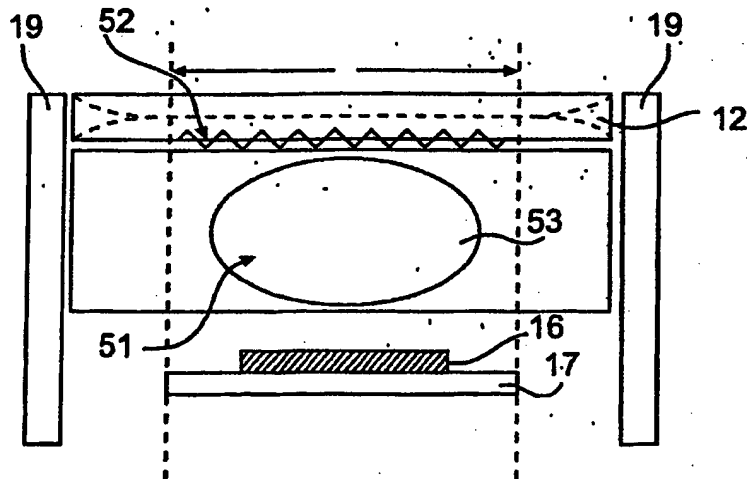


FIG. 1B

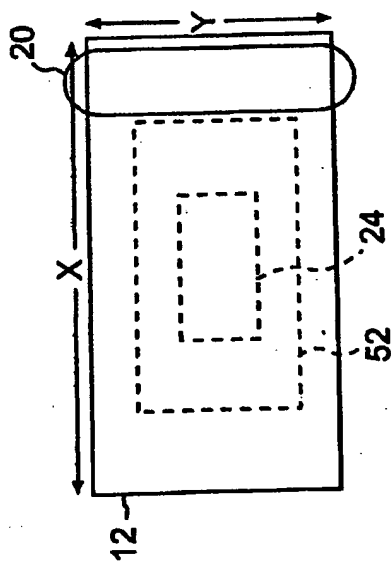


FIG. 2

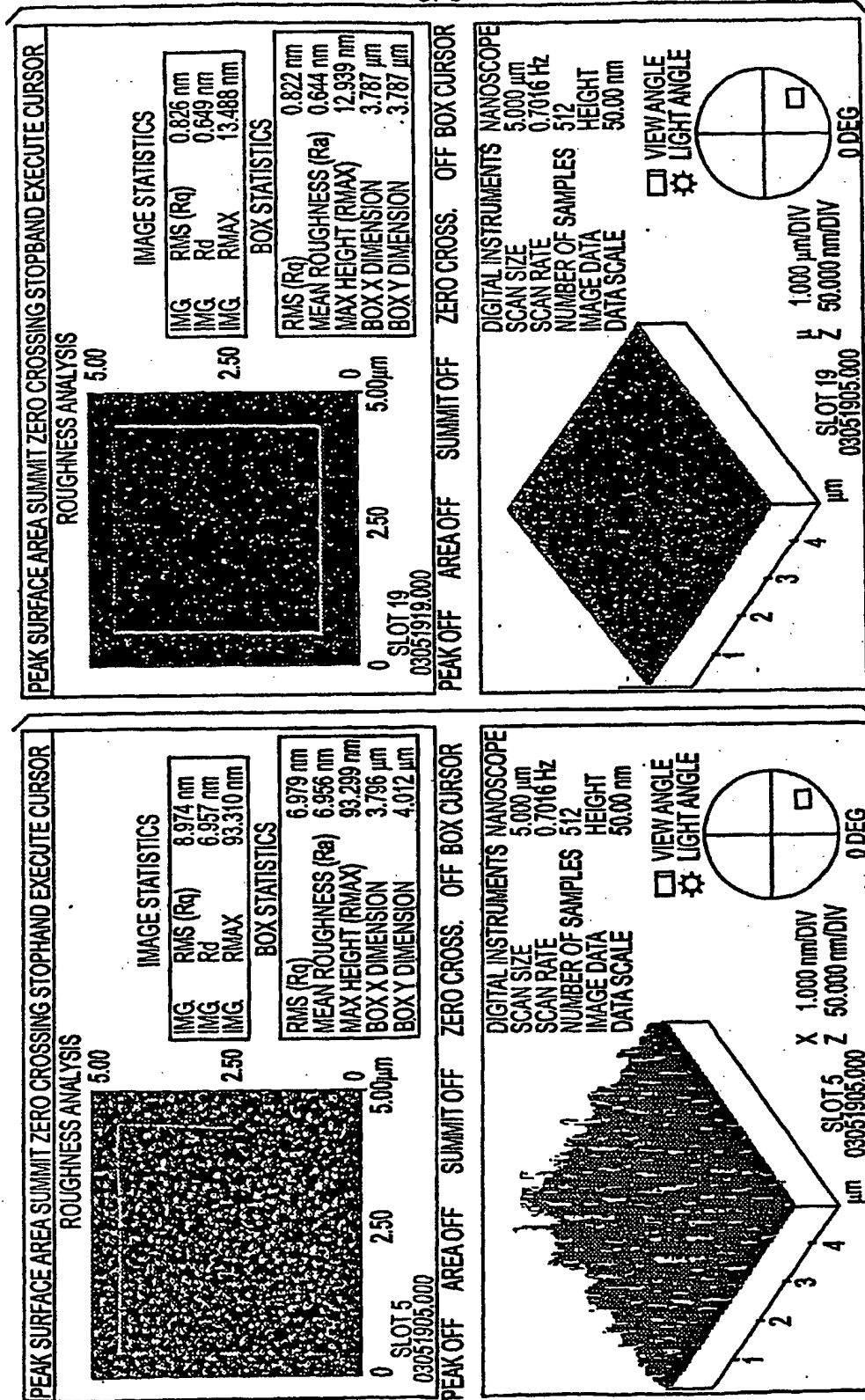


FIG. 3B

FIG. 3A

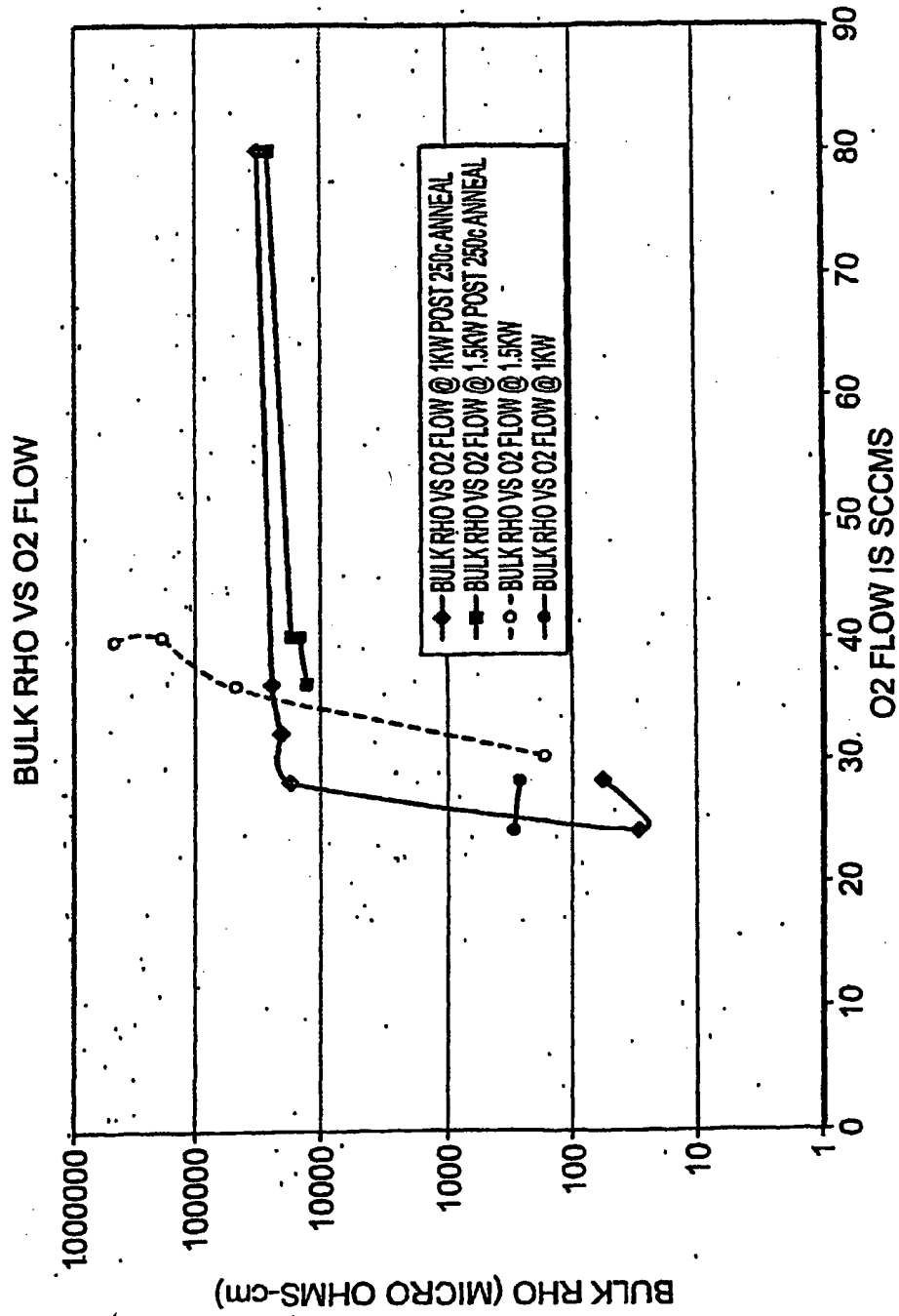


FIG. 4

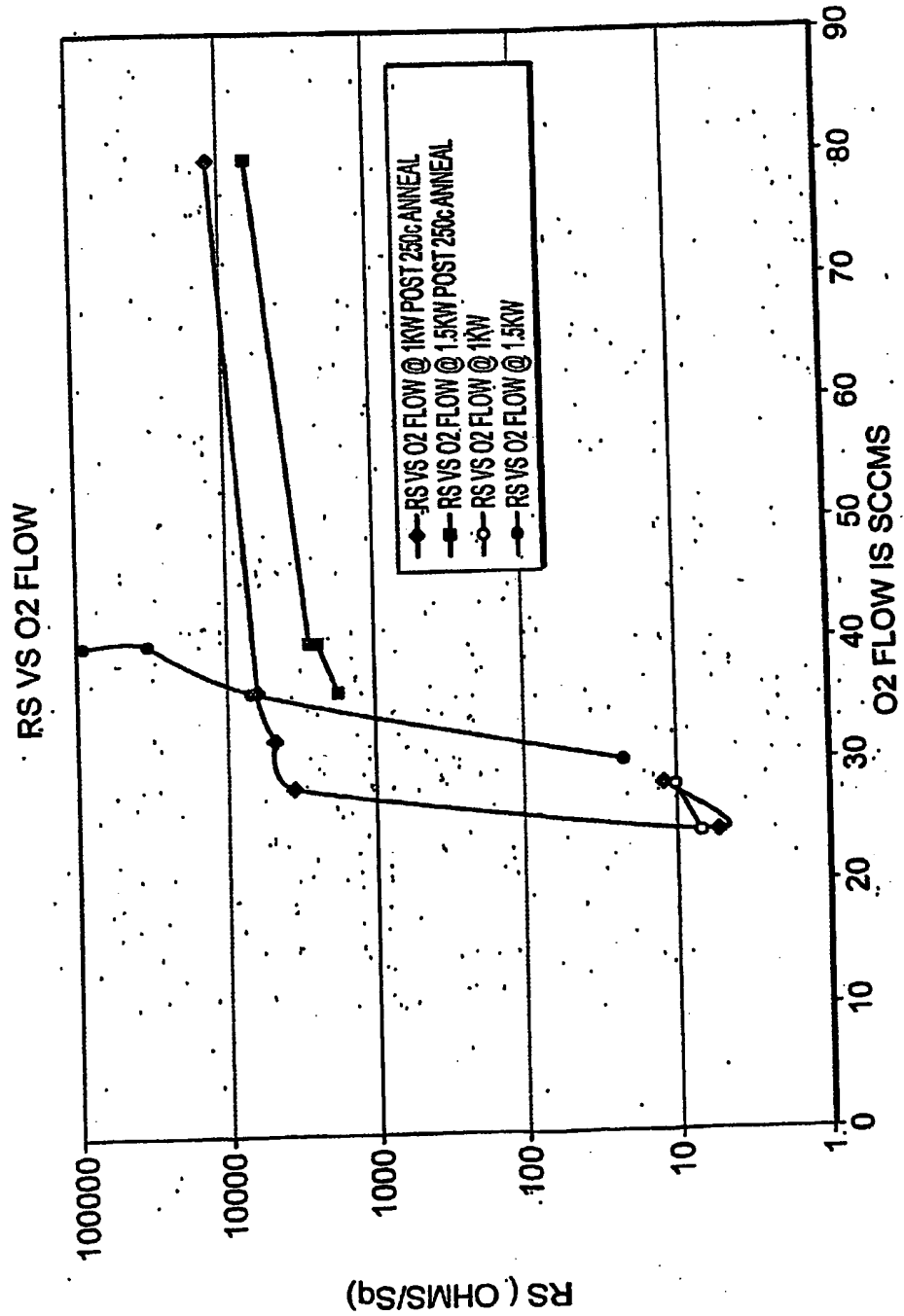


FIG. 5

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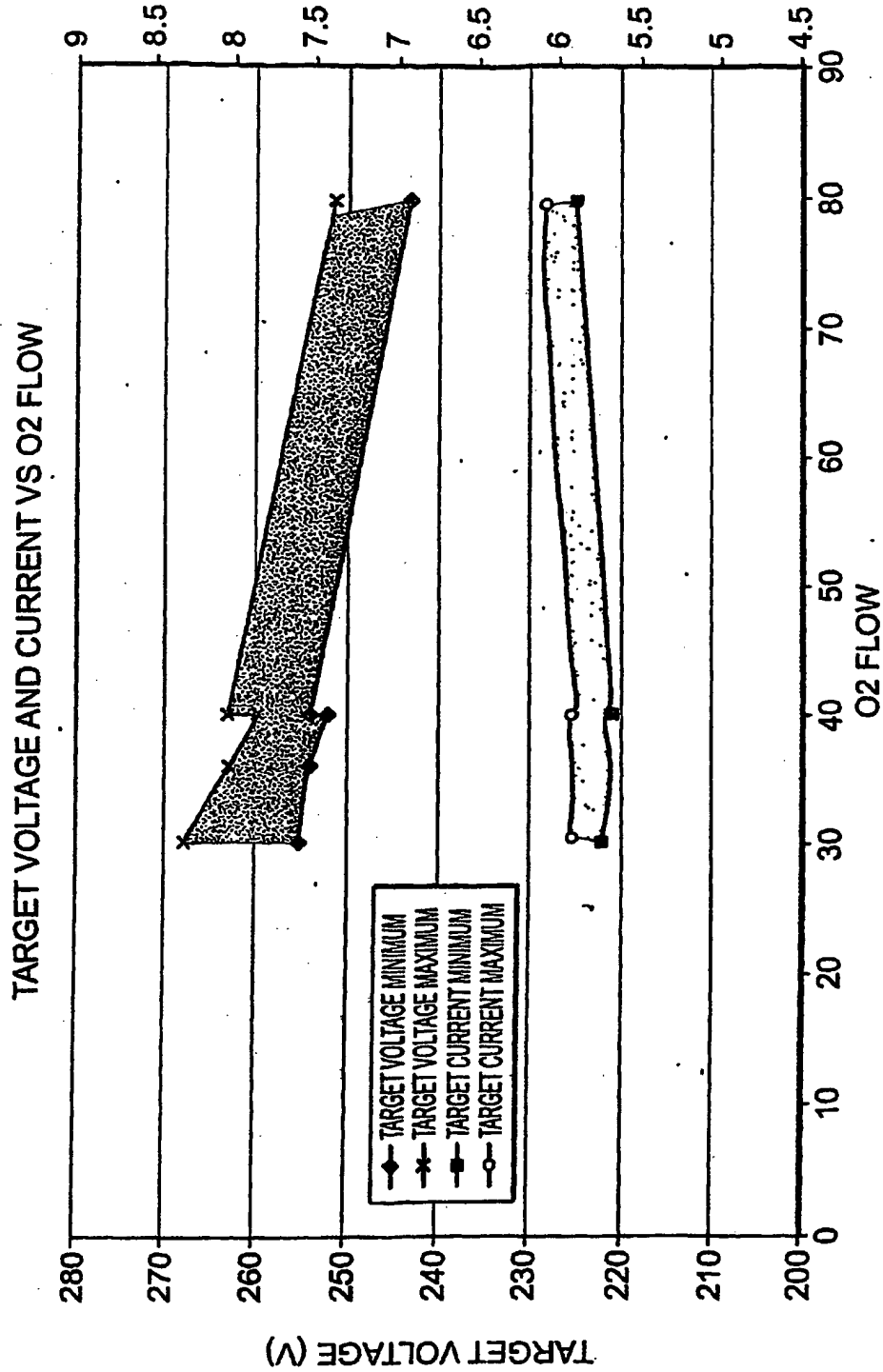


FIG. 6

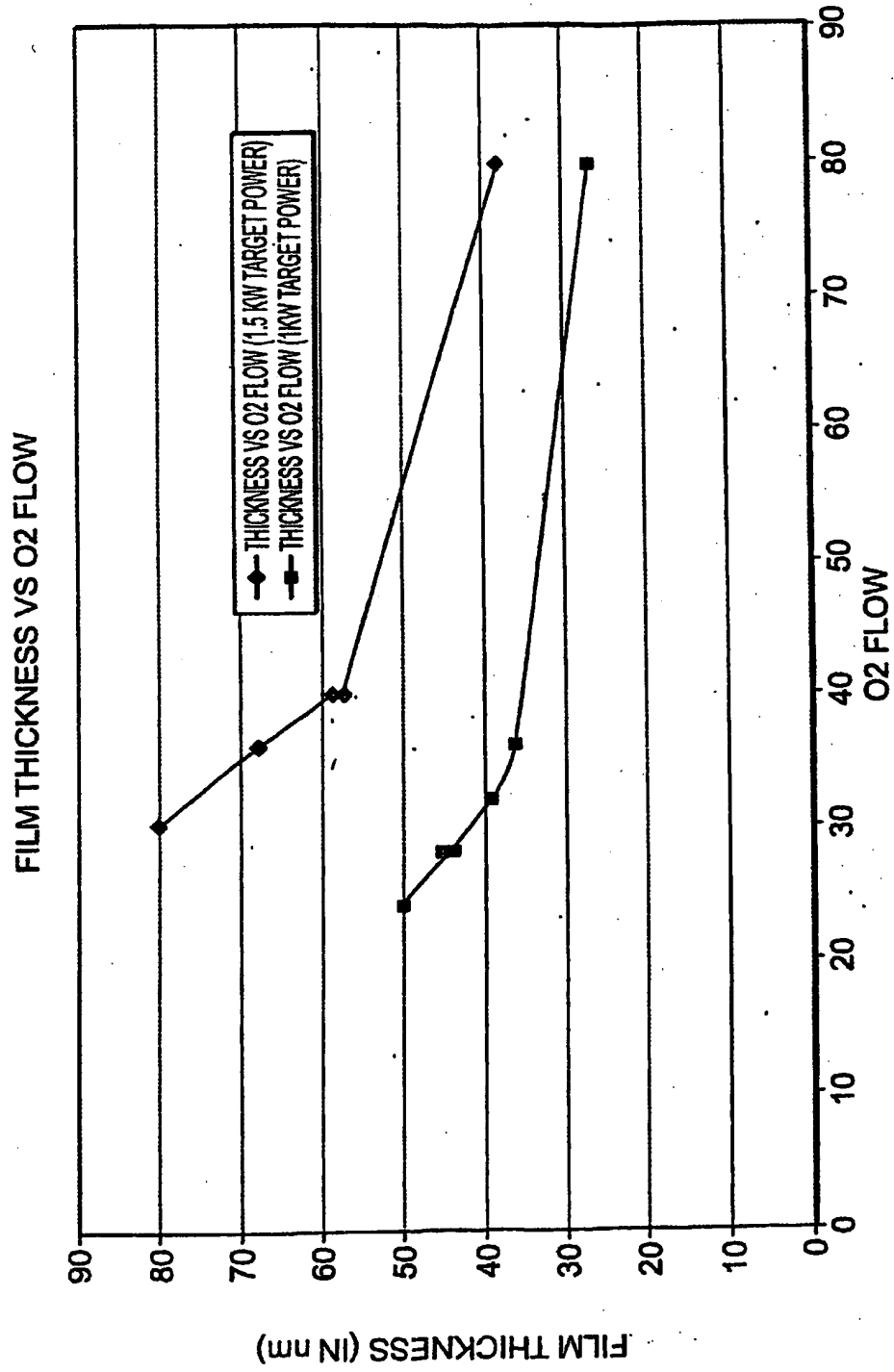


FIG. 7

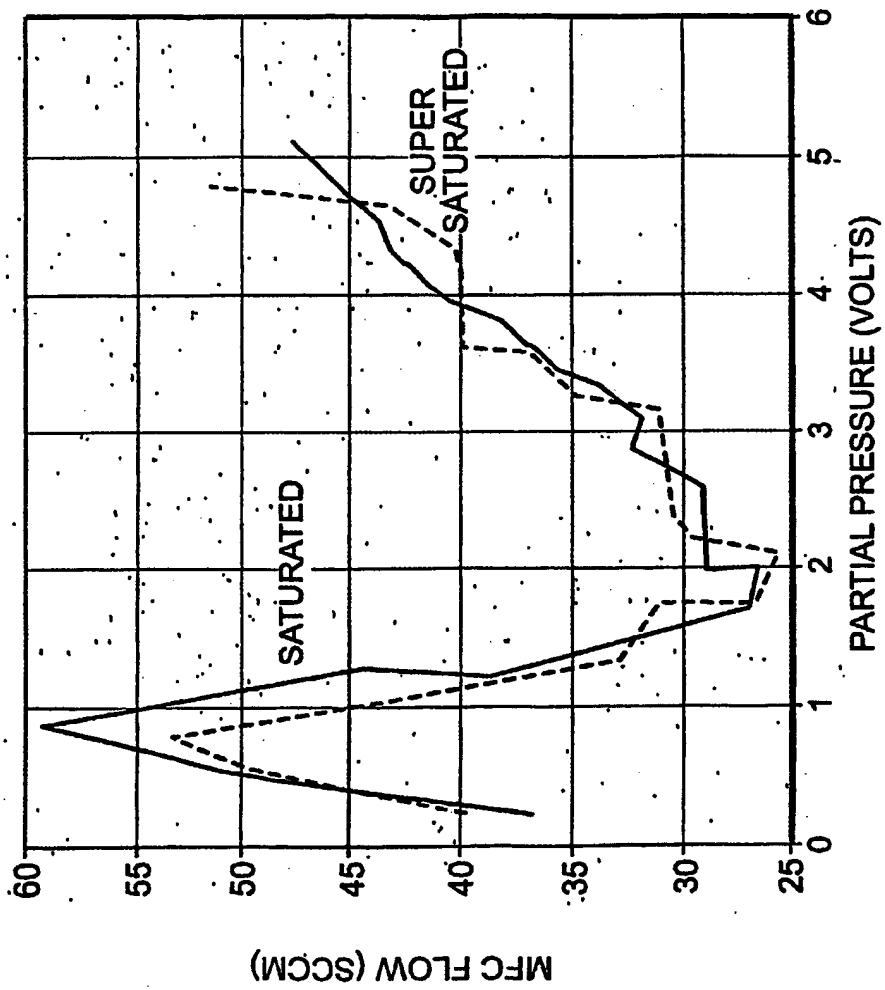


FIG. 8

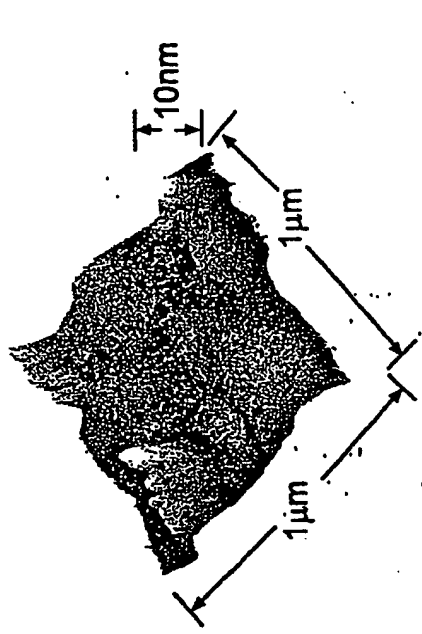


FIG. 9B



FIG. 9D

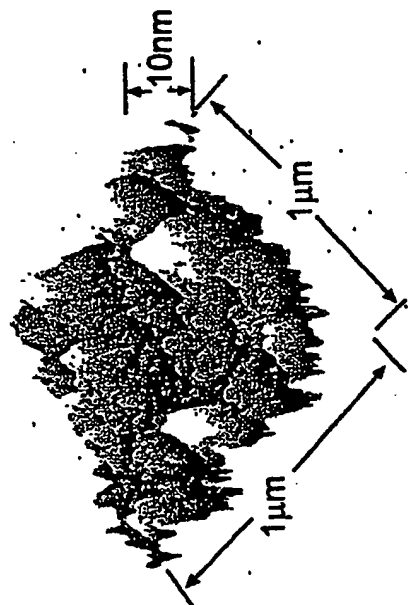


FIG. 9A

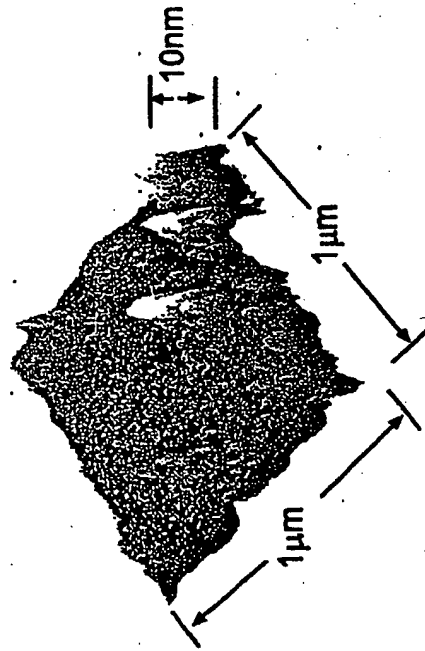


FIG. 9C

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(19) World Intellectual Property
Organization
International Bureau



(43) International Publication Date
9 December 2004 (09.12.2004)

PCT

(10) International Publication Number
WO 2004/106582 A2

- (51) International Patent Classification⁷: C23C 14/14, 14/08, 14/34, H01L 21/316
- (21) International Application Number: PCT/US2004/014524
- (22) International Filing Date: 21 May 2004 (21.05.2004)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data: 60/473,375 23 May 2003 (23.05.2003) US
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- (81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.
- (84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).
- Published:
— without international search report and to be republished upon receipt of that report
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 2004/106582 A2

(54) Title: ENERGY CONVERSION AND STORAGE FILMS AND DEVICES BY PHYSICAL VAPOR DEPOSITION OF TITANIUM AND TITANIUM OXIDES AND SUB-OXIDES

(57) Abstract: High density oxide films are deposited by a pulsed-DC, biased reactive sputtering process from a titanium containing target to form high quality titanium containing oxide films. A method of forming a titanium based layer or film according to the present invention includes depositing a layer of titanium containing oxide by pulsed-DC, biased reactive sputtering process on a substrate. In some embodiments, the layer is TiO₂. In some embodiments, the layer is a sub-oxide of Titanium. In some embodiments, the layer is Ti_xO_y, wherein x is between about 1 and about 4 and y is between about 1 and about 7. In some embodiments, the layer can be doped with one or more rare-earth ions. Such layers are useful in energy and charge storage, and energy conversion technologies.

Energy Conversion and Storage Films and Devices by Physical Vapor Deposition of Titanium and Titanium Oxides and sub-Oxides

Related Applications

The present invention claims priority to U.S. Provisional Application Serial No. 60/473,375, "Energy Conversion and Storage Devices by Physical Vapor Deposition of Titanium Oxides and Sub-Oxides," by Richard E. Demaray and Hong Mei Zhang, filed on May 23, 2003, herein incorporated by reference in its entirety.

Background

1. Field of the Invention

[0001] The present invention is related to fabrication of thin films for planar energy and charge storage and energy conversion and, in particular, thin films deposited of titanium and titanium oxides, sub oxides, and rare earth doped titanium oxides and sub oxides for planar energy and charge storage and energy conversion.

2. Discussion of Related Art

[0002] Currently, titanium oxide layers are not utilized commercially in energy storage, charge storage, or energy conversion systems because such layers are difficult to deposit, difficult to etch, are known to have large concentrations of defects, and have poor insulation properties due to a propensity for oxygen deficiency and the diffusion of oxygen defects in the layers. Additionally, amorphous titania is difficult to deposit due to its low recrystallization temperature (about 250 °C), above which the deposited layer is often a mixture of crystalline anatase and rutile structures.

[0003] However, such amorphous titania layers, if they can be deposited in sufficient quality, have potential due to their high optical index, $n \sim 2.7$, and their high dielectric constant, k less than or equal to about 100. Further, they have substantial chemical stability. There are no known volatile halides and titania is uniquely resistant to mineral acids. Amorphous titania is thought to have the further advantage that there are no grain boundary mechanisms for electrical breakdown, chemical corrosion, or optical scattering. It is also well known that the sub oxides of titanium have unique and useful properties. *See, e.g.*, Hayfield, P.C.S., "Development of a

New Material- Monolithic Ti_4O_7 Ebonix Ceramic”, Royal Society Chemistry, ISBN 0-85405-984-3, 2002. Titanium monoxide, for example, is a conductor with a uniquely stable resistivity with varying temperature. Additionally, Ti_2O_3 , which can be pinkish in color, is known to have semiconductor type properties. However, these materials have not found utilization because of their difficult manufacture in films and their susceptibility to oxidation. Further, Ti_4O_7 demonstrates both useful electrical conductivity and unusual resistance to oxidation. Ti_4O_7 , however, is also difficult to fabricate, especially in thin film form.

[0004] Additional to the difficulty of fabricating titanium oxide or sub oxide materials in useful thin film form, it also has proven difficult to dope these materials with, for example, rare earth ions, in useful or uniform concentration.

[0005] Therefore, utilization of titanium oxide and suboxide films, with or without rare earth doping, has been significantly limited by previously available thin film processes. If such films could be deposited, their usefulness in capacitor, battery, and energy conversion and storage technologies would provide for many value-added applications.

[0006] Current practice for construction of capacitor and resistor arrays and for thin film energy storage devices is to utilize a conductive substrate or to deposit the metal conductor or electrode, the resistor layer, and the dielectric capacitor films from various material systems. Such material systems for vacuum thin films, for example, include copper, aluminum, nickel, platinum, chrome, or gold depositions, as well as conductive oxides such as ITO, doped zinc oxide, or other conducting materials.

[0007] Materials such as chrome-silicon monoxide or tantalum nitride are known to provide resistive layers with 100 parts per million or less resistivity change per degree Centigrade for operation within typical operating parameters. A wide range of dielectric materials such as silica, silicon nitride, alumina, or tantalum pentoxide can be utilized for the capacitor layer. These materials typically have dielectric constants k of less than about twenty four (24). In contrast, TiO_2 either in the pure rutile phase or in the pure amorphous state can demonstrate a dielectric constant as high as 100. See, e.g., R. B. van Dover, “Amorphous Lanthanide-Doped TiO_2 Dielectric Films,”

Appl. Phys Lett., Vol. 74, no. 20, p. 3041-43 (May 17, 1999).

[0008] It is well known that the dielectric strength of a material decreases with increasing value of dielectric constant k for all dielectric films. A 'figure of merit' (FM) is therefore obtained by the product of the dielectric constant k and the dielectric strength measured in Volts per cm of dielectric thickness. Capacitive density of 10,000 to 12,000 pico Farads /mm² is very difficult to achieve with present conductors and dielectrics. Current practice for reactive deposition of titanium oxide has achieved a figure-of-merit, FM, of about 50 (k MV/cm). See J.-Y. Kim et al., "Frequency-Dependent Pulsed Direct Current Magnetron Sputtering of Titanium Oxide Films," J. Vac. Sci. Technol. A 19(2), Mar/Apr 2001.

[0009] Therefore, there is an ongoing need for titanium oxide and titanium sub-oxide layers, and rare-earth doped titanium oxide and titanium sub-oxide layers, for various applications.

Summary

[0010] In accordance with the present invention, high density oxide films are deposited by a pulsed-DC, biased, reactive sputtering process from a titanium containing target. A method of forming a titanium based layer or film according to the present invention includes depositing a layer of titanium containing oxide by pulsed-DC, biased reactive sputtering process on a substrate. In some embodiments, the layer is TiO₂. In some embodiments, the layer is a sub-oxide of Titanium. In some embodiments, the layer is Ti_xO_y wherein x is between about 1 and about 4 and y is between about 1 and about 7.

[0011] In some embodiments of the invention, the figure of merit of the layer is greater than 50. In some embodiments of the invention, the layer can be deposited between conducting layers to form a capacitor. In some embodiments of the invention, the layer includes at least one rare-earth ion. In some embodiments of the invention, the at least one rare-earth ion includes erbium. In some embodiments of the invention, the erbium doped layer can be deposited between conducting layers to form a light-emitting device. In some embodiments of the invention, the erbium doped layer can be an optically active layer deposited on a light-emitting device. In some embodiments of the invention, the layer can be a protective layer. In some

embodiments, the protective layer can be a catalytic layer.

[0012] In some embodiments of the invention, the layer and a TiO_2 layer can be deposited between conducting layers to form a capacitor with decreased roll-off characteristics with decreasing thickness of the TiO_2 layer. In some embodiments, the TiO_2 layer can be a layer deposited according to some embodiments of the present invention.

[0013] These and other embodiments of the present invention are further discussed below with reference to the following figures.

Short Description of the Figures

[0014] Figures 1A and 1B illustrate a pulsed-DC biased reactive ion deposition apparatus that can be utilized in the deposition according to the present invention.

[0015] Figure 2 shows an example of a target that can be utilized in the reactor illustrated in Figures 1A and 1B.

[0016] Figures 3A and 3B illustrate various configurations of layers according to embodiments of the present invention.

[0017] Figures 4A and 4B illustrate further various configurations of layers according to embodiments of the present invention.

[0018] Figure 5 shows another layer structure involving one or more layers according to the present invention.

[0019] Figure 6 shows a transistor gate with a TiO_y layer according to the present invention.

[0020] Figure 7 illustrates the roll-off of the dielectric constant with decreasing film thickness.

[0021] Figure 8 illustrates data points from a bottom electrode that helps reduce or eliminate the roll-off illustrated in Figure 7.

[0022] Figures 9A and 9B illustrate an SEM cross-section of a Ti_4O_7 target obtained from Ebonex™ and an SEM cross section of the $\text{Ti}_4\text{O}_{6.8}$ film deposited from the Ebonex™ target according to the present invention.

[0023] Figure 10 shows the industry standard of thin-film capacitor performance in comparison with layers according to some embodiments of the present invention.

[0024] Figure 11 shows the performance of various thin films deposited according

to the present invention in a capacitor structure.

[0025] Figure 12 shows a cross-section TEM and diffraction pattern amorphous and crystalline layers of TiO_2 on n^{++} wafers.

[0026] Figure 13 shows a comparison of the leakage current for TiO_2 films according to embodiments of the present invention with and without erbium ion doping.

[0027] Figures 14A and 14B show a photoluminescence signal measured from a 5000 Å layer of 10% erbium containing TiO_2 deposited from a 10% erbium doped TiO conductive target and a photoluminescence signal measured from the same layer after a 30 minute 250 °C anneal.

[0028] In the figures, elements having the same designation have the same or similar functions.

Detailed Description

[0029] Miniaturization is driving the form factor of portable electronic components. Thin film dielectrics with high dielectric constants and breakdown strengths allow production of high density capacitor arrays for mobile communications devices and on-chip high-dielectric capacitors for advanced CMOS processes. Thick film dielectrics for high energy storage capacitors allow production of portable power devices.

[0030] Some embodiments of films deposited according to the present invention have a combination of high dielectric and high breakdown voltages. Newly developed electrode materials allow the production of very thin films with high capacitance density. The combination of high dielectric and high breakdown voltages produce thick films with new levels of available energy storage according to $E=1/2CV^2$.

[0031] Deposition of materials by pulsed-DC biased reactive ion deposition is described in U.S. Patent Application Serial No. 10/101863, entitled "Biased Pulse DC Reactive Sputtering of Oxide Films," to Hongmei Zhang, et al., filed on March 16, 2002. Preparation of targets is described in U.S. Patent Application Serial No. 10/101,341, entitled "Rare-Earth Pre-Alloyed PVD Targets for Dielectric Planar Applications," to Vassiliki Milonopoulou, et al., filed on March 16, 2002. U.S. Patent

Application Serial No. 10/101863 and U.S. Patent Application Serial No. 10/101,341 are each assigned to the same assignee as is the present disclosure and each is incorporated herein in their entirety. Additionally, deposition of materials is further described in U.S. Patent 6,506,289, which is also herein incorporated by reference in its entirety.

[0032] Figure 1A shows a schematic of a reactor apparatus 10 for sputtering of material from a target 12 according to the present invention. In some embodiments, apparatus 10 may, for example, be adapted from an AKT-1600 PVD (400 X 500 mm substrate size) system from Applied Komatsu or an AKT-4300 (600 X 720 mm substrate size) system from Applied Komatsu, Santa Clara, CA. The AKT-1600 reactor, for example, has three deposition chambers connected by a vacuum transport chamber. These AKT reactors can be modified such that pulsed DC (PDC) power is supplied to the target and RF power is supplied to the substrate during deposition of a material film. The PDC power supply 14 can be protected from RF bias power 18 by use of a filter 15 coupled between PDC power supply 14 and target 12.

[0033] Apparatus 10 includes a target 12 which is electrically coupled through a filter 15 to a pulsed DC power supply 14. In some embodiments, target 12 is a wide area sputter source target, which provides material to be deposited on substrate 16. Substrate 16 is positioned parallel to and opposite target 12. Target 12 functions as a cathode when power is applied to it and is equivalently termed a cathode. Application of power to target 12 creates a plasma 53. Substrate 16 is capacitively coupled to an electrode 17 through an insulator 54. Electrode 17 can be coupled to an RF power supply 18. Magnet 20 is scanned across the top of target 12.

[0034] For pulsed reactive dc magnetron sputtering, as performed by apparatus 10, the polarity of the power supplied to target 12 by power supply 14 oscillates between negative and positive potentials. During the positive period, the insulating layer on the surface of target 12 is discharged and arcing is prevented. To obtain arc free deposition, the pulsing frequency exceeds a critical frequency that depends on target material, cathode current and reverse time. High quality oxide films can be made using reactive pulsed DC magnetron sputtering in apparatus 10.

[0035] Pulsed DC power supply 14 can be any pulsed DC power supply, for example

an AE Pinnacle plus 10K by Advanced Energy, Inc. With this example supply, up to 10 kW of pulsed DC power can be supplied at a frequency of between 0 and 350 KHz. In some embodiments, the reverse voltage is 10% of the negative target voltage. Utilization of other power supplies will lead to different power characteristics, frequency characteristics, and reverse voltage percentages. The reverse time on this embodiment of power supply 14 can be adjusted to between 0 and 5 μ s.

[0036] Filter 15 prevents the bias power from power supply 18 from coupling into pulsed DC power supply 14. In some embodiments, power supply 18 can be a 2 MHz RF power supply, for example a Nova-25 power supply made by ENI, Colorado Springs, Co.

[0037] Therefore, filter 15 can be a 2 MHz band sinusoidal rejection filter. In some embodiments, the bandwidth of the filter can be approximately 100 kHz. Filter 15, therefore, prevents the 2 MHz power from the bias to substrate 16 from damaging power supply 18.

[0038] However, both RF sputtered and pulsed DC sputtered films are not fully dense and may typically have columnar structures. These columnar structures are detrimental to thin film applications. By applying a RF bias on wafer 16 during deposition, the deposited film can be densified by energetic ion bombardment and the columnar structure can be substantially eliminated or completely eliminated.

[0039] In the AKT-1600 based system, for example, target 12 can have an active size of about 675.70 X 582.48 by 4 mm in order to deposit films on substrate 16 that have dimension about 400 X 500 mm. The temperature of substrate 16 can be held at between -50C and 500C by introduction of back-side gas in a physical or electrostatic clamping of the substrate, thermo-electric cooling, electrical heating, or other methods of active temperature control. In Figure 1A, a temperature controller 22 is shown to control the temperature of substrate 16. The distance between target 12 and substrate 16 can be between about 3 and about 9 cm. Process gas can be inserted into the chamber of apparatus 10 at a rate up to about 200 sccm while the pressure in the chamber of apparatus 10 can be held at between about .7 and 6 millitorr. Magnet 20 provides a magnetic field of strength between about 400 and about 600 Gauss directed

in the plane of target 12 and is moved across target 12 at a rate of less than about 20-30 sec/scan. In some embodiments utilizing the AKT 1600 reactor, magnet 20 can be a race-track shaped magnet with dimension about 150 mm by 600 mm.

[0040] Figure 2 illustrates an example of target 12. A film deposited on a substrate positioned on carrier sheet 17 directly opposed to region 52 of target 12 has good thickness uniformity. Region 52 is the region shown in Figure 1B that is exposed to a uniform plasma condition. In some implementations, carrier 17 can be coextensive with region 52. Region 24 shown in Figure 2 indicates the area below which both physically and chemically uniform deposition can be achieved, where physical and chemical uniformity provide refractive index uniformity, for example. Figure 2 indicates that region 52 of target 12 that provides thickness uniformity is, in general, larger than region 24 of target 12 providing thickness and chemical uniformity. In optimized processes, however, regions 52 and 24 may be coextensive.

[0041] In some embodiments, magnet 20 extends beyond area 52 in one direction, the Y direction in Figure 2, so that scanning is necessary in only one direction, the X direction, to provide a time averaged uniform magnetic field. As shown in Figures 1A and 1B, magnet 20 can be scanned over the entire extent of target 12, which is larger than region 52 of uniform sputter erosion. Magnet 20 is moved in a plane parallel to the plane of target 12.

[0042] The combination of a uniform target 12 with a target area 52 larger than the area of substrate 16 can provide films of highly uniform thickness. Further, the material properties of the film deposited can be highly uniform. The conditions of sputtering at the surface of target 12, such as the uniformity of erosion, the average temperature of the plasma at the target surface and the equilibration of the target surface with the gas phase ambient of the process are uniform over a region which is greater than or equal to the region to be coated with a uniform film thickness. In addition, the region of uniform film thickness is greater than or equal to the region of the film which is to have highly uniform optical properties such as index of refraction, density, transmission, or absorptivity.

[0043] Target 12 can be formed of any materials, but is typically metallic materials such as, for example, combinations of In and Sn. Therefore, in some embodiments,

target 12 includes a metallic target material formed from intermetallic compounds of optical elements such as Si, Al, Er and Yb. Additionally, target 12 can be formed, for example, from materials such as La, Yt, Ag, Au, and Eu. To form optically active films on substrate 16, target 12 can include rare-earth ions. In some embodiments of target 12 with rare earth ions, the rare earth ions can be pre-alloyed with the metallic host components to form intermetallics. See U.S. Application Serial No. 10/101,341. [0044] In several embodiments of the invention, material tiles are formed. These tiles can be mounted on a backing plate to form a target for apparatus 10. A wide area sputter cathode target can be formed from a close packed array of smaller tiles. Target 12, therefore, may include any number of tiles, for example between 2 to 20 individual tiles. Tiles are finished to a size so as to provide a margin of non-contact, tile to tile, less than about 0.010" to about 0.020" or less than half a millimeter so as to eliminate plasma processes that may occur between adjacent ones of the tiles. The distance between the tiles of target 12 and the dark space anode or ground shield 19 in Figure 1B can be somewhat larger so as to provide non contact assembly or provide for thermal expansion tolerance during processing, chamber conditioning, or operation.

[0045] As shown in Figure 1B, a uniform plasma condition can be created in the region between target 12 and substrate 16 in a region overlying substrate 16. A plasma 53 can be created in region 51, which extends under the entire target 12. A central region 52 of target 12, can experience a condition of uniform sputter erosion. As discussed further below, a layer deposited on a substrate placed anywhere below central region 52 can then be uniform in thickness and other properties (i.e., dielectric, optical index, or material concentrations). In addition, region 52 in which deposition provides uniformity of deposited film can be larger than the area in which the deposition provides a film with uniform physical or optical properties such as chemical composition or index of refraction. In some embodiments, target 12 is substantially planar in order to provide uniformity in the film deposited on substrate 16. In practice, planarity of target 12 can mean that all portions of the target surface in region 52 are within a few millimeters of a planar surface, and can be typically within 0.5 mm of a planar surface.

[0046] Figure 3A illustrates deposition of a layer 102 according to the present invention deposited on a substrate 101. In some embodiments, layer 102 can be a conducting protective layer of TiO_y . Figure 3B shows a first layer 102 according to the present invention deposited over a second layer 103, which can also be a layer according to some embodiments of the present invention. In some embodiments, first layer 102 can be a conducting protective layer and second layer 103 can be a titanium or other conducting layer. Layer 103 is deposited on substrate 101.

[0047] The fabrication of high density capacitor and resistor arrays as well as high energy storage solid state devices can be accomplished with embodiments of processes according to the present invention on a wide variety of substrates such as silicon wafers or glass or plastic sheets at low temperature and over wide area. With reference to Figure 3B, layer 102 can be an amorphous film of TiO_2 , which is deposited by a process such as that described in U.S. Application Serial No. 10/101,341. Utilization or formation of a conducting layer 103 such as TiO or Ti_4O_7 between a conducting layer of titanium, which is substrate 101, and the dielectric TiO_2 layer 102 is shown in the present invention to substantially reduce or eliminate the 'roll off' of the dielectric constant k with decreasing film thickness below about 1000 Angstroms. Consequently, capacitors fabricated from titanium on low temperature substrates result in high value planar capacitors and capacitor arrays with very high capacitive density and low electrical leakage. Such electrical arrays are useful for shielding and filtering and buffering high frequency and may be used in stationary as well as in portable electronic devices.

[0048] In particular, the low temperature deposition of amorphous titania capacitors provides for the fabrication of integrated passive electronic circuits on plastic and glass. It also provides for the integration of such devices on other electronic devices and arrays at low temperature.

[0049] Similarly, a conducting layer of TiO or Ti_4O_7 as layer 103 in Figure 3B, deposited between a conducting layer of titanium as layer 101 and a layer of titania as layer 102 of Figure 3B can be deposited so as to provide an increase in the surface smoothness by planarization of the titanium in layer 101 or other metallurgical conductive substrate layer 101 of Figure 3B. Consequently, roughness or asperity

based defects can be minimized or eliminated. As an example, charge injection from a metallurgical electrode can be decreased at the interface with a dielectric. The titanium based dielectric layer can be formed on a smooth conducting oxide layer, which according to some theories can prevent charge depletion of the high k dielectric layer, decrease point charge accumulation and support dipole formation at the conductor-dielectric interface, sometimes referred to as dipole coupling. These features are important to prevent the roll-off of the dielectric strength of the dielectric layer as the layer thickness is decreased below about 1000 Å. It is consequently useful in the formation of thin layers having high capacitive value.

[0050] A thick film of dielectric material may be deposited having a high dielectric strength for the storage of electrical energy. Such energy is well known to increase with the square of the applied Voltage. For example, in Figure 3B layer 102 can be a thick layer of dielectric according to the present invention. Layer 104 in Figure 3B, then, can be a conducting layer deposited on layer 102 while layer 103 is a conducting layer deposited between a substrate 101 and layer 102 to form a capacitor. As the dielectric strength of the amorphous dielectric layer of layer 102 increases in proportion to its thickness, the energy storage also increases effectively as the square of the thickness. It is shown that both record capacitance density and electrical energy storage density result for films according to the present invention. For thick film applications, smoothing of the metallurgical electrode by a conductive sub-oxide can decrease leakage at the interface in high voltage applications.

[0051] Protective conductive sub-oxide films of titanium can also be deposited on conductive and insulating substrates to protect them from harmful chemical attack while acting as conducting layers. For example, as illustrated in Figure 3A layer 102 can be a protective conductive sub-oxide film deposited on substrate 101. These layers can be used to protect an electrode, which can be substrate 101, from oxidation in the gas phase and in the liquid phase as well as the solid phase. Examples of such applications include electrolytic energy storage or as an active electrode surface for catalytic reactions and energy conversion such as in the oxygen-hydrogen fuel cell. Transparent oxides and semi-transparent sub-oxides can be deposited sequentially so that the conducting sub-oxides are protected by the transparent non-conducting oxides

for purposes of photovoltaic or electrochromic energy conversion devices. It is well known that organic based photovoltaic cells are enhanced by the presence of titania in the organic absorbing layer. Layers according to the present invention can be utilized both for the conductivity of electricity, the enhancement of the organic absorber, as well as the overall protection of the device.

[0052] TiO_2 layers, for example, can photocatalytically produce ozone in the presence of sunlight. However, in the course of such activity, the TiO_2 layer can build up a fixed charge. Absent a metallurgical conductor, as shown in Figure 3B layer 102 can be a catalytic oxide while layer 103 can be a conducting suboxide while substrate 101 is a dielectric substrate such as glass or plastic and layer 104 is absent. In such a two-layer device, where the oxide is provided on the surface of the sub-oxide, the sub-oxide can form an electrode so that electric charge can be conducted to the oxide layer for enhanced photochemical photolysis such as in an AC device, or for the purpose of charge dissipation.

[0053] Protective conductive sub-oxide films of titanium can also be deposited on conductive and insulating substrates to protect them from harmful chemical attack while acting as conducting layers for electrolytic energy storage or as an active electrode for catalytic energy conversion. Transparent and semi-transparent oxides can be deposited sequentially so that the conducting suboxides are protected by the transparent non-conducting oxide for purposes of protecting layered devices.

Alternatively, it is well known that certain crystalline suboxides of titania, collectively referred to as Magnelli phases, possess unusual levels of durability to mineral acid solutions and other corrosive gaseous or liquid environments. Hayfield, P.C.S., "Development of a New Material- Monolithic Ti_4O_7 Ebonix Ceramic", Royal Society Chemistry, ISBN 0-85405-984-3, 2002 describes these in detail and discusses many applications of the monolithic suboxides. Hayfield also explains that the basis of conductivity of sub-oxides is due to the presence of the Ti^{+2} cation in layers having the stoichiometry TiO . Of the several compositions, Ti_4O_7 in particular is known to possess both useful conductivity and also chemical resistance to both anodization, which would decrease its conductivity, as well as reduction, which would decrease its chemical durability. Therefore, as shown in Figure 3A, substrate 101 can be a

metallurgical substrate such as aluminum or titanium and layer 102 can be Ti_4O_7 . An example is the catalytic of H_2 and O_2 to make water and electricity.

[0054] In this disclosure, an amorphous coating layer according to embodiments of the present invention, derived from a crystalline target of Ti_4O_7 , can obtain a similar composition as described above, measured as $Ti_4O_{6.8}$. Similar useful levels of chemical conductivity can be obtained. The sputtered film was dense, adherent, and also displayed robust durability to immersion in concentrated mineral acid and oxidizing solution. A similar material was deposited directly from a titanium target using the subject reactive sputtering process.

[0055] The increased density of the amorphous sputtered film according to embodiments of the present invention such as film 102 shown in Figure 3A can provide high levels of impermeability. Planarization can also be achieved by layer 102 over structures on substrate 101. Layer 102 can therefore achieve 'atomic' smooth surfaces on otherwise rough substrates. The sputtering process according to the present invention also allows the formation of a continuous range of stoichiometry between what are, in their crystalline environment, 'line compounds' with whole number integer ratios of titanium cations to oxygen atoms. In the present amorphous films, as long as one Ti^{+2} has a nearest neighbor cation in the amorphous glass matrix with the Ti^{+2} valence, conductive paths will be available in the sputtered film.

[0056] The sputtered sub-oxides also have the advantage that they can be layered, without removal from the vacuum system, with metallic titanium, other sub-oxides, as well as TiO_2 for connection to electrical conduction and insulation. This feature provides the utility of multiplayer depositions by integrated processes in one vacuum chamber. Where thick films of a particular sub-oxide are desired, a target 12 (Figure 1) fabricated of the desired sub-oxide can be utilized. TiO is particularly a good conductor and possesses very stable resistivity with temperature variation. Ti_2O_3 is a semiconductor. The higher oxygen-containing Magnelli compositions obtain higher resistivity as well as increased chemical stability and robustness and can be utilized as a resistive layer or as a protective, conductive layer.

[0057] Erbium doped TiO_2 is known to display useful levels of

photoluminescence. And rare earth doped titanium oxide is known to display decreased levels of electrical leakage current under conditions of high electrical field. Layer 102 of Figure 3B, deposited according to some embodiments of the present invention, then can be erbium doped TiO_2 and therefore displays very high level of breakdown and very low leakage under electrical stress. Additionally, a capacitor can be formed by deposition of conductors as layers 103 and 104 on a substrate 101. Consequently, capacitive and energy storage devices formed from rare earth doped layers formed according to the present invention are extremely useful for very high field applications such as capacitors, high voltage dielectric batteries, and electro luminescent devices and also for low-leakage devices.

[0058] A TiO or erbium-doped TiO target, target 12 of Figure 1A, can be formed by mixing of TiO powder or TiO powder and Erbium or Erbium-Oxide powder. TiO powder can be formed from the partial oxygenation in a controlled furnace. The mixed powder is then hipped under a controlled environment (for example hydrogen or CO_2) to a high density to form tiles. As discussed above, such tiles can be mounted to form target 12. Additionally, other rare-earth doped titanium containing targets can be formed in the same fashion.

[0059] As an example, a layer of erbium doped titania or titania containing alloy deposited by means of the present invention, could be coupled as a continuous oxide layer to a photo diode constructed proximate to dielectric layer 102 of Figure 3A. Such an arrangement could provide an optical means for the measurement of the applied electrical field or the leakage current.

[0060] Alternatively, such a rare earth doped dielectric layer 102 might be coupled to conducting transparent oxides so that a light wave device might be provided for the conversion of electrical energy to light energy. In another embodiment, a titanium oxide containing a rare earth ion can be deposited directly on a light emitting diode device so that the rare earth ion can absorb some or all of the light emitted by the diode and re-fluoresce that light at another wavelength. In this embodiment, layer 102 can be a rare earth containing titanium oxide or sub oxide and substrate 101 includes a light emitting diode. An example of this may be the conversion of blue light from a LED to yellow-green light by layer 102. In that case,

layer 102 may be cerium doped titanium oxide or sub-oxide. Partial absorption of the blue light by layer 102 with conversion to yellow-green light by layer 102 would result in a white light source. Other colors of light can be obtained by doping the titanium oxide or sub-oxide with other rare earth ions.

[0061] Figures 4A and 4B illustrate further stackings of layers according to embodiments of the present invention. For example, layer 201 can be a TiO_2 dielectric protective deposited over a conducting layer 103 on substrate 101. Figure 4B can show dielectric protective layer 201 deposited over conducting protective layer 102 of TiO_y , which is deposited on a metal conducting layer 103 on substrate 101. The TiO_y conducting protective layer can act as a smoothing layer, resulting in a better barrier layer in dielectric 201. The end result is a better roll-off characteristic than has previously been obtained.

[0062] In general, layer 102 can be formed of any Ti_xO_y layer or rare earth doped Ti_xO_y layer according to the present invention. As illustrated here, layers of various compositions of Ti_xO_y , with or without rare-earth doping, have various properties. In some embodiments of the invention, x can be between about 1 and about 4 and y can be between about 1 and about 7.

[0063] Figure 5 shows an example of a capacitor stack according to the present invention. A metal conducting layer 103 is deposited on substrate 101. A conducting protective layer 102 is deposited over conducting layer 103 and a TiO_2 dielectric protective layer is deposited over the protective conducting layer 102. Another protective conducting layer 102 can be deposited over the TiO_2 dielectric layer and a metal layer can be deposited over the protective conducting layer 102. The resulting capacitor stack has upper and lower smoothing due to the two TiO_y layers and results in improved roll-off characteristics in the dielectric constant. Such capacitor stacks can be very useful in energy storage devices.

[0064] Figure 6 shows a transistor structure according to the present invention. A source 401, drain 402 and gate structure 404 are deposited on a semiconducting substrate 403. An intermediate dielectric 400 can then be deposited over the source, drain and gate structure. A protective conducting layer 102, which can be formed of TiO_y , can then be deposited over an opening in the intermediate dielectric layer 400

followed by a conducting layer 103. The protective conducting layer 102 prevents roll-off of the gate dielectric 404.

Example 1. Deposition of Ti₄O₇ film

[0065] In this example, Ti₄O₇ films were deposited using a Pulse DC scanning magnetron PVD process as was previously described in U.S. Application Serial No. 10/101,341. The target was a about 1mm thick, about 16.5x12.5 mm² tiles of titanium oxide target obtained from a sheet of Ebonex™ which compounded of bulk Ti₄O₇ was bonded onto a backing plate. Ebonex™ can be obtained from Atraverda Ltd., Oakham Business Park, Mansfield, UK. A pulsed DC generator from Advanced Energy (Pinnacle Plus) was used as the target power supply. The pulsing frequency can be varied from 0-350 KHz. Reversed duty cycle can be varied from 1.3μs to 5μs depending on the pulsing frequency. Target power was fixed at 2 KW and pulsing frequency was 200KHz during deposition, Ar flow rate is 100sccm. The deposition rate at this condition is 14Å/sec over a 40 by 50 cm substrate 101. A 100 W at 2 MHz bias was supplied to the substrate. The bias power supply can be an RF supply produced by ENI.

[0066] Utilizing the above parameters, a layer 102 of Figure 3A was deposited on a substrate 101 of 150mm p-type Si wafer. The sheet resistance was measured using 4 point probe to be 140 ohms/sq, with film thickness of 1.68μm. The resistivity of the resulting film is measured to be 0.023 ohms-cm. The composition of film was determined using EDX to be Ti₄O_{6.8}.

Example 2. Deposition of TiO₂ on Ti-Ti₄O₇ film Stack

[0067] In this example, TiO₂ films were deposited using a 2MHz RF biased, Pulse DC scanning magnetron PVD process as was previously described in U.S. Application Serial No. 10/101,341. The substrate size can be up to 600x720mm². The target was a ~7mm thick, ~630x750 mm² Ti plate of 99.9% purity. A pulsed DC generator, or PDC power supply from Advanced Energy (Pinnacle Plus) was used as the target power supply. The pulsing frequency can be varied from 0-350 KHz. Reversed duty cycle can be varied from 1.3μs to 5μs depending on the pulsing

frequency. An ENI RF generator and ENI Impedance matching unit were used for the substrate bias. A 100 W with a 2 MHz RF generator, which can be an EFI supply, was utilized. The chamber base pressure was kept below 2×10^{-7} Torr. The substrate temperature was below 200°C during deposition.

[0068] A systematic DOE (design of experiments) were carried out on both n++ type bare Si wafers and Al metallized wafers. All n++ wafers were HF cleaned just before loading into the chamber for deposition. A series of 150nm thick, Al films were deposited onto the bare Si wafers using the same PVD system at low temperature (<100°C).

[0069] The total PDC target power, pulsing frequency, oxygen partial pressure, and substrate bias power were variables in the DOE. Total gas flow of Ar and O₂ were kept constant at 100 sccm. The PDC target power was between 4 and 7 kW with a pulsing frequency of between 100 and 250 kHz. The oxygen flow rate ranged from 30 to 60%. The bias power ranged from 0 to 300 W at 2 Mhz. Both dielectric strength and breakdown voltage were measured using a mercury probe. Film thickness in this DOE range from 100nm to 270nm.

[0070] Therefore, with reference to Figure 3B, layer 101 is the Si wafer substrate, layer 103 is the 150 nm thick Al layer, layer 102 is the Ti₄O₇ layer, and layer 104 is TiO₂. Figure 7 shows the thickness dependence of the dielectric constant of layer 102, showing the roll off effect. The capacitance of the layer stack 101, 103, 102, and 104 was measured with a mercury electrode impressed upon layer 104 and coupled to layer 103. The precise thickness of dielectric layer 104 was measured optically. The dielectric constant of layer 104 was then calculated from the measured capacitance. As shown in Figure 7, the TiO₂ film thickness decreases, so does the dielectric constant of the TiO₂ film.

[0071] However, this roll-off effect can be greatly reduced or eliminated in certain embodiments of the present invention. Figure 8 shows two additional data points shown as circles which represent the dielectric constant of thin TiO₂ layers for layer 104 with Ti-Ti₄O₇ deposited as layer 102 of Figure 3B.

Example 3. Deposition of TiO₂ on Ti-TiO_x (x<2) film Stack

[0072] A layer of TiO₂ was deposited on a titanium coated substrate. About 2000 Å of Ti metal was deposited at 7KW of PDC target power, with Ar flow of 100 sccm and bias power of 200W. After Ti deposition, TiO₂ was deposited in the same chamber without oxide burn in. This process resulted in a Ti-TiO_y-TiO₂ (y<2) film stack. The k value of a 200Å film was as high as 60.

[0073] Figures 9A and 9B illustrate an SEM cross-section of a Ti₄O₇ Ebonex™ target (Figure 9A) and an SEM cross section of the Ti₄O_{6.8} layer (Figure 9B) deposited from the Ebonex™ target according to the present invention. The deposited film shows smooth deposition of the layer. The Ebonex™ target shown in Figure 9A shows an open porosity material with high roughness. The deposited layer shown in Figure 9B, however, shows a highly dense layer with a smooth surface condition.

[0074] Table I shows the effects of the dielectric properties of TiO₂ deposited according the present invention in comparison with previously obtained values. The values for the previously obtained reactive sputtering was taken from the paper "Frequency-Dependent Pulsed Direct Current magnetron Sputtering of Titanium Oxide Films," by J. Y. Kim et al., J. Vac. Sci. Techn., A 19(2), Mar/Apr. 2001. The values for PDC PVD with bias was experimentally obtained from layers deposited as described in Example 2 above.

Table I

Process	V _{bd} (Mv/cm)	K	FM
Reactive Sputtering	0.46 ~ 1.35	34 ~ 65.9	19 ~ 50
PDC physical Vapor Deposition with Bias	3.48	83	288

[0075] As can be seen from Table I, the breakdown voltage V_{bd} is significantly improved in layers according to the present invention. Further, the dielectric constant of the resulting layer is also higher. The figure of merit (FM) then for the deposited

layer was 288, very much higher than that report by Kim et al. The reference Kim et al. was the reference reporting the best quality TiO₂ films available at the time of filing of the prior application to which this disclosure claims priority.

[0076] Figure 10 shows data of capacitance made with layers according to the present invention in processes as described in Example 2 above are shown in comparison with available industry values. As is observed in Figure 10, layers of TiO₂ deposited according to the present invention have higher dielectric breakdown voltages than other dielectric films utilized in industry, which is represented by the solid line. However, due to the roll-off in dielectric constant K in films below about 1000 Å in thickness (as is indicated in the top two points in Figure 10), a capacitance density above about 5000 or 6000 pF/mm² could not be achieved using thinner films. This is also shown in Figure 7.

[0077] However, combined with the use of a conductive sub-oxide and the higher dielectric constant of thinner films as shown in Figure 11, a capacitance density of 12000 pF/mm² can be achieved with a 500 Å thickness film and a capacitance density of greater than 24000 pF/mm² can be achieved with a 220 Å film, as is shown in Figure 11. These film stacks were deposited as described in Example 3 above.

[0078] Figure 12 shows a deposited layer 102 on a substrate 101 formed of n++ silicon wafer. Layer 102 is formed of TiO₂ deposited according to the present invention. As shown in the SEM cross-section, the TiO₂ layer shows several layers. A layer 1201 is formed of SiO₂ formed on substrate 101 and is formed about 20 Å thick. An amorphous layer 1202 of thickness about 250 Å is then formed above layer 1201. Finally, a crystalline TiO₂ layer 1203 is formed about 4000 Å thick. In some embodiments of the present invention, a continuous deposition on a substrate results in a first amorphous layer deposited at initially cooler temperature followed by a further crystalline layer deposited during the increased temperature of the process. A diffraction pattern inset in Figure 12 illustrates the crystalline nature of layer 1203.

[0079] Table II tabulates data taken from a number of bi-layer film such as that shown in Figure 12 and completely amorphous films formed by repeated initial deposition layers at cool deposition conditions. Films near 1000 Å of thickness are compared and display similar values for the dielectric constant. However, the

amorphous film exhibits much higher dielectric breakdown strengths. Due to the similar thickness and values of the dielectric constant, the two films exhibit similar values for capacitance. However, the amorphous film illustrates superior breakdown voltage and therefore has a higher figure of merit (FM). These trends are more pronounced in the thicker films with thicknesses close to 2000 Å. In this case, the values of the dielectric constant and capacitance are nearly identical but again there is a significantly higher breakdown voltage in the amorphous film, which results in a significant improvement in the figure of merit for the amorphous films.

Table II

Film Thickness (nm)	k	V _{bd} (MV/cm)	FM	C (pF/mm ²)	Breakdown Voltage (V)	Film Morphology
969	63	3.6	227	540	348	Bi-layer
1036	62	6.4	396	538	660	Amorphous
2020	98	3.5	335	429	705	Bi-Layer
2322	98	5.5	539	429	1110	Amorphous

[0080] Therefore, it is clear that amorphous TiO₂ films have much better performance. As discussed above, those layers are the result of low temperature depositions. Therefore, as was demonstrated with the data shown in Table II, one method of producing thick amorphous TiO₂ layers is to simply utilize a sequence of low temperature depositions, halting the deposition prior to thermal heating of the depositing film. However, this method can take a significant amount of production time for thick films. Another embodiment of obtaining thick TiO₂ amorphous films is to apply active cooling to the substrate in an amount sufficient to provide continuously amorphous TiO₂ films.

[0081] Figure 13 shows a comparison of the leakage current for TiO₂ films according to embodiments of the present invention with and without erbium ion doping. The lower data points in Figure 13 are from capacitors formed from films deposited from a 10 at. % Er doped TiO target. The target was electrically

conductive. One example of the 10% doped film of 1000 Å thickness was formed with 60 sccm Ar, 6 sccm O₂, with a target power of 3 kW, bias power of 100 W, with a deposition time of 200 sec on a metal coated glass wafer. With the metal coating forming a copper titanium lower electrode and a titanium copper gold upper electrode patterned as 1X1 mm, discrete capacitors was then formed. The layers corresponding to the upper data points were deposited from a pure titanium target with no erbium doping on a TaN substrate with an evaporated platinum upper electrode. This structure of the bottom data is illustrated in Figure 4B where, for example, layer 101 is a glass substrate, layer 103 is a copper titanium layer, layer 102 is the erbium doped TiO₂ layer, and layer 201 is a titanium copper gold layer.

[0082] As can be seen in Figure 13, the leakage current density is reduced by many orders of magnitude by addition of erbium.

[0083] Figures 14A and 14B show a photoluminescence signal with excitation at 580 nm and measurement at 1.53 μm, measured from a 5000 Å layer of 10% erbium containing TiO₂ deposited from a 10% erbium doped TiO conductive target and a photoluminescence signal measured from the same layer after a 30 minute 250 °C anneal, respectively. Table III shows similar data for several layers deposited from the erbium-doped TiO conductive target.

Table III

Thickness	Before Anneal	Anneal (°C)	After Anneal
5000 Å	6704	150	5809
5000 Å	6493	200	4042
5000 Å	6669	250	2736
5000 Å	6493	300	3983
1 μm	6884	150	6743
1 μm	5197	200	3685
1 μm	6253	250	3612
1 μm	5324	300	3381

[0084] According to some explanations of the reduction of leakage current in layers as illustrated by Figure 13, fast electrons that have sufficient energy to excite

the erbium ion would cause the rare earth ion to undergo excitation upon electron impact or passage within a distance sufficient for energy exchange. Consequently, the leakage current electrons capable of causing ionization within the dielectric oxide would be reduced by electron collisions with erbium ions. Excited state ions have at least two relaxation mechanisms for disposal of the energy: radiative and non-radiative. In radiative relaxation, the excited ion emits light. In non-radiative relaxation, the excited ion undergoes a cooperative process with vibrational modes of its host dielectric oxide and produces a vibration which is the elemental form of heat. In the data illustrated in Figure 13, it was not possible to observe light in the leakage test, but photoluminescence was observed from optical excitation of the similar 10% Er doped TiO₂ deposited from the 10% Er doped TiO conductive target, as shown in Table III.

[0085] As can be seen from the data in Table III, an erbium doped layer of titanium oxide was shown to fluoresce strongly under optical excitation by light of a wavelength 580 nm, using a Phillips PhotoLuminescence Microscope, model no. PLM-100. The target was electrically conductive and sputtered at a higher rate and a lower oxygen partial pressure than characteristic of a metallic titanium target. One example of the 10% doped film of 2,032 angstroms was 60 sccm Ar, 6 sccm O₂, with a target power of 3 kW, bias power of 100 W, with a deposition time of 300 sec.

[0086] The level of photoluminescence observed from the layer was similar to that obtained in as-deposited and annealed films providing commercial levels of optical absorption and fluorescence for applications to planar waveguide amplifiers having at least 15 dB gain for signals as weak as -40dB at the 1.5 micron wavelength utilized for photonic C band communications.

[0087] Such a device can be illustrated with Figure 3B, where layer 103 can be a conductive layer deposited on substrate 101, layer 102 can be a rare-earth doped TiO₂ layer deposited according to embodiments of the present invention, and layer 104 can be a further conductive layer or a conductive transparent layer to form an metal-insulating-metal (MIM) capacitor structure. Such a structure could function as a light emitting layer under either DC or AC electrical excitation. In another embodiment, layer 103 can be a lift-off layer such as CaF₂ or other organic material, layer 102 is

the rare-earth doped TiO₂ layer, and layer 104 is absent, then upon lift-off or upon transfer of layer 102, a free standing or applied layer having electroluminescent or photoluminescent applications can be provided over a selected device.

[0088] Thin films according to the present invention can be utilized in advanced display devices, electrical energy storage and conversion, and to form optical and electronic films with scratch resistance and barrier properties. Advanced display product applications include OLED encapsulation, barriers for flexible polymer substrates, outcoupling mirrors and anti-reflection coatings, transparent conducting oxides, and semiconducting materials for active matrix displays. Electrical energy storage and conversion applications include high density capacitor arrays for mobile communication devices, on-chip high "K" capacitors for advanced CMOS, and high voltage energy storage for portable power devices. Other applications include touch-sensitive devices and durable bar code scanners and see-through sensors as well as implantable biometric devices.

[0089] The embodiments described in this disclosure are examples only and are not intended to be limiting. Further, the present invention is not intended to be limited by any particular theory or explanation presented to explain experimental results. As such, examples of titanium oxide and titanium sub-oxide films illustrated herein and their applications are not intended to be limiting. One skilled in the art may contemplate further applications or films that are intended to be within the spirit and scope of the present invention. As such, the invention is limited only by the following claims.

Claims

We claim:

1. A method of forming a titanium based layer, comprising:
depositing a layer of titanium containing oxide by pulsed-DC, biased reactive sputtering process on a substrate.
2. The method of claim 1, wherein the layer is TiO₂.
3. The method of claim 2, wherein the figure of merit of the layer is greater than 50.
4. The method of claim 2, wherein the layer is deposited between conducting layers to form a capacitor.
5. The method of claim 2, wherein the layer includes at least one rare-earth ion.
6. The method of claim 5, wherein the layer is deposited between conducting layers to form a capacitor.
7. The method of claim 5, wherein the at least one rare-earth ion includes erbium.
8. The method of claim 5, wherein the layer is deposited between conducting layers to form a light-emitting device.
9. The method of claim 5, wherein the layer is an optically active layer deposited on a light-emitting device.
10. The method of claim 5, wherein the layer is an optically active layer applied to a light-emitting device.
11. The method of claim 1, wherein the layer is a sub-oxide of Titanium.
12. The method of claim 11, wherein the figure of merit of the layer is greater than 50.
13. The method of claim 11, wherein the layer is deposited between conducting layers to form a capacitor.
14. The method of claim 11, wherein the layer includes at least one rare-earth ion.
15. The method of claim 14, wherein the layer is deposited between conducting layers to form a capacitor.
16. The method of claim 14, wherein the at least one rare-earth ion includes erbium.
17. The method of claim 14, wherein the layer is deposited between conducting layers to form a light-emitting device.

18. The method of claim 14, wherein the layer is an optically active layer deposited on a light-emitting device.
19. The method of claim 14, wherein the layer is an optically active layer applied to a light-emitting device.
20. The method of claim 2, wherein the layer is a protective layer.
21. The method of claim 20, wherein the protective layer is a catalytic layer.
22. The method of claim 20, wherein the protective layer includes at least one rare-earth ion.
23. The method of claim 1, wherein the layer is Ti_xO_y wherein x is between about 1 and about 4 and y is between about 1 and about 7.
24. The method of claim 23, wherein the figure of merit of the layer is greater than 50.
25. The method of claim 23, further including depositing an TiO_2 layer on the layer wherein the layer and the TiO_2 layers are deposited between conducting layers to form a capacitor with decreased roll-off characteristics with decreasing thickness of the TiO_2 layer.
26. The method of claim 23, wherein the TiO_2 layer is an amorphous layer deposited by a pulsed DC, biased, reactive ion process.
27. The method of claim 23, wherein the layer includes at least one rare-earth ion.
28. The method of claim 27, wherein the at least one rare-earth ion includes erbium.
29. The method of claim 27, wherein the layer is deposited between conducting layers to form a light-emitting device.
30. The method of claim 27, wherein the layer is an optically active layer deposited on a light-emitting device.
31. The method of claim 27, wherein the layer is an optically active layer applied to a light-emitting device.
32. The method of claim 23, wherein the layer is a conducting oxide.
33. The method of claim 32, wherein the substrate is a conducting electrode and the layer is a protective layer.
34. The method of claim 33, wherein the protective layer is a catalytic layer.

35. The method of claim 33, wherein the protective layer includes at least one rare-earth ion.
36. The method of claim 32, wherein the substrate is a dielectric and the layer is a protective layer.
37. The method of claim 36, wherein the protective layer is a catalytic layer.
38. The method of claim 1, further including
 - controlling the temperature of the substrate during deposition.
39. The method of claim 38, wherein controlling the temperature includes active temperature control.
40. The method of claim 1, wherein the layer is an amorphous layer.
41. The method of claim 1, wherein the substrate includes a transistor structure.
42. A titanium based layer, comprising:
 - a layer compounded from titanium and oxygen deposited by pulsed-DC, biased reactive sputtering process on a substrate.
43. The layer of claim 42, wherein the layer is TiO_2 .
44. The layer of claim 43, wherein the figure of merit of the layer is greater than 50.
45. The layer of claim 43, wherein the layer is deposited between conducting layers to form a capacitor.
46. The layer of claim 43, wherein the layer includes at least one rare-earth ion.
47. The layer of claim 46, wherein the layer is deposited between conducting layers to form a capacitor.
48. The layer of claim 46, wherein the at least one rare-earth ion includes erbium.
49. The layer of claim 46, wherein the layer is deposited between conducting layers to form a light-emitting device.
50. The layer of claim 46, wherein the layer is an optically active layer deposited on a light-emitting device.
51. The layer of claim 46, wherein the layer is an optically active layer applied to a light-emitting device.
52. The layer of claim 42, wherein the layer is sub-oxide of Titanium.
53. The layer of claim 52, wherein the figure of merit is greater than 50.

54. The layer of claim 52, wherein the layer is deposited between conducting layers to form a capacitor.
55. The layer of claim 52, wherein the layer includes at least one rare-earth ion.
56. The layer of claim 55, wherein the layer is deposited between conducting layers to form a capacitor.
57. The layer of claim 55, wherein the at least one rare-earth ion includes erbium.
58. The layer of claim 55, wherein the layer is deposited between conducting layers to form a light-emitting device.
59. The layer of claim 55, wherein the layer is an optically active layer deposited on a light-emitting device.
60. The layer of claim 55, wherein the layer is an optically active layer applied to a light-emitting device.
61. The layer of claim 43, wherein the layer is a protective layer.
62. The layer of claim 61, wherein the protective layer is a catalytic layer.
63. The layer of claim 61, wherein the protective layer includes at least one rare-earth ion.
64. The layer of claim 42, wherein the layer is Ti_xO_y wherein x is between about 1 and about 4 and y is between about 1 and about 7.
65. The layer of claim 64, wherein the figure of merit is greater than 50.
66. The layer of claim 64, further including depositing an TiO_2 layer on the layer wherein the layer and the TiO_2 layers are deposited between conducting layers to form a capacitor with decreased roll-off characteristics with decreasing thickness of the TiO_2 layer.
67. The layer of claim 64, wherein the TiO_2 layer is an amorphous layer deposited by a pulsed DC, biased, reactive ion process.
68. The layer of claim 64, wherein the layer includes at least one rare-earth ion.
69. The layer of claim 68, wherein the at least one rare-earth ion includes erbium.
70. The layer of claim 68, wherein the layer is deposited between conducting layers to form a light-emitting device.
71. The layer of claim 68, wherein the layer is an optically active layer deposited on a light-emitting device.

72. The layer of claim 68, wherein the layer is an optically active layer applied to a light-emitting device.
73. The layer of claim 64, wherein the layer is a conducting oxide.
74. The layer of claim 73, wherein the substrate is a conducting electrode and the layer is a protective layer.
75. The layer of claim 74, wherein the protective layer is a catalytic layer.
76. The layer of claim 74, wherein the protective layer includes at least one rare-earth ion.
77. The layer of claim 73, wherein the substrate is a dielectric and the layer is a protective layer.
78. The layer of claim 77, wherein the protective layer is a catalytic layer.
79. The layer of claim 42, further including
controlling the temperature of the substrate during deposition.
80. The layer of claim 79, wherein controlling the temperature includes active temperature control.
81. The layer of claim 42, wherein the substrate includes a transistor structure.
82. The layer of claim 42, wherein the layer is an amorphous layer.
83. A target, comprising:
hipped TiO having composition TiO.
84. The target of claim 83, further including at least one rare-earth dopant.
85. A method of forming a target, comprising:
forming a TiO powder;
mixing the TiO powder to form a mix;
hipping the mix under a controlled atmosphere to form tiles; and
forming a target from the tiles.
86. The method of claim 85, further including mixing at least one rare-earth oxide powder with the mix.
87. The method of claim 86, wherein the at least one rare-earth oxide includes erbium oxide.

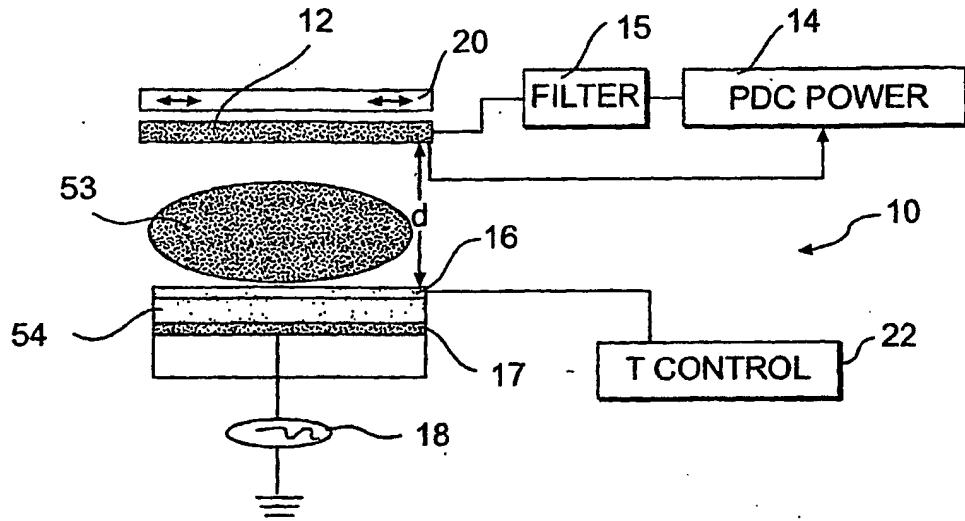


FIG. 1A

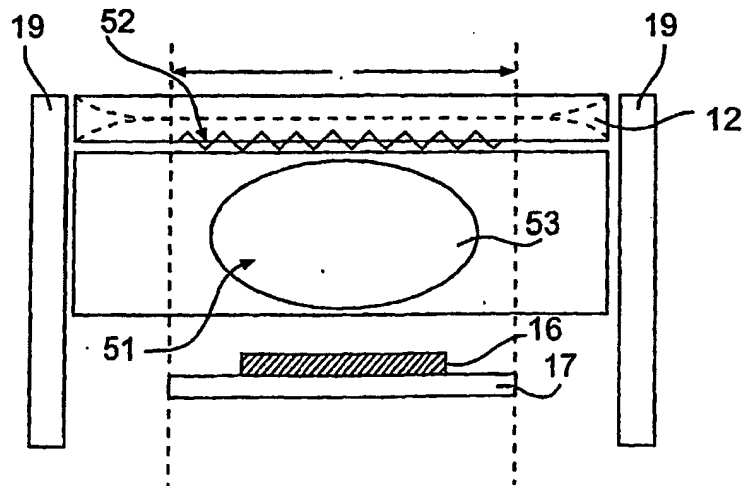


FIG. 1B

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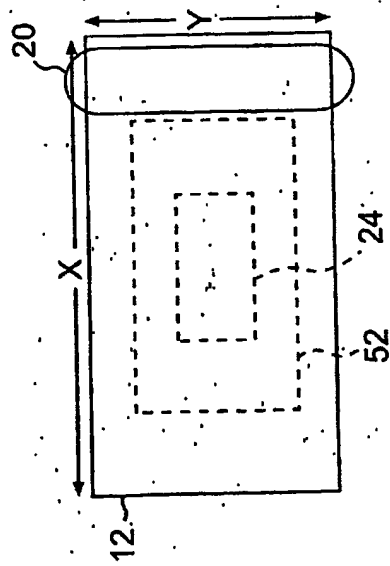


FIG. 2

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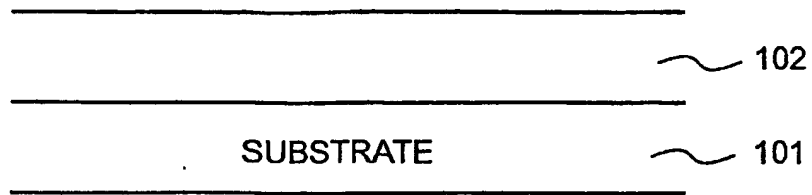


FIG. 3A

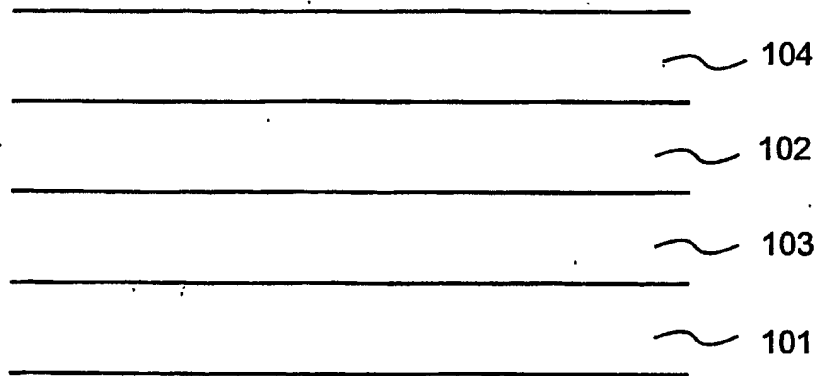


FIG. 3B

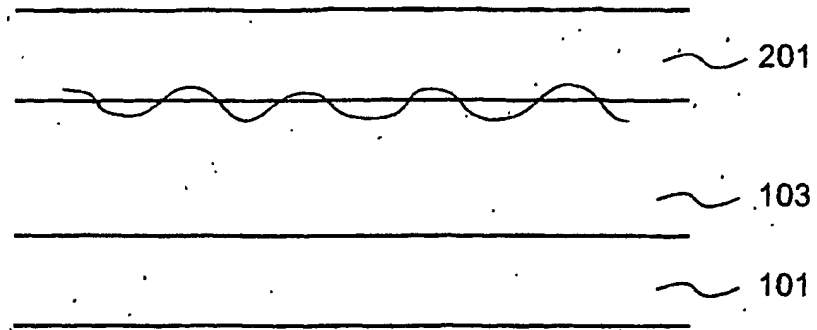


FIG. 4A

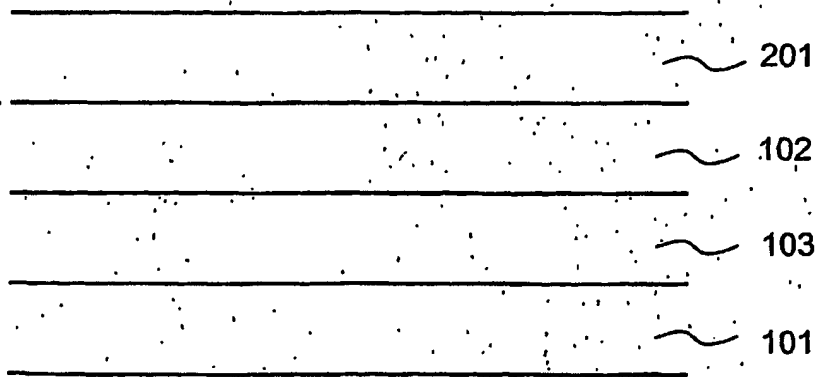


FIG. 4B

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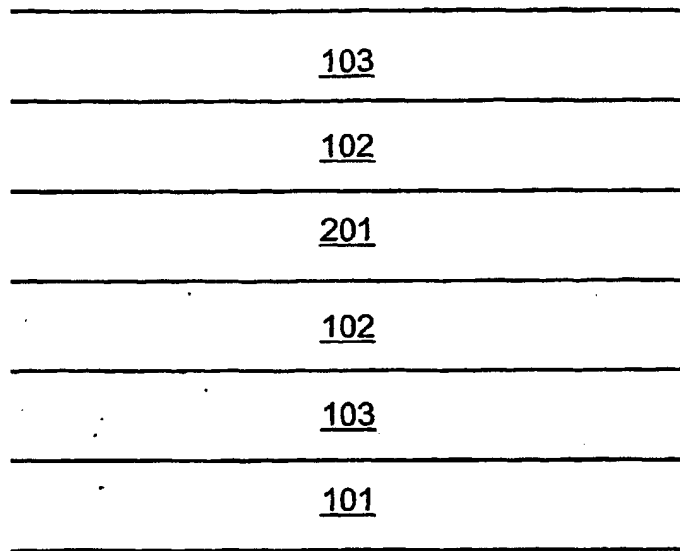


FIG. 5

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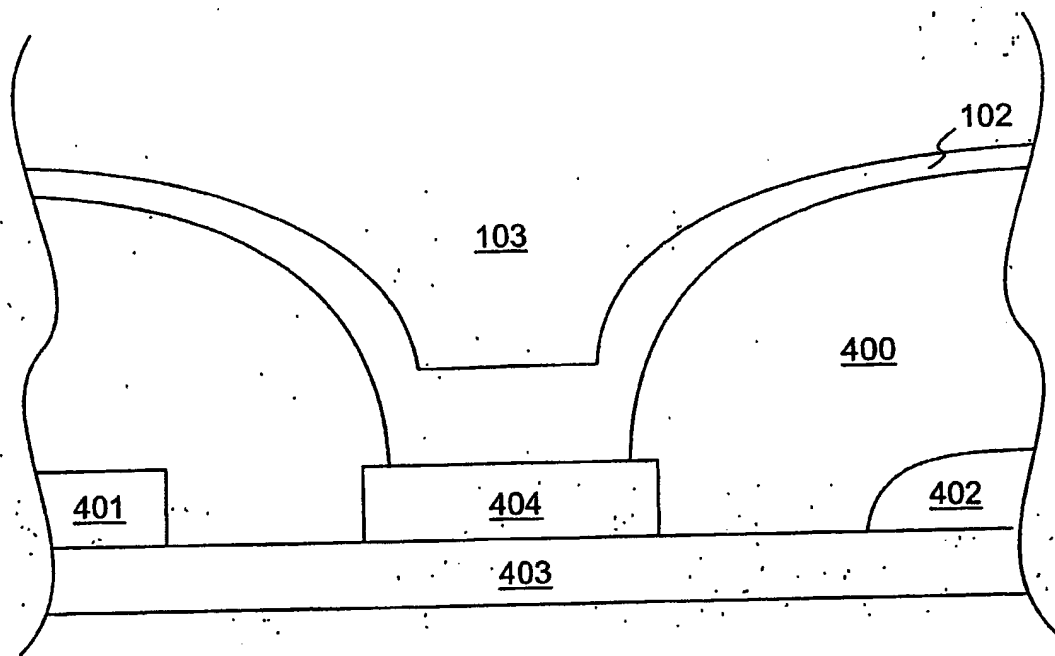


FIG. 6

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▣ k (ON Al)
▣ k (ON n++)

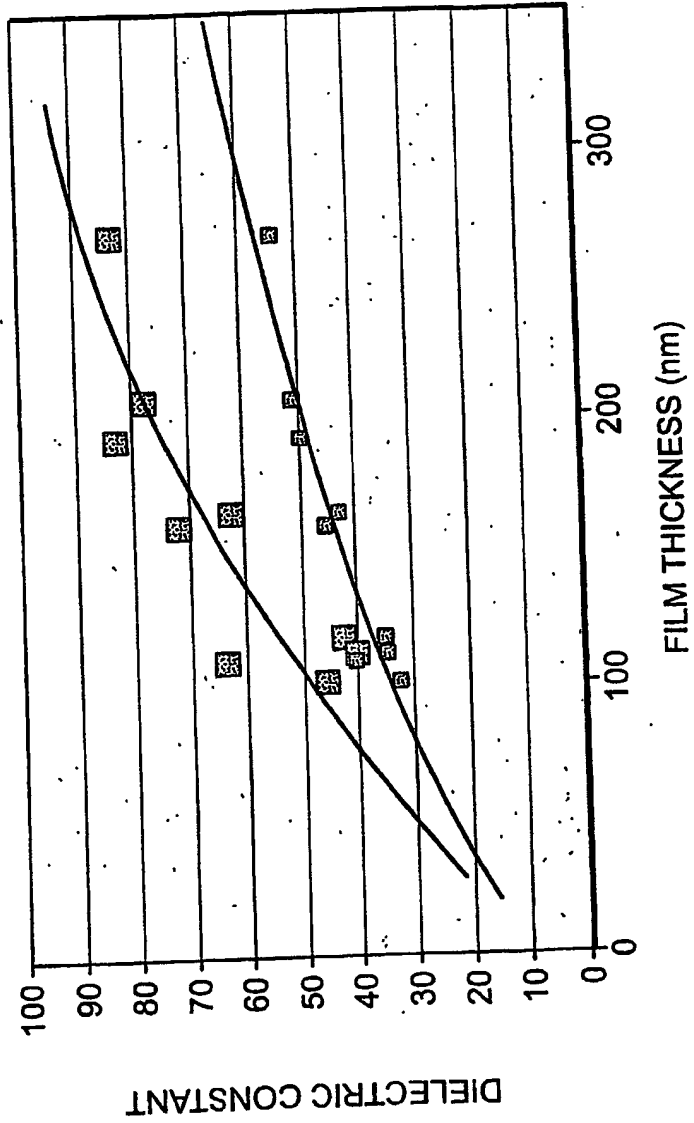


FIG. 7

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■ k (ON·Al)
■ k (ON n++)

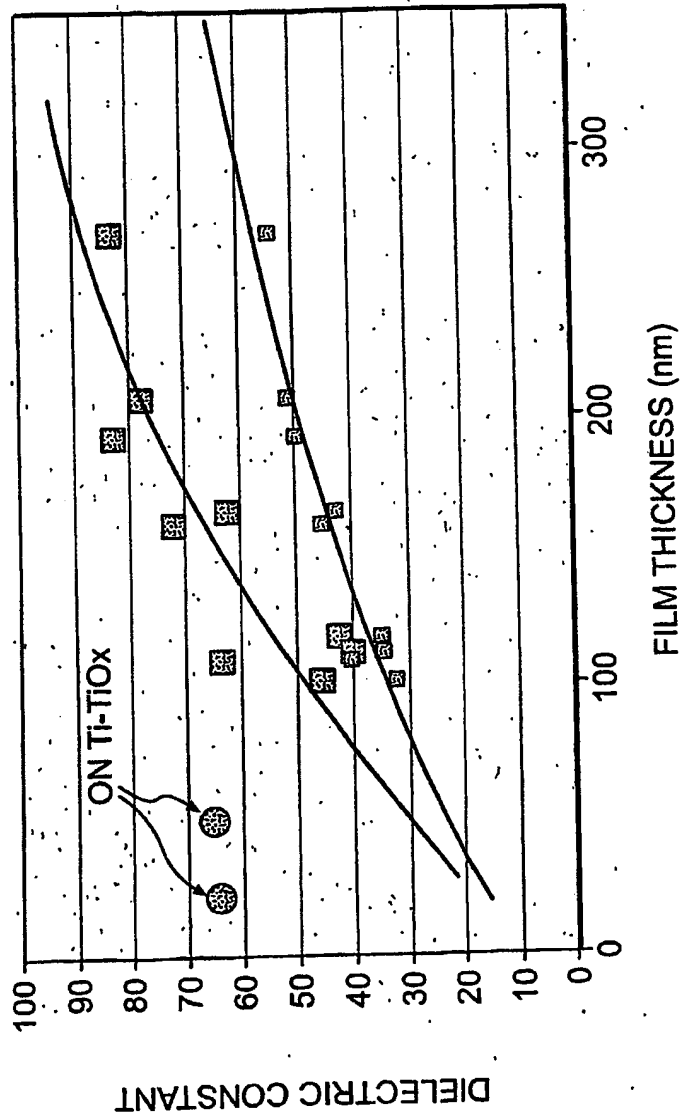


FIG. 8

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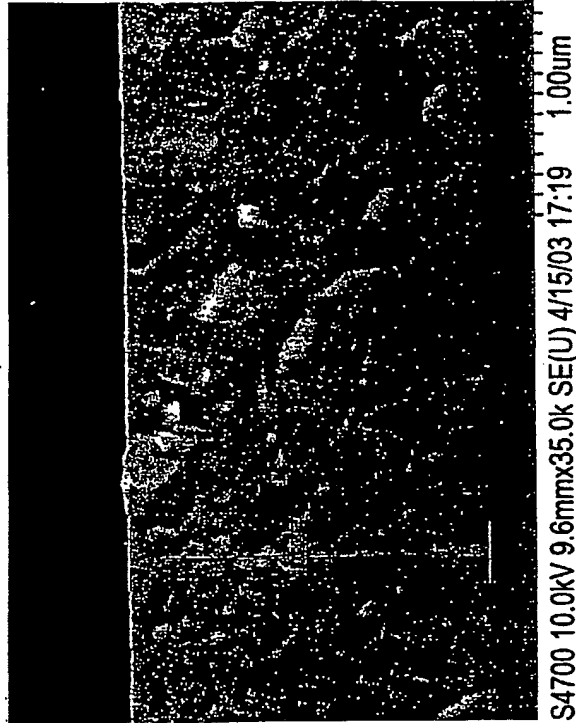


FIG. 9B

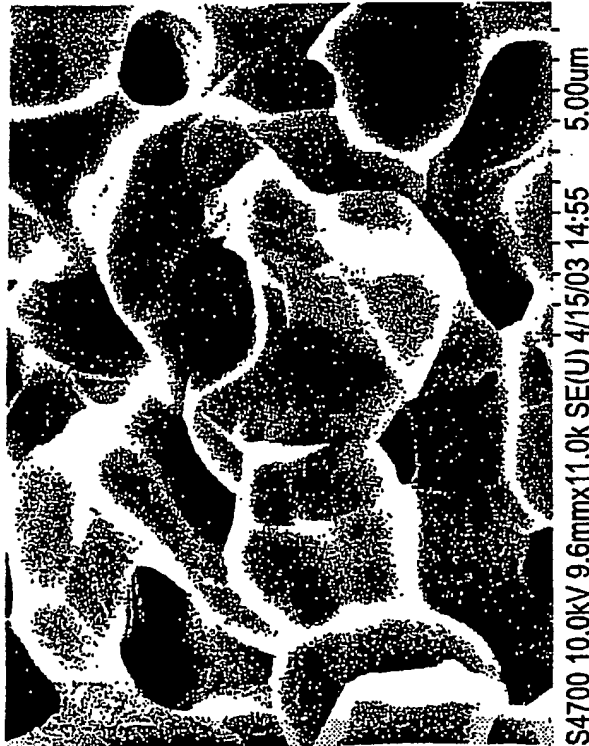


FIG. 9A

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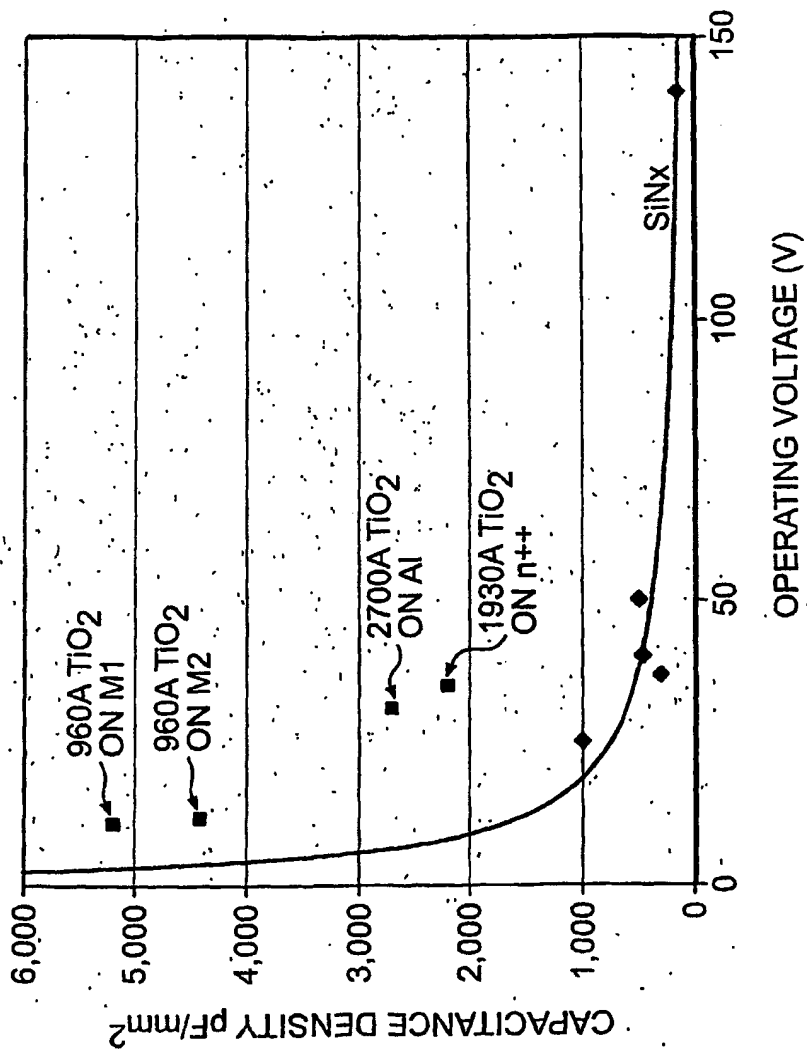


FIG. 10

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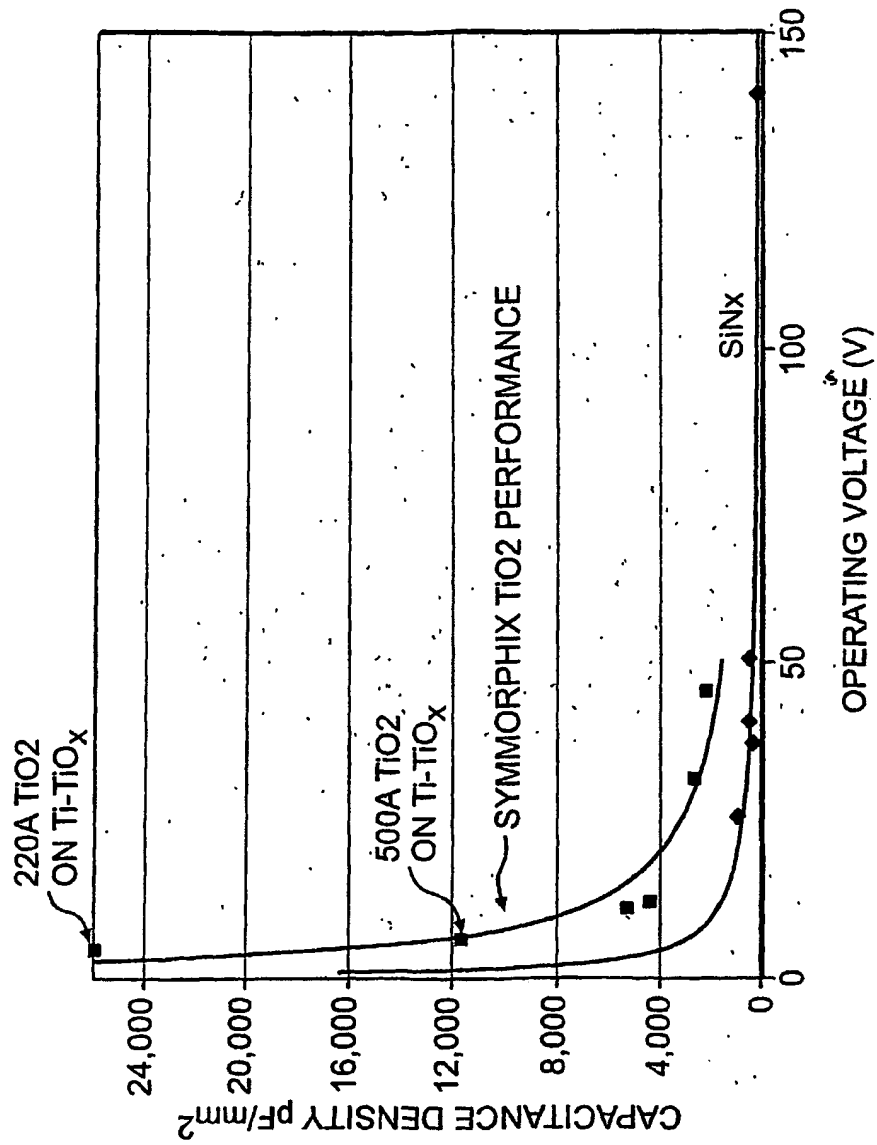


FIG. 11

12/15

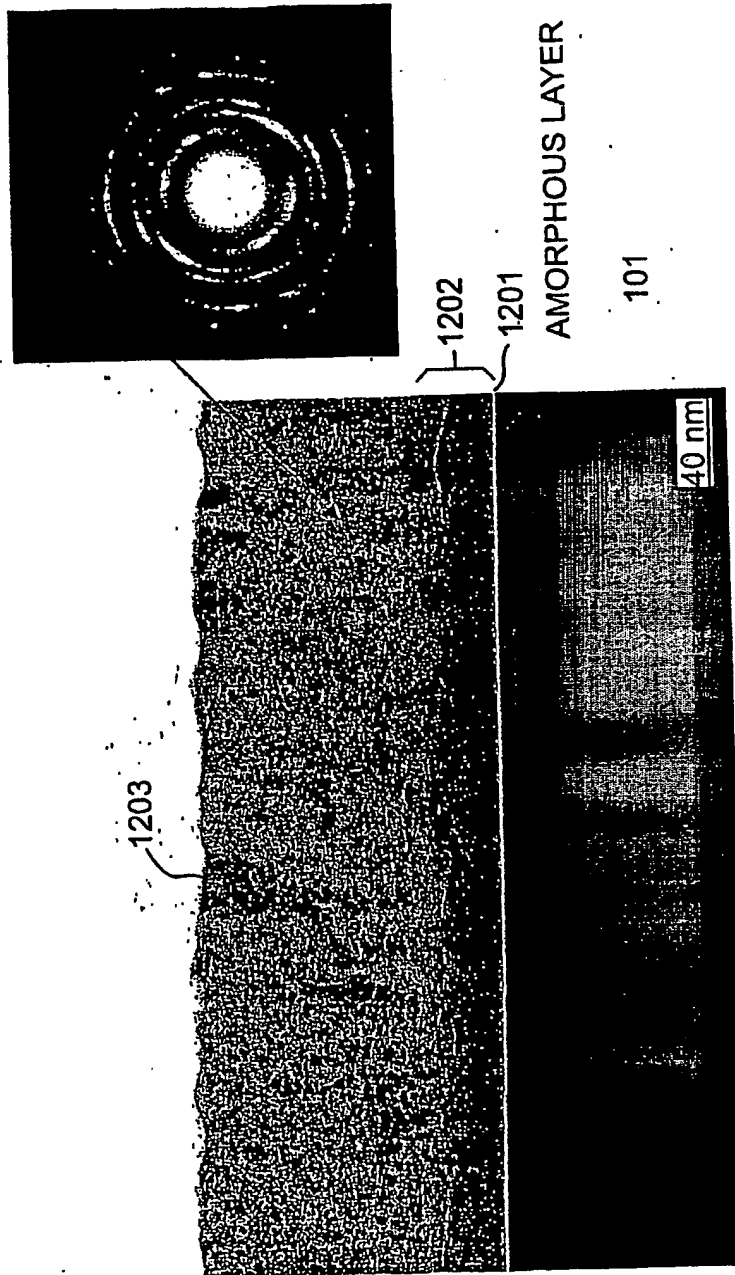


FIG. 12

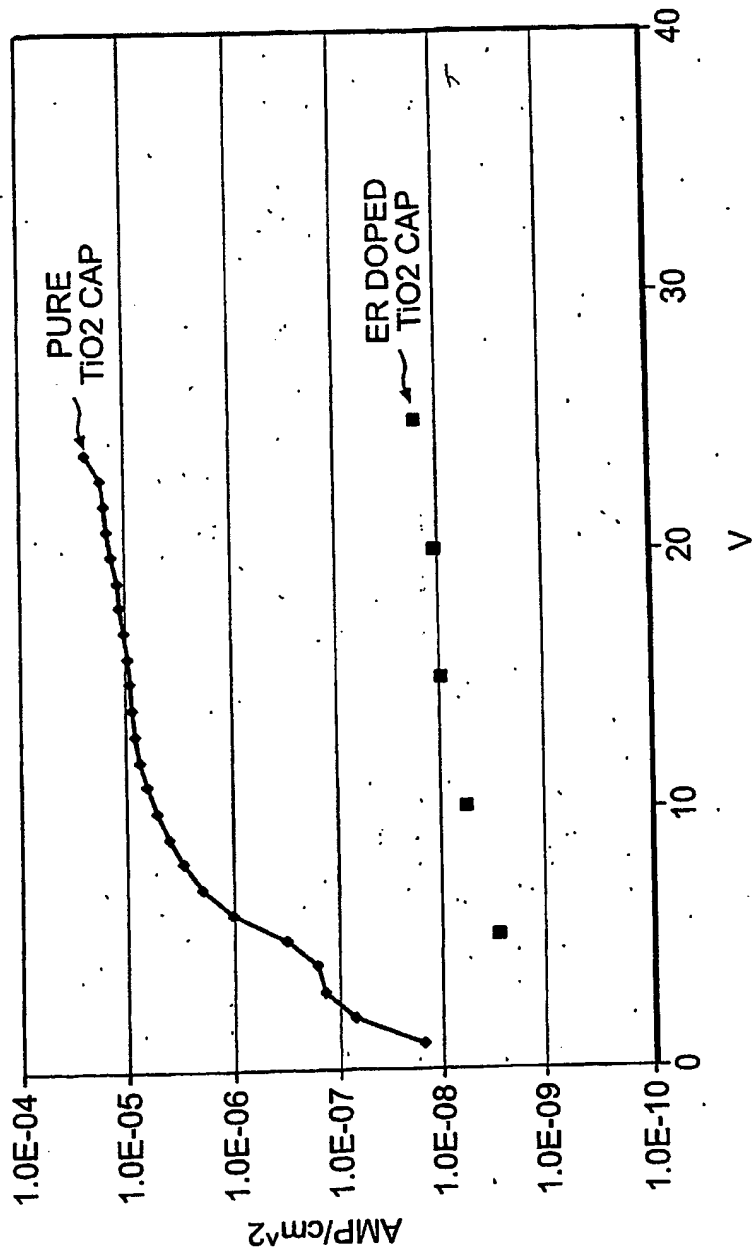


FIG. 13

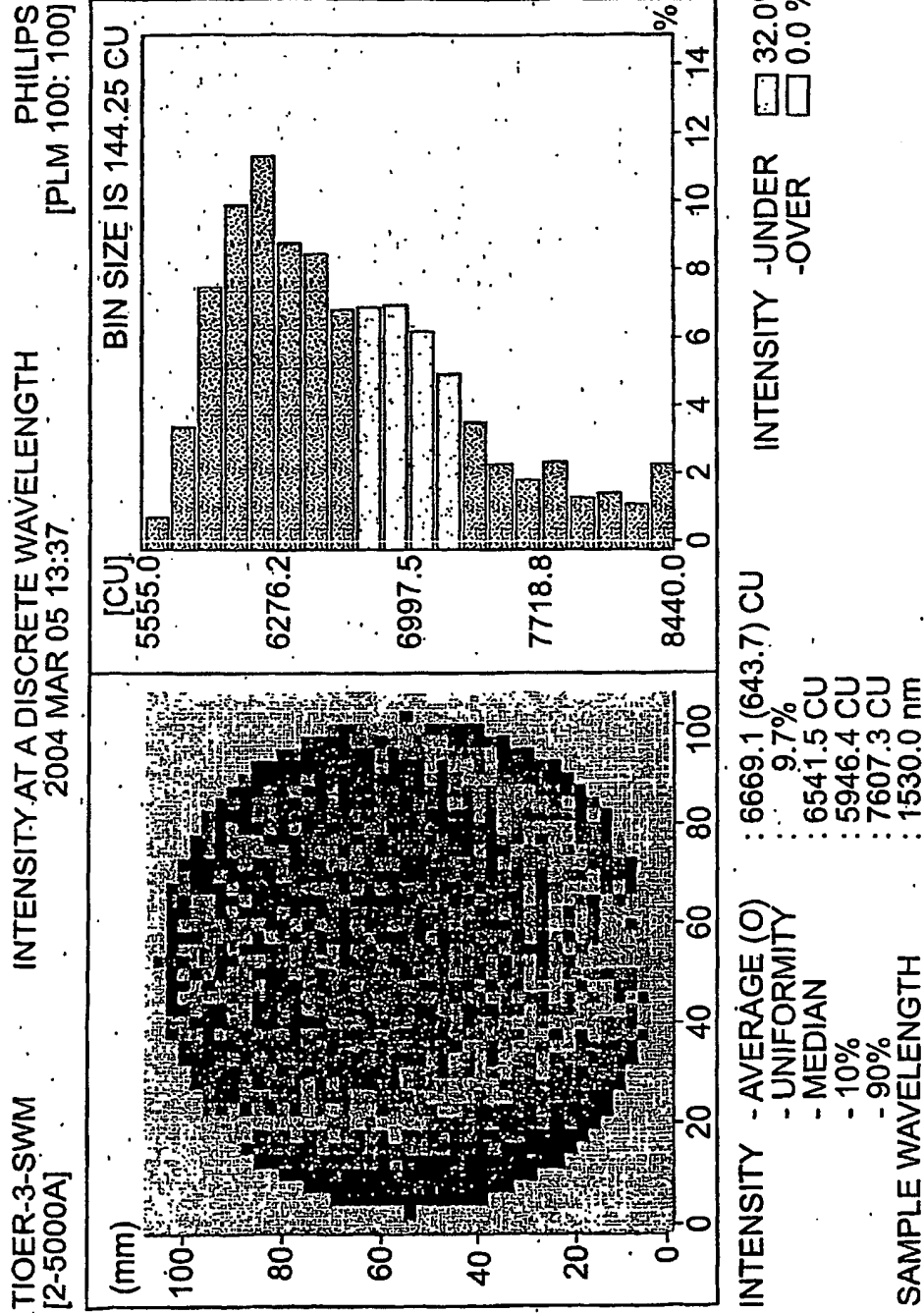
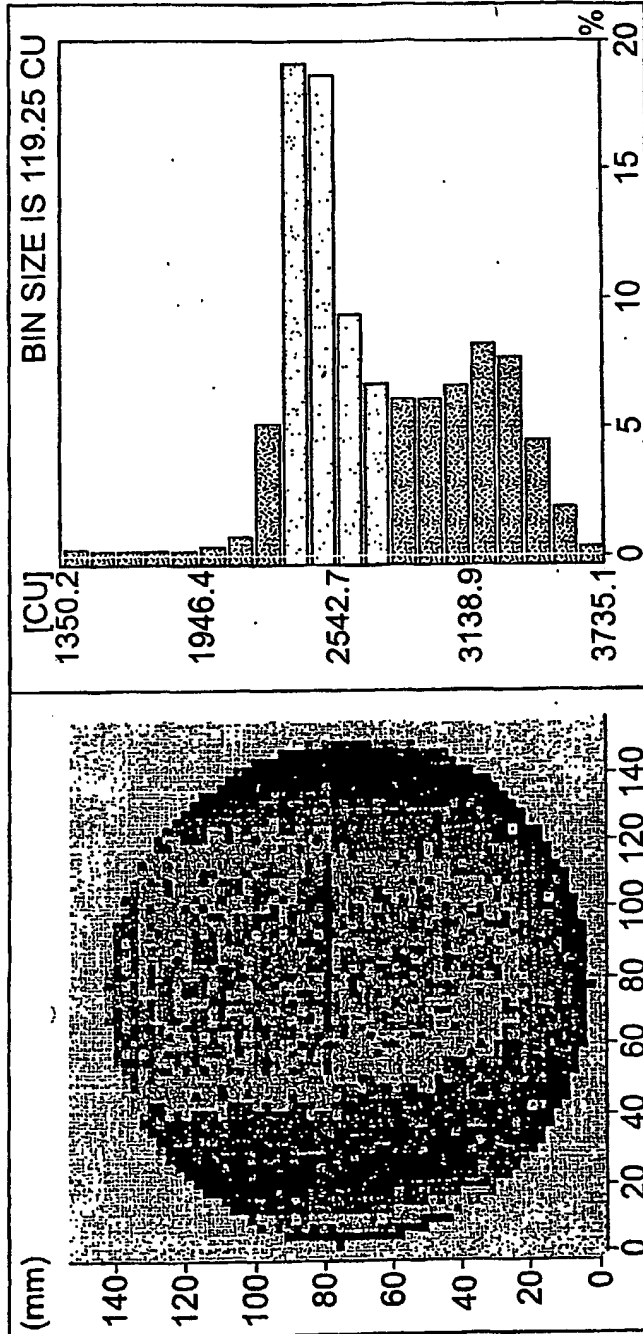


FIG. 14A

TIOER-3-SWM
[2-5000A]

INTENSITY AT A DISCRETE WAVELENGTH
2004 MAR 08 08:00

PHILIPS
[PLM 100: 100]



INTENSITY - AVERAGE (O)
 - UNIFORMITY : 2736.0 (382.3) CU
 - MEDIAN : 14.0 %
 - 10% : 2615.1 CU
 - 90% : 2330.0 CU

INTENSITY - UNDER : 34.9%
 INTENSITY - OVER : 0.0%

INTENSITY - AVERAGE (O)
 - UNIFORMITY : 2736.0 (382.3) CU
 - MEDIAN : 14.0 %
 - 10% : 2615.1 CU
 - 90% : 2330.0 CU

SAMPLE WAVELENGTH : 1530.0 nm

FIG. 14B

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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/954,182	10/01/2004	Hongmei Zhang	09140-0016-01000	9873

22852 7590 03/09/2006

FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER
LLP
901 NEW YORK AVENUE, NW
WASHINGTON, DC 20001-4413

EXAMINER

ESTRADA, MICHELLE

ART UNIT PAPER NUMBER

2823

DATE MAILED: 03/09/2006

Please find below and/or attached an Office communication concerning this application or proceeding.

Office Action Summary

Application No. 10/954,182	Applicant(s) ZHANG ET AL.	
Examiner Michelle Estrada	Art Unit 2823	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

- 1) Responsive to communication(s) filed on 21 December 2005.
- 2a) This action is **FINAL**.
- 2b) This action is non-final.
- 3) Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims

- 4) Claim(s) 41-43, 45-62, 85 and 87-89 is/are pending in the application.
4a) Of the above claim(s) _____ is/are withdrawn from consideration.
- 5) Claim(s) 61, 62 and 85 is/are allowed.
- 6) Claim(s) 41-43 and 45-60 is/are rejected.
- 7) Claim(s) 87-89 is/are objected to.
- 8) Claim(s) _____ are subject to restriction and/or election requirement.

Application Papers

- 9) The specification is objected to by the Examiner.
- 10) The drawing(s) filed on 21 December 2005 is/are: a) accepted or b) objected to by the Examiner.
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

Priority under 35 U.S.C. § 119

- 12) Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
a) All b) Some * c) None of:
1. Certified copies of the priority documents have been received.
2. Certified copies of the priority documents have been received in Application No. _____.
3. Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

* See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

- 1) Notice of References Cited (PTO-892)
- 2) Notice of Draftsperson's Patent Drawing Review (PTO-948)
- 3) Information Disclosure Statement(s) (PTO-1449 or PTO/SB/08)
Paper No(s)/Mail Date 7/25/05, 12/21/05.
- 4) Interview Summary (PTO-413)
Paper No(s)/Mail Date. _____.
- 5) Notice of Informal Patent Application (PTO-152)
- 6) Other: _____.

DETAILED ACTION

Claim Rejections - 35 USC § 103

The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

Claims 45, 47, 49, 51, 52, 59 and 60 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff et al. (6,117,279) in view of D'Couto et al. (6,673,716).

Re claims 59 and 60, Smolanoff et al. disclose providing a process gas between the target (16) and a substrate (15); providing pulsed DC power to the target (Col. 5, lines 50-55); providing a magnetic field to the target (Col. 6, lines 1-7); and wherein a material is deposited on the substrate (Col. 5, lines 22-26); and an oxide film is formed by reactive sputtering (Col. 6, lines 15+).

Smolanoff et al. do not disclose wherein the material sputtered is in the metallic mode or in the poison mode.

D'Couto et al. disclose that a material can be sputter neither in the poison mode or metallic mode (Col. 1, line 65-Col. 2, line 20).

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff et al. and D'Couto et al. to enable the sputtering mode step of Smolanoff et al. to be performed according to the teachings of D'Couto et al. because

one of ordinary skill in the art would have been motivated to look to alternative suitable methods of performing the disclosed sputtering step of Smolanoff et al. and art recognized suitability for an intended purpose has been recognized to be motivation to combine. See MPEP 2144.07.

Re claim 45, Smolanoff et al. disclose wherein the magnetic field is provided by a moving magnetron (Col. 5, lines 39-49).

Re claim 47, Smolanoff et al. disclose wherein the process gas includes a mixture of oxygen and argon (Col. 7, lines 22-27).

Re claim 49, Smolanoff et al. disclose wherein the process gas further includes nitrogen (Col. 7, lines 25-26).

Re claim 51, Smolanoff et al. disclose further including uniformly sweeping the target with a magnetic field (Col. 6, lines 1-6).

Re claim 52, Smolanoff et al. disclose wherein sweeping the target with a magnetic field includes sweeping a magnet in one direction across the target where the magnet extends beyond the target in the opposite direction (Col. 6, lines 1-6).

Claims 41, 42, 46, 48 and 50 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff in view of D'Couto et al. as applied to claims 45, 47, 49, 51, 52, 59 and 60 above, and further in view of Chen et al. (2004/0077161).

Re claim 41, Smolanoff et al. disclose wherein the process gas includes oxygen.

Re claim 42, Smolanoff et al. disclose wherein the process gas includes N_2 or NH_3 .

The combination of Smolanoff et al. and D'Couto et al. does not disclose wherein the target is a metallic target.

Re claims 41 and 42, Chen et al. disclose forming a coating layer in a substrate; applying a magnetic field to a target; wherein the target is a metallic target.

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff et al., D'Couto et al. and Chen et al. to enable the target material of Smolanoff et al. to be the same according to the teachings of Chen et al. because one of ordinary skill in the art would have been motivated to look to alternative suitable target materials of the disclosed target of Smolanoff et al. and art recognized suitability for an intended purpose has been recognized to be motivation to combine. See MPEP 2144.07.

Re claim 46, Chen et al. disclose further including holding the temperature of the substrate substantially constant (Page 3, Paragraph [0046]).

Re claim 48, Chen et al. disclose wherein the oxygen flow is adjusted by the mass flow controllers; thereby it will adjust the index refraction of the film.

Re claim 50, Chen et al. disclose wherein providing pulsed DC power to a target includes providing pulsed DC power to a target, which has an area larger than that of the substrate (See fig. 3).

Art Unit: 2823

Claims 43 and 53-58 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff et al. in view of D'Couto et al. as applied to claims 45, 47, 49, 51, 52, 59 and 60 above, and further in view of Milonopoulou et al. (2003/0175142).

The combination of Smolanoff et al. and D'Couto et al. does not disclose wherein the target is a ceramic target.

Milonopoulou et al. disclose forming a coating layer on a substrate; providing a target (12), which is ceramic (Abstract).

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff et al., D'Couto et al. and Chen et al. to enable the target material of Smolanoff et al. to be the same according to the teachings of Milonopoulou et al. because one of ordinary skill in the art would have been motivated to look to alternative suitable target materials of the disclosed target of Smolanoff et al. and art recognized suitability for an intended purpose has been recognized to be motivation to combine. See MPEP 2144.07.

Re claim 53, Milonopoulou et al. disclose wherein the target is an alloyed target (Abstract).

Re claim 54, Milonopoulou et al. disclose wherein the alloyed target includes one or more rare earth ions.

Re claim 55, Milonopoulou et al. disclose wherein the alloyed target includes Si and Al.

Re claim 56, Milonopoulou et al. disclose wherein the alloyed target includes one or more elements taken from a set consisting of Si, Al, Er and Yb.

Re claim 57, Milonopoulou et al. disclose wherein the alloyed target is a tiled target.

Re claim 58, Milonopoulou et al. disclose wherein each tiled target is formed by prealloy atomization and hot isostatic pressing of a powder (Page 2, Paragraph [0020]).

Allowable Subject Matter

Claims 87-89 are objected to as being dependent upon a rejected base claim, but would be allowable if rewritten in independent form including all of the limitations of the base claim and any intervening claims.

Claims 61, 62 and 85 are allowed.

Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Michelle Estrada whose telephone number is 571-272-1858. The examiner can normally be reached on Monday through Friday.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Matthew Smith can be reached on 571-272-1907. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 571-272-2800.

Art Unit: 2823

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).



Michelle Estrada
Primary Examiner
Art Unit 2823

ME
March 6, 2006



*Approved
MR
3/6/06*

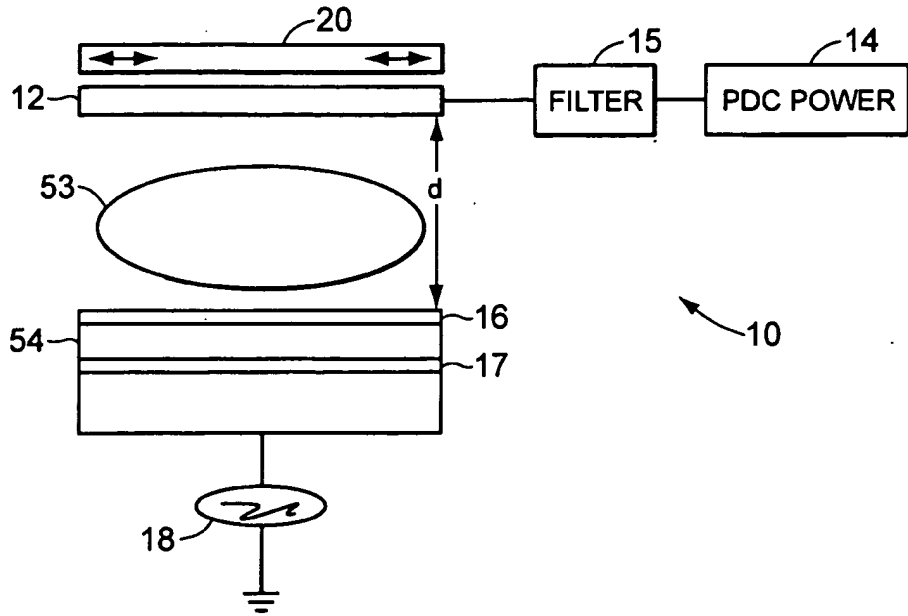


FIG. 1A

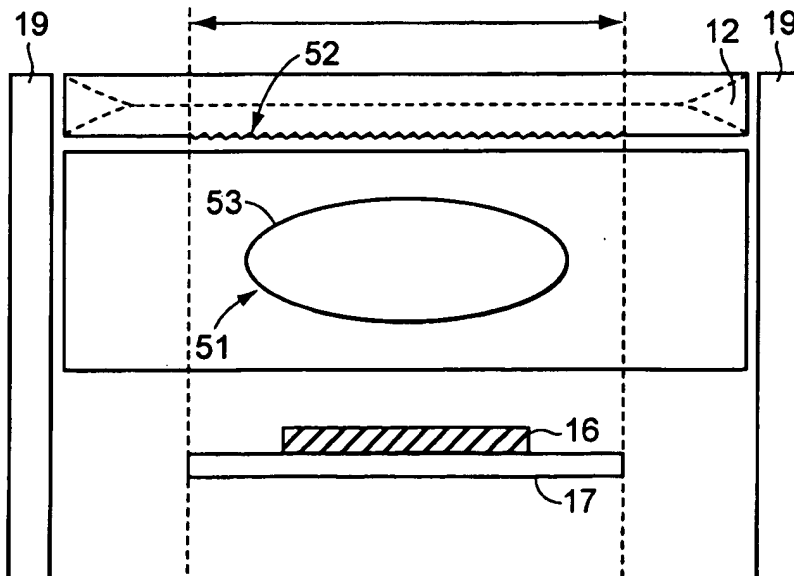


FIG. 1B





EXPRESS MAIL NO. EV 727732357 US

IDS Form PTO/SB/08: Substitute for Form 1449A/PTO INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Use as many sheets as necessary)			Complete if Known		
Sheet	1	of	2	Application Number	10/954,182
				Filing Date	October 1, 2004
				First Named Inventor	ZHANG, Hongmei
				Art Unit	2823
				Examiner Name	ESTRADA, Michelle
				Attorney Docket Number	9140.0016-01

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS

Examiner Initials	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
<i>ME</i>		US 2003/0141186 A1	07-31-2003	Wang et al.	
<i>ME</i>		US 2001/041460 A1	11-15-2001	Wiggins	
<i>ME</i>		US 2001/0027159 A1	10-04-2001	Kaneyoshi	
<i>ME</i>		US 6,232,242	05-15-2001	Hata et al.	
<i>ME</i>		US 6,117,279	09-12-2000	Smotanoff et al.	
<i>ME</i>		US 5,738,731 (09-00)	04-04-1998	Shindo	
<i>ME</i>		US 5,538,796	07-23-1996	Schaffer	
<i>ME</i>		US 5,309,302	03-14-1997 ⁵⁻²⁻⁹⁴	Heit Vollmann	
<i>ME</i>		US 5,296,089	03-22-1994	Chen et al.	
<i>ME</i>		US 5,173,271	12-22-1992	Chen et al.	
<i>ME</i>		US 4,587,225	05-06-1986	Tsukuma et al.	
<i>ME</i>		RE 32,449	06-30-1987	Claussen	

already in 892
filed 3/25/05

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

FOREIGN PATENT DOCUMENTS

Examiner Initials	Cite No. ¹	Foreign Patent Document	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁶
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)				
<i>ME</i>		EP 1 092 689 A1	04-18-2004	BPS Aizenau GmbH		
<i>ME</i>		EP 1068899 A1	01-17-2001	Nippon Sheet Glass Co., Ltd.		
<i>ME</i>		EP 0 639 655 A1	02-22-1995	Asahi Glass Co, Ltd.		
<i>ME</i>		EP 0 652 308 A2	10-13-1994	Mega Chips Corp.		
<i>ME</i>		JP 7-233469	09-05-1995	Asahi Glass Co, Ltd.		
<i>ME</i>		WO 00/21898 A1	04/20/2000	Samsung Electronics Co.		

no translation

Examiner Signature	<i>Michelle Estrada</i>	Date Considered	2/27/06
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

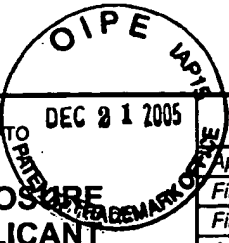
IDS Form PTO/SB/08: Substitute for form 1449A/PTO				Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>				Application Number	10/954,182
				Filing Date	October 1, 2004
				First Named Inventor	ZHANG, Hongmei
				Art Unit	2823
				Examiner Name	ESTRADA, Michelle
Sheet	2	of	2	Attorney Docket Number	9140.0016-01

NON PATENT LITERATURE DOCUMENTS

Examiner Initials	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
ME		Crowder, et al., "Low-Temperature Single-Crystal Si TFT's Fabricated on Si Films Processed via Sequential Lateral Solidification," IEEE, vol. 19, no. 8 (August 1998), pages 306-308.	
ME		Greene et al., "Morphological and electrical properties of rf sputtered Y203-doped ZrO2 thin films," J. Vac. Sci. Technol., vol. 13, no. 1 (Jan/Feb 1976), pages 72-75.	
ME		Hwang, Man-Soo et al., "The effect of pulsed magnetron sputtering on the properties of indium tin oxide thin films," Elsevier Science B.V., P. 29-33, (2003).	
ME		Im, et al. "Controlled Super-lateral Growth of Si Films for Microstructural Manipulation and Optimization," Materials Science Program (1998), pages. 603-617.	
ME		Im, et al., "Crystalline Si Films for Integrated Active-Matrix LiquidCrystal Displays," MrS Bulletin (March 1996), pages. 39-48.	
ME		Im, et al., "Single-crystal Si films for thin-film transistor devices," American Institute of Physics (1997), pages. 3434-3436.	
ME		Tukamoto, H. et al., "Electronic Conductivity of LiCoO ₂ and Its Enhancement by Magnesium Doping," J. Electrochem. Soc., vol. 44, no. 9, pages 3164-3168 (September 1997).	
ME		Response to Office Action filed on March 14, 2005 in U.S. Serial No. 10/291,179 (Attorney Docket No. 09140-0001-00).	
ME		Office Action issued on June 15, 2005 in U.S. Serial No. 10/291,179 (Attorney Docket No. 09140-0001-00).	
ME		Office Action issued on March 17, 2005 in U.S. Serial No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
ME		Response to Office Action filed on June 17, 2005 in U.S. Serial No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
ME		Office Action issued on July 8, 2005 in U.S. Serial No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
ME		Office Action dated January 13, 2005, received in Application No. 10/101,863 (Attorney Docket No. 09140.0016-00).	
ME		Response to office Action filed on June 10, 2005 in U.S. Serial No. 10/101,863 (Attorney Docket No. 09140.0016-00).	
ME		Office Action issued on March 14, 2005 in U.S. Serial No. 10/789,953 (Attorney Docket No. 09140-0030-00).	
ME		Office Action issued March 24, 2005 in U.S. Application No. 10/851,542 (Attorney Docket No. 09140.0033-00).	

Examiner Signature	<i>Michelle Estrada</i>	Date Considered	2/27/04
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.



EXPRESS MAIL NO. EV 758329240 US

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Complete if Known

INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>		Application Number	10/954,182
		Filing Date	October 1, 2004
		First Named Inventor	Hongmei ZHANG
		Art Unit	2823
		Examiner Name	ESTRADA, Michelle
		Attorney Docket Number	9140.0016-01

Sheet 1 of 1

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner Initials [*]	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
ME		US 2002/0076133 A1	06-20-2002	Li et al.	
ME		US 5,478,456	12-26-1995	Humpal et al.	
ME		US 6,846,765 B2	01-25-2005	Imamura et al.	

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

FOREIGN PATENT DOCUMENTS						
Examiner Initials [*]	Cite No. ¹	Foreign Patent Document	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁴
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)				

NON PATENT LITERATURE DOCUMENTS			
Examiner Initials [*]	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
ME		DOREY, R.A., "Low temperature micromoulding of functional ceramic devices," Grant summary for GR/S84156/01 for the UK Engineering and Physical Sciences Research Council, 2 pages (2004).	
ME		HOWSON, R.P., "The reactive sputtering of oxides and nitrides," <i>Pure & Appl. Chem.</i> 66(6):1311-1318 (1994).	
ME		Office Action issued September 21, 2005 in U.S. Application No. 11/100,856 (Attorney Docket No. 09140.0015-01).	
ME		Office Action issued on August 4, 2005, in U.S. Application No. 10/101,863 (Attorney Docket No. 09140.0016-00).	
ME		Office Action issued on August 8, 2005 in U.S. Application No. 10/101,341 (Attorney Docket No. 09140-0017-00).	
ME		Office Action issued on October 3, 2005 in U.S. Application No. 10/650,461 (Attorney Docket No. 09140-0025-00).	
ME		Office Action issued on March 24, 2005 in U.S. Application No. 10/851,542 (Attorney Docket No. 09140-0033-00).	
ME		Response to Office Action filed July 25, 2005 in U.S. Application No. 10/851,542 (Attorney Docket No. 09140-0033-00).	
ME		Office Action issued on October 19, 2005 in U.S. Application No. 10/851,542 (Attorney Docket No. 09140.0033-00).	

Examiner Signature	<i>Michelle Estrada</i>	Date Considered	2/27/06
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

Notice of References Cited	Application/Control No. 10/954,182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.	
	Examiner Michelle Estrada	Art Unit 2823	Page 1 of 1

U.S. PATENT DOCUMENTS

*	Document Number Country Code-Number-Kind Code	Date MM-YYYY	Name	Classification	
*	A	US-6,673,716 B1	01-2004	D'Couto et al.	438/656
	B	US-			
	C	US-			
	D	US-			
	E	US-			
	F	US-			
	G	US-			
	H	US-			
	I	US-			
	J	US-			
	K	US-			
	L	US-			
	M	US-			

FOREIGN PATENT DOCUMENTS

*	Document Number Country Code-Number-Kind Code	Date MM-YYYY	Country	Name	Classification
	N				
	O				
	P				
	Q				
	R				
	S				
	T				

NON-PATENT DOCUMENTS

*	Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)
U	
V	
W	
X	

*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)
 Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.



PATENT
Customer No. 22,852
Attorney Docket No. 9140.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
)	
ZHANG, Hongmei et al.)	Group Art Unit: 2823
)	
Application No.: 10/954,182)	Examiner: ESTRADA, Michelle
)	
Filed: October 1, 2004)	
)	Confirmation No.: 9873
For: BIASED PULSE DC REACTIVE)	
SPUTTERING OF OXIDE FILMS)	

MAIL STOP AMENDMENT

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

AMENDMENT AND RESPONSE TO OFFICE ACTION

In reply to the Office Action mailed March 9, 2006, the period for response having been extended to August 9, 2006, by a request for extension of two months and authorization for the Commissioner to charge the fee to deposit account, please amend the above-identified application as follows:

Amendments to the Claims are reflected in the listing of claims that begins on page 2 of this paper.

Remarks/Arguments follow the amendment sections on page 6 of this paper.

AMENDMENTS TO THE CLAIMS:

This listing of claims will replace all prior versions and listings of claims in the application:

Claims 1-40 (Canceled).

Claim 41 (Previously presented): The method of claims 59, 60, or 85, wherein the target is a metallic target and the process gas includes oxygen.

Claim 42 (Currently amended): The method of claims 43, 59, 60, or 85, wherein the target is a metallic target and the process gas includes one or more of a set consisting of N₂, NH₃, CO, NO, CO₂, halide containing gasses.

Claim 43 (Currently amended): ~~The method of claims 59, 60, or 85,~~ A method of depositing a film on a substrate, comprising:

providing a process gas between a target and a substrate;

providing pulsed DC power to the target through a narrow band-rejection filter;

providing a magnetic field to the target; and

wherein a material is deposited on the substrate,

wherein the target is a ceramic target.

Claim 44 (Canceled).

Claim 45 (Currently amended): The method of claims 43, 59, 60, or 85, wherein the magnetic field is provided by a moving magnetron.

Claim 46 (Currently amended) The method of claims 43, 59, 60, or 85, further including holding the temperature of the substrate substantially constant.

Claim 47 (Currently amended): The method of claims 43, 59, 60, or 85, wherein the process gas includes a mixture of Oxygen and Argon.

Claim 48 (Currently amended): The method of claims 43, 59, 60, or 85, wherein the Oxygen flow is adjusted to adjust the index of refraction of the film.

Claim 49 (Currently amended): The method of claims 43, 59, 60, or 85, wherein the process gas further includes nitrogen.

Claim 50 (Currently amended): The method of claims 43, 59, 60, or 85, wherein providing pulsed DC power to a target includes providing pulsed DC power to a target which has an area larger than that of the substrate.

Claim 51 (Currently amended): The method of claims 43, 59, 60, or 85, further including uniformly sweeping the target with a magnetic field.

Claim 52 (Previously presented): The method of claim 51 wherein uniformly sweeping the target with a magnetic field includes sweeping a magnet in one direction across the target where the magnet extends beyond the target in the opposite direction.

Claim 53 (Currently amended): The method of claims 43, 59, 60, or 85, wherein the target is an alloyed target.

Claim 54 (Previously presented): The method of claim 53 wherein the alloyed target includes one or more rare-earth ions.

Claim 55 (Previously presented): The method of claim 53 wherein the alloyed target includes Si and Al.

Claim 56 (Previously presented): The method of claim 53 wherein the alloyed target includes one or more elements taken from a set consisting of Si, Al, Er, Yb, Zn, Ga, Ge, P, As, Sn, Sb, Pb, Ag, Au, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy Ho, Tm, and Lu.

Claim 57 (Previously presented): The method of claim 53 wherein the alloyed target is a tiled target.

Claim 58 (Previously presented): The method of claim 57 wherein each tile of the tiled target is formed by prealloy atomization and hot isostatic pressing of a powder.

Claim 59 (Currently amended): A method of depositing a film on a substrate, comprising:

- providing a process gas between a target and a substrate;
- providing pulsed DC power to the target through a narrow band-rejection filter;
- providing a magnetic field to the target; and
- wherein a material is deposited on the substrate, and an oxide film is formed by reactive sputtering in metallic mode.

Claim 60 (Currently amended): A method of depositing a film on a substrate, comprising:

- providing a process gas between a target and a substrate;
- providing pulsed DC power to the target through a narrow band-rejection filter;
- providing a magnetic field to the target; and
- wherein a material is deposited on the substrate, and an oxide film is formed by reactive sputtering in poison mode.

Claim 61 (Previously presented): A method of depositing a film on a substrate, comprising:

- providing a process gas between a metallic target and a substrate;
- providing pulsed DC power to the target;
- providing a magnetic field to the target; and
- reconditioning a metallic target;
- wherein a material is deposited on the substrate.

Claim 62 (Previously presented): The method of claim 61, wherein reconditioning the

metallic target includes:

reactive sputtering in the metallic mode and then reactive sputtering in the poison mode.

Claims 63-84 (Canceled).

Claim 85 (Previously presented): A method of depositing a film on a substrate, comprising:

providing a process gas between a target and a substrate;

providing pulsed DC power to the target;

providing a magnetic field to the target; and

wherein a material is deposited on the substrate, and an oxide film is formed by reactive sputtering in a mode between a metallic mode and a poison mode.

Claim 86 (Canceled).

Claim 87 (Currently amended): The method according to claims 43, 59, 60, or 85, ~~further comprising:~~ wherein the narrow band-rejection filter rejects power at an RF frequency, and further including

~~providing a narrow band RF filter between the pulsed DC power supply and the target; and~~

providing an RF bias to the substrate at the RF frequency.

Claim 88 (Currently amended): The method according to claim 87, wherein the ~~narrow-band RF~~ narrow band-rejection filter has a bandwidth of about 100 kHz.

Claim 89 (Currently amended): The method according to claim 87, wherein the RF ~~bias~~ has a frequency of frequency is about 2 MHz.

Claims 90-92 (Canceled).

REMARKS

Claims 41-43, 45-62, 85, and 87-89 are pending in this application. The Examiner has rejected claims 41-43 and 45-60, objected to claims 87-89, and allowed claims 61, 62, and 85. In this Amendment, claims 42, 43, 45-51, 53, 59-60, and 87-89 have been amended. Claim 43 has been amended to be independent. Claims 42, 45-51, 53, and 87 have been amended to adjust their dependency. Claims 87-89 have been further amended for clarity to better claim the invention. Claims 59 and 60 have been amended as described below.

Claim Rejections under 35 U.S.C. § 103

Claims 45, 47, 49, 51, 52, 59, and 60

The Examiner rejected claims 45, 47, 49, 51, 52, 59, and 60 under 35 U.S.C. 103(a) as being unpatentable over U.S. Patent No. 6,117,279 ("Smolonoff et al.") in view of U.S. Patent No. 6,673,716 ("D' Couto et al.").

In the interest of furthering prosecution, Claims 59 and 60 have been amended to clarify that the pulsed DC power is supplied to the target through "a narrow band-rejection filter." Neither Smolonoff nor D' Couto teach a narrow band-rejection filter between the pulsed-DC power and the target. Further, the band-rejection filter is the subject matter of claim 87, which the Examiner has indicated would be allowable if rewritten in independent form.

Claims 45, 47, 49, 51, and 52 are multiply-dependent claims that depend from claims 43, 59, 60, or 85. The Examiner has indicated that claim 85 is allowable. Claims 59-60 are allowable, as discussed above. Claim 43, which also recites "a narrow band-rejection filter," is allowable over Smolonoff and D' Couto at least for the same reasons as discussed above.

Claims 41, 42, 46, 48, and 50

The Examiner rejected claims 41, 42, 46, 48, and 50 under 35 U.S.C. 103(a) as being unpatentable over Smolonoff et al. in view of D’Couto et al. as applied to claims 45, 47, 49, 51, 52, 59, and 60, and further in view of U.S. Published Application No. 2004/0077161 (“Chen et al.”).

As discussed above, claims 43, 59, 60, and 85 are allowable over Smolonoff in view of D’Couto. Chen does not cure the defects in Smolonoff and D’Couto, and therefore claims 43, 59, 60, and 85 are allowable over the combination of Smolonoff, D’Couto, and Chen. Claim 41 is a multiply dependent claim that depends from claims 59, 60, or 85 and is therefore allowable for at least the same reasons as is claims 59, 60, and 85. Claims 42, 46, 48, and 50 are multiple dependent claims that depend from claims 43, 59, 60, or 85 and are therefore allowable for at least the same reasons as is claims 43, 59, 60, and 85.

Claims 43 and 53-58

The Examiner has rejected claims 43 and 53-58 under 35 U.S.C. § 103(a) as being unpatentable over Smolonoff et al. in view of D’Couto et al. as applied to claims 45, 47, 49, 51, 52, 59, and 60 above, and further in view of Milonopoulou et al. (2003/0175142).

As an initial matter, Milonopoulou can not be considered prior art to the present application. Milonopoulou was co-filed on March 16, 2002 along with the parent application of the present application (U.S. Application No. 10/101,863). These two applications cross-reference each other and are incorporated by reference one into the other. Milonopoulou is incorporated into the present application in Paragraph [0048], as amended in the preliminary

amendment filed with the present continuation application on September 30, 2004. The parent of the present application is incorporated by reference in Paragraph [0039] of Milonopoulou.

As discussed above, claims 43, 59, and 60 are allowable over the combination of Smolonoff and D' Couto. Claims 53-58 depend from claims 43, 59, 60, and 85 and are therefore allowable for at least the same reasons as is claims 43, 59, 60, and 85.

Allowable Subject Matter

The Examiner objected to claims 87-89 as being independent upon a rejected base claim, but would be allowable if rewritten in independent form including all of the limitations of the base claim and any intervening claims. The Examiner has further indicated that claims 61, 62, and 85 are allowed.

Subject matter from claim 87 has been incorporated in independent claims 43, 59, and 60. Current claims 87-89 are multiply dependent and depend from claims 43, 59, 60, or 85. Claims 87-89, therefore, are allowable for at least the same reasons as are claims 43, 59, 60, and 85.

Conclusion

In view of the foregoing amendments and remarks, Applicants respectfully request reconsideration and reexamination of this application and the timely allowance of the pending claims.

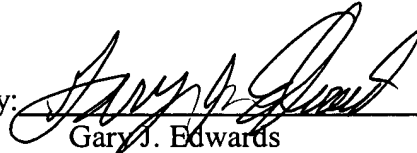
Please grant any extensions of time required to enter this response and charge any additional required fees to Deposit Account No. 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Date: August 9, 2006

By:



Gary J. Edwards
Reg. No. 41,008

**EXPRESS MAIL LABEL NO.
EV 901562545 US**



08-11-06

IFW

PATENT
Customer No. 22,852
Attorney Docket No. 9140.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
)	
ZHANG, Hongmei et al.)	Group Art Unit: 2823
)	
Application No.: 10/954,182)	Examiner: ESTRADA, Michelle
)	
Filed: October 1, 2004)	
)	Confirmation No.: 9873
For: BIASED PULSE DC REACTIVE)	
SPUTTERING OF OXIDE FILMS)	

MAIL STOP AMENDMENT
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

PETITION FOR EXTENSION OF TIME

Applicants petition for a two month extension of time to reply to the Office action of March 9, 2006. The Commissioner is hereby authorized to charge the fee of \$450.00 to Deposit Account No. 06-0916.

Please grant any extensions of time required to enter this response and charge any additional required fees to our deposit account 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

08/14/2006 MR/ANCO 00000010 060916 10954182

Date: August 9, 2006

By: *Gary J. Edwards*
Gary J. Edwards
Reg. No. 41,008

01 08-11-06 450.00 DA

**EXPRESS MAIL LABEL NO.
EV 901562545 US**



PATENT
Customer No. 22,852
Attorney Docket No. 9140.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)
)
ZHANG, Hongmei et al.) Group Art Unit: 2823
)
Application No.: 10/954,182) Examiner: ESTRADA, Michelle
)
Filed: October 1, 2004)
) Confirmation No.: 9873
For: BIASED PULSE DC REACTIVE)
SPUTTERING OF OXIDE FILMS)

MAIL STOP AMENDMENT

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

FIFTH SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT
UNDER 37 C.F.R. § 1.97(c)

Pursuant to 37 C.F.R. §§ 1.56 and 1.97(c), Applicants bring to the attention of the Examiner the documents on the attached listing. This Information Disclosure Statement is being filed after the events recited in Section 1.97(b) but, to the undersigned's knowledge, before the mailing date of either a Final action, Quayle action, or a Notice of Allowance. Under the provisions of 37 C.F.R. § 1.97(c), the Commissioner is hereby authorized to charge the fee of \$180.00 to Deposit Account No. 06-0916 as specified by Section 1.17(p).

Copies of the listed non-patent literature documents are attached. ^{08/14/2006} ^{180.00 DA} ⁰⁶⁻⁰⁹¹⁶ ¹⁰⁹⁵⁴¹⁸²
patents and patent publications are not enclosed.

Applicants respectfully request that the Examiner consider the listed documents and indicate that they were considered by making appropriate notations on the attached form.

This submission does not represent that a search has been made or that no better art exists and does not constitute an admission that each or all of the listed documents are material or constitute "prior art." If the Examiner applies any of the documents as prior art against any claims in the application and Applicants determine that the cited documents do not constitute "prior art" under United States law, Applicants reserve the right to present to the office the relevant facts and law regarding the appropriate status of such documents.

Applicants further reserve the right to take appropriate action to establish the patentability of the disclosed invention over the listed documents, should one or more of the documents be applied against the claims of the present application.

If there is any fee due in connection with the filing of this Statement, please charge the fee to our Deposit Account No. 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Dated: August 9, 2006

By: 

Gary J. Edwards
Reg. No. 41,008

**Express Mail Label No.
EV 901562545 US**

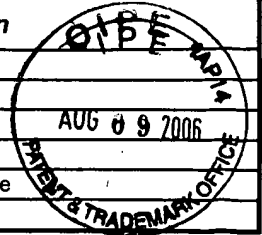
**INFORMATION DISCLOSURE
STATEMENT BY APPLICANT**

(Use as many sheets as necessary)

Sheet 1 of 3

Complete if Known

Application Number	10/954,182
Filing Date	October 1, 2004
First Named Inventor	ZHANG, Hongmei
Art Unit	2823
Examiner Name	ESTRADA, Michelle
Attorney Docket Number	9140.0016-01

**U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS**

Examiner Initials [*]	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
		US 4,082,569	04/04/1978	Evans, Jr.	
		US 4,710,940	12/01/1987	Sipes, Jr.	
		US 4,785,459	11/15/1988	Baer	
		US 5,435,826	07/25/1995	Sakakibara et al.	
		US 5,472,795	12/05/1995	Atita	
		US 5,512,147	04/30/1996	Bates et al.	
		US 5,569,520	10/29/1996	Bates	
		US 5,597,660	01/28/1997	Bates et al.	
		US 5,612,152	03/18/1997	Bates	
		US 5,645,626	07/08/1997	Edlund et al.	
		US 5,702,829	12/30/1997	Paidassi et al.	
		US 6,045,626	04/04/2000	Yano et al.	
		US 6,168,884 B1	01/02/2001	Neudecker et al.	
		US 6,236,793 B1	05/22/2001	Lawrence et al.	
		US 6,242,132 B1	06/05/2001	Neudecker et al.	
		US 6,306,265 B1	10/23/2001	Fu et al.	
		US 6,365,300 B1	04/02/2002	Ota et al.	
		US 6,760,520 B1	07/06/2004	Medin et al.	
		US 6,818,356 B1	11/16/2004	Bates	
		US 6,884,327 B2	04/26/2005	Pan et al.	
		US 2001/0031122 A1	10/18/2001	Lackritz et al.	
		US 2002/0014406 A1	02/07/2002	Takashima	
		US 2002/0115252 A1	08/22/2002	Haukka et al.	
		US 2003/0035906 A1	02/20/2003	Memarian et al.	
		US 2003/0178637 A1	09/25/2003	Chen et al.	
		US 2003/0185266 A1	10/02/2003	Henrichs	
		US 2004/0043557 A1	03/04/2004	Haukka et al.	

Examiner Signature		Date Considered	
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

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EV 901562545 US

IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known		
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>			<i>Application Number</i>	10/954,182	
			<i>Filing Date</i>	October 1, 2004	
			<i>First Named Inventor</i>	ZHANG, Hongmei	
			<i>Art Unit</i>	2823	
			<i>Examiner Name</i>	ESTRADA, Michelle	
Sheet	2	of	3	<i>Attorney Docket Number</i>	9140.0016-01

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
		US 2005/0183946 A1	08/25/2005	Pan et al.	
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Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

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Examiner Initials [*]	Cite No. ¹	Foreign Patent Document	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁶
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)				
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Examiner Initials [*]	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
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Examiner Signature		Date Considered	
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

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			<i>Examiner Name</i>	ESTRADA, Michelle
			<i>Attorney Docket Number</i>	9140.0016-01
Sheet	3	of	3	

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		Office Action issued on March 23, 2006, in U.S. Application No. 10/650,461 (Atty. Docket No. 09140-0025-00).	
		Response to Office Action filed on July 24, 2006, in U.S. Application No. 10/650,461 (Atty. Docket No. 09140-0025-00).	
		Response to Office Action filed March 2, 2006 in U.S. Application No. 10/789,953 (Atty. Docket No. 09140.0030-00).	
		Final Office Action issued on May 19, 2006 in U.S. Application No. 10/789,953 (Atty. Docket No. 09140.0030-00).	
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		Specification as filed September 2, 2005, for U.S. Appl. No. 11/218,652 (Atty. Docket No. 09140.0052-00).	

Examiner Signature		Date Considered	
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**Express Mail Label No.
EV 901562545 US**

PTO 06-6046

German Patent No. DE 37 38 738 C1

POWDER-METALLURGICAL PROCESS FOR THE PREPARATION OF TARGETS

Dr. Stephan Schittny et al.

UNITED STATES PATENT AND TRADEMARK OFFICE
WASHINGTON, D.C. AUGUST 2006
TRANSLATED BY THE MCELROY TRANSLATION COMPANY

FEDERAL REPUBLIC OF GERMANY
GERMAN PATENT OFFICE
PATENT NO. DE 37 38 738 C1
(Patentschrift)

Int. Cl.⁴: B 22 F 7/00
H 01 L 21/203

Filing No.: P 37 38 738.3-24

Filing Date: November 14, 1987

Publication Date of Patent Grant: January 26, 1989

POWDER-METALLURGICAL PROCESS FOR THE PREPARATION OF TARGETS

[Pulvermetallurgisches Verfahren zur Herstellung von Targets]

Inventors: Dr. Stephan Schittny et al.

Patent Holder: Degussa AG

Publications Taken in Consideration
For Judging Patentability: DE 35 37 191 A1

Claims

1. Process for the preparation of highly pure mechanically stable and dense targets of rare earths and the transition metals iron, cobalt and/or nickel for cathode evaporation and vaporization installations by means of the action of pressure and temperature on a corresponding powder mixture in an inert-gas atmosphere or under vacuum, characterized in that the rare earths are inserted with the corresponding transition metals in the form of master alloys.

2. The process of Claim 1, characterized in that master alloys having a eutectic composition are inserted.

Description

The invention pertains to a process for the preparation of highly pure, mechanically stable and dense targets of rare earths and the transition metals iron, cobalt and/or nickel for cathode

evaporation and vaporization installations by means of the action of pressure and temperature on a corresponding powder mixture in an inert-gas atmosphere or under vacuum

Targets are required in installations for cathode evaporation and for the vaporization of objects. With the aid of cathode evaporation (sputtering) and vaporization, a thin layer can be produced on a substrate, the applications for which span from functional layers in electronics and data systems engineering through corrosion-protective layers and anti-abrasion layers to optical layers for decorative and thermic purposes.

In cathode evaporation, an electrical gas discharge occurs between cathode (target) and counter electrode, during which impacting ions eject particles of atomic size from the target, which condense on substrates arranged in the area of the counter electrode.

Either inert gases such as argon or helium, or reactive gases, such as oxygen, nitrogen or acetylene are put in at low pressure as a gas discharge atmosphere.

In inert gas sputtering, the target commonly is composed of the material of which the layer to be formed shall be composed, while in reactive sputtering, ejected target particles react with the reaction gas and are condensed as a layer in the form of a reaction product.

In the vaporization process, the target material is thermally vaporized in a vacuum by means of electron beam heating or resistance heating and deposited as a thin layer on the substrate.

Targets commonly can be prepared using melting technology with corresponding secondary processing by means of non-cutting and tensioning processes or with powder metallurgy, by means of compressing and sintering corresponding powders or powder mixtures.

For target materials, which owing to their composition contain higher portions of brittle phases, preparation processes using melting technology prove to be problematic, since such targets when cooling after casting exhibit fissures owing to thermal stress and can crumble into pieces. In addition, such materials cannot for the most part be mechanically machined, such that certain desirable target geometries cannot be prepared.

Powder-metallurgical processes exhibit problems with those target materials containing higher portions of reaction-sensitive components and that react, e.g., with atmospheric oxygen. Due to the large specific surface area of the powder and the resulting reactivity, high-value target qualities having low oxygen content cannot be produced using powder metallurgy.

In addition, products prepared using powder metallurgy for the most part exhibit a partially open residual porosity, which is intolerable for oxygen sensitive target materials due to the potential oxidation of the entire target.

These preparation difficulties appear particularly for targets containing a predominance of rare earths and transition metals.

A powder-metallurgical process for the preparation of targets of rare earths and the transition metals iron, cobalt and nickel is known from DE-OS 35 37 191, for which a powder mixture of rare earths and the aforementioned transition metals is subjected to hot forming under decreased pressure and under inert gas at temperatures below the eutectic point, with a brittle intermetallic compound developing at the boundary between the rare earth metal and the transition metal. This process has the disadvantage that hot forming must be carried out expensively for a minimum of at least two hours, by means of which noticeable quantities of brittle intermetallic phases are formed, that powder preparation for rare earths is associated with difficulties and that the oxygen content in the powder of rare earths is still relatively high.

It was thus the problem of the present invention to develop a process for the preparation of highly pure, mechanically stable and dense targets of rare earths and the transition metals iron, cobalt and/or nickel for cathode evaporation and vaporization installations by means of the action of pressure and temperature on a corresponding powder mixture in an inert-gas atmosphere or under vacuum that without great technical expense supplies a product containing limited portions of and a fine distribution of brittle phases and as little oxygen as possible.

This problem is solved in accordance with the invention by means of the fact that the rare earths are inserted with the corresponding transition metals in the form of master alloys.

Preferably, master alloys having a eutectic composition are inserted.

Reaction sensitive rare earths are processed into powders and/or shavings not as such, but as master alloys of rare earths and the corresponding transition metals, which in comparison to pure components feature lower melting points, are well intermixed with powders and/or shavings of the transition metals and are compacted into targets. The compacting of said mixture can be carried out by means of sintering, hot-rolling, hot-pressing, hot-forging, high-temperature isostatic pressing or combinations thereof in a vacuum or under inert gas.

The conditions for compacting (temperature, pressure, time, degree of deformation) are selected such that a mechanically stable compound material emerges, the grain structure of which, as a non-equilibrium state, features only limited portions of brittle equilibrium phases formed by means of diffusion at the boundaries of the rare earth phases and the transition metal phases. Through the use of master alloys between rare earths and transition metals, relatively low compacting temperatures and brief compacting times can be selected. This leads to a decreased technical expense and to a limited formation of diffusion zones of brittle non-equilibrium phases at the contact locations of the powder grains and in the grains.

Surprisingly, it has been shown that fundamentally simpler, highly pure and low-oxygen powders or shavings can be prepared out of master alloys of rare earths with corresponding transition metals rather than out of pure rare earths. These master alloys may be used to produce shavings and powders, e.g., by means of machining, such as milling or filing under protective

gas, which is not possible without difficulty with pure rare earth metals, owing to their ductility, even with subsequent grinding. During the comminution process, the brittle phase portions of the most extremely fine grain structure of master alloys causes the formation of particles having a suitable grain shape and grain size distribution, enabling a powder preparation having a good yield and very low oxygen content (< 500 ppm).

Likewise in powder preparation by means of inert gas atomization, lower values of impurities and more limited oxygen content in the powder are achieved from lower melting master alloys owing to lower reaction rates with the crucible material and with the atmosphere at lower process temperatures and owing to the briefer residence time in the installation. Moreover, the atomization parameters (superheating, cooling rates, process gas pressure, etc.) can be optimized to a wide degree, such that the powder yield and the grain fraction can be adjusted more favorably in comparison to atomization of pure rare earths.

Surprisingly, the use of master alloys of rare earths and transition metals leads to compacting times considerably briefer in comparison to those with the use of pure rare earths, signifying a significant economic advantage of this process.

As a rule, compacting times are less than one hour and, more often than not, less than 30 minutes. Apart from this, targets prepared in accordance with the invention exhibit more favorable mechanical and magnetic properties in comparison to targets produced with powder metallurgy from pure metals. As has been shown, this is based on an advantageous distribution of portions present in the grain structure of free iron, cobalt and/or nickel, on the limited quantities of intermetallic phases and on the residual eutectic originating from the master alloys.

The following examples shall explain the process in accordance with the invention in greater detail:

1) In order to prepare highly pure, compact, mechanically stable sputter targets, first a master alloy of 80 atom% terbium and 20 atom% iron is prepared in a vacuum induction oven (10^{-5} Pa). Subsequent pulverization and further processing is carried out exclusively under argon protective gas. With the aid of a wave-milling cutter, the ingot is machined to a coarse powder. A terbium-iron powder having an average grain size $\leq 105 \mu\text{m}$ and having a yield of 70-80% is obtained by means of subsequent grinding in a ball mill. The desired target composition of, e.g., 67 atom% iron, 25 atom% terbium and 8 atom% cobalt is regulated by means of a 20 minute mixing of the corresponding iron, cobalt and master alloy powder quantities in an asymmetric moved mixer.

The processes of high-temperature isostatic pressing (HIP), hot-rolling and hot-forging in a capsule were employed in order to compact the powder mixture into targets.

For high-temperature isostatic pressing, process parameters of 820°C final temperature, 200 Mpa pressure and 20 minute residence time at the final temperature were selected. Sheet

steel cans were used as containers, which prior to being brought into the installation were evacuated at 300°C for over 3 hours. This produces mechanically stable, machinable molding bodies featuring a non-equilibrium grain structure of iron grains, zones of intermetallic phases and residual eutectic. The density is > 99% of the theoretical density, the oxygen content less than 1200 ppm. Longer residence times of, e. g., 1-2 hours at 820°C result in a wide reaching formation of large-surface zones of the intermetallic equilibrium phases, which causes an undesirable brittleness of the grain structure. It is for this reason that residence times as brief as possible are to be observed.

For compacting by means of hot-rolling, the powder mixture is filled in a sheet iron capsule, which is evacuated and welded shut. Conversion occurs at 650°C up to a degree of conversion of approximately 60% in several passes in a period of few minutes. After removal of the capsule material, a stable, highly pure target is yielded having a density of $\approx 99\%$ of the theoretical density and an oxygen content of 1200 ppm. The grain structure is of a form similar to that for compacting by means of high-temperature isostatic pressing.

For hot-forging, the powder mixture likewise is filled in a sheet steel can and evacuated. The forging process can be carried out at 800°C in a 200 ton forge. The achievable density is, at 60% conversion $\approx 99\%$ of the theoretical density.

2. A different type of powder preparation is atomization of a melt in an inert-gas flow. For the use of low-melting point master alloys of the composition 66 atom% gadolinium, 18 atom% iron and 16 atom% cobalt having a melting temperature of approximately 620°C, an atomizing temperature of 700-800°C can be selected. This comparatively very low atomizing temperature enables, for extremely reactive rare earth materials, the preparation of low-oxygen, very pure master alloy powders, since the reaction of the melt with the crucible material and with the atmosphere is comparatively limited. Moreover, the master alloy melt exhibits a favorable viscosity and surface tension at these temperatures, such that the atomized powder features good yields of e.g., 80-90% at a grain size $\leq 88 \mu\text{m}$. With the aid of the mixing and compacting processes described in Example 1, highly pure, compact, stable molding bodies having oxygen contents < 1000 ppm likewise are obtained. The non-equilibrium grain structures in turn are composed of iron grains, zones of intermetallic phases and residual eutectic.

Since the intermetallic phases are present in a fine distribution in the master alloys, and the grain structure is not very brittle, the blend portion of free iron can be reduced in comparison with the intermetallic phases by varying the composition of the master alloys. The magnetic properties of the targets, which are important for magnetron sputtering, can be improved by said means.

DERWENT- 1989-032907
ACC-NO:

DERWENT- 198905
WEEK:

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TITLE: High purity, dense targets for cathodic sputtering -
produced from rare earth and transition metals iron,
cobalt and/or nickel in inert atmos., etc.

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PATENT-ASSIGNEE: DEGUSSA AG [DEGS]

PRIORITY-DATA: 1987DE-3738738 (November 14, 1987)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
DE <u>3738738</u>	C January 26, 1989	N/A	004	N/A

APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO	APPL-DATE
DE 3738738C	N/A	1987DE-3738738	November 14, 1987

INT-CL (IPC): B22F007/00, H01L021/20

ABSTRACTED-PUB-NO: DE 3738738C

BASIC-ABSTRACT:

High-purity, mechanically stable and dense targets are produced for cathodic sputtering and vaporisation systems from rare earths and the transition metals Fe, Co and/or Ni from powder mixts, under the effect of pressure and temp., in inert atmos. or vacuum. High-purity, low-0 powders or chips are mfd. from pre-alloyed rare earths with the corresp. transition metal (s). Chips are mfd. under inert atmos.,

while powders are produced by atomisation of the pre-alloyed material, having relatively low m.pt.

ADVANTAGE - Prod. has low proportion and fine distribution of brittle phases, with lowest possible O2 content.

CHOSEN- Dwg. 0/0
DRAWING:

TITLE- HIGH PURE DENSE TARGET CATHODE SPUTTER PRODUCE RARE EARTH
TERMS: TRANSITION METAL IRON COBALT NICKEL INERT ATMOSPHERE

DERWENT-CLASS: M13 M22 P53 U11

CPI-CODES: M13-G02; M22-H03G;
EPI-CODES: U11-C05C2; U11-C09A;

SECONDARY-ACC-NO:

CPI Secondary Accession Numbers: C1989-014270

Non-CPI Secondary Accession Numbers: N1989-025076

19 BUNDESREPUBLIK
DEUTSCHLAND

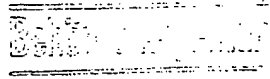


DEUTSCHES
PATENTAMT

12 Patentschrift
11 DE 37 38 738 C 1

51 Int. Cl. 4:
B22F 7/00
H 01 L 21/203

21 Aktenzeichen: P 37 38 738.3-24
22 Anmeldetag: 14. 11. 87
43 Offenlegungstag: —
45 Veröffentlichungstag
der Patenterteilung: 26. 1. 89



DE 3738738 C 1

Innerhalb von 3 Monaten nach Veröffentlichung der Erteilung kann Einspruch erhoben werden

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56 Für die Beurteilung der Patentfähigkeit
in Betracht gezogene Druckschriften:
DE 35 37 191 A1

54 Pulvermetallurgisches Verfahren zur Herstellung von Targets

Zur Herstellung von hochreinen, mechanisch stabilen und dichten Targets aus seltenen Erden und den Übergangsmetallen Eisen, Kobalt und Nickel auf pulvermetallurgischem Weg vermeidet man hohe Gehalte und ungünstige Verteilungen an spröden Phasen und größere Sauerstoffgehalte im Werkstoff durch Verwendung von Vorlegierungen aus seltenen Erden und den Übergangsmetallen anstelle reinen seltenen Erden.

DE 3738738 C 1

Patentansprüche

1. Verfahren zur Herstellung von hochreinen mechanisch stabilen und dichten Targets für Kathodenzerstäubungs- und Bedampfungsanlagen aus Seltenen Erden und den Übergangsmetallen Eisen, Kobalt und/oder Nickel durch Einwirkung von Druck und Temperatur auf entsprechende Pulvergemische in Inertgasatmosphäre oder unter Vakuum, dadurch gekennzeichnet, daß die Seltenen Erden in Form von Vorlegierungen mit den entsprechenden Übergangsmetallen eingesetzt werden.
2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß Vorlegierungen mit eutektischer Zusammensetzung eingesetzt werden.

Beschreibung

Die Erfindung betrifft ein Verfahren zur Herstellung von hochreinen, mechanisch stabilen und dichten Targets für Kathodenzerstäubungs- und Bedampfungsanlagen aus Seltenen Erden und den Übergangsmetallen Eisen, Kobalt und/oder Nickel durch Einwirkung von Druck und Temperatur auf entsprechende Pulvergemische in Inertgasatmosphäre oder unter Vakuum.

Targets werden in Anlagen zur Kathodenzerstäubung und zur Bedampfung von Gegenständen benötigt. Mit Hilfe der Kathodenzerstäubung (Sputtern) und des Bedampfens können dünne Schichten auf Substraten erzeugt werden, deren Anwendung von funktionalen Schichten in der Elektronik und Datentechnik über Korrosions- und Verschleißschichten bis zu optischen Schichten für dekorative und wärmetechnische Zwecke reicht.

Beim Kathodenzerstäuben findet zwischen Kathode (Target) und Gegenelektrode eine elektrische Gasentladung statt, bei der von den aufrallenden Ionen aus dem Target Teilchen von atomarer Größe herausgeschlagen und auf Substraten, die im Bereich der Gegenelektrode angeordnet sind, niedergeschlagen werden.

Als Gasentladungsatmosphäre werden entweder inerte Gase, wie beispielsweise Argon oder Helium, oder reaktive Gase, wie z. B. Sauerstoff, Stickstoff oder Acetylen, bei geringem Druck eingesetzt.

Beim Inertgassputtern besteht das Target üblicherweise aus dem Material, aus dem die zu bildende Schicht bestehen soll, während beim Reaktivputtern die herausgeschlagenen Targetteilchen mit dem Reaktionsgas reagieren und in Form eines Reaktionsproduktes als Schicht niedergeschlagen werden.

Bei den Bedampfungsverfahren wird das Targetmaterial im Vakuum durch Elektronenstrahl- oder Widerstandsbeheizung thermisch verdampft und als dünne Schicht auf dem Substrat abgeschieden.

Die Targets können üblicherweise schmelztechnisch hergestellt werden mit entsprechender Nachbearbeitung durch umformende und spannende Verfahren, oder pulvermetallurgisch durch Pressen und Sintern entsprechender Pulver bzw. Pulvergemische.

Bei Targetmaterialien, die aufgrund ihrer Zusammensetzung höhere Anteile an spröden Phasen enthalten, erweisen sich schmelztechnische Herstellverfahren als problematisch, da solche Targets beim Abkühlen nach dem Gießen aufgrund von thermischen Spannungen Risse aufweisen und in Stücke zerfallen können. Darüber hinaus sind solche Materialien meist nicht mechanisch bearbeitbar, so daß bestimmte wünschenswerte Targetgeometrien nicht herstellbar sind.

Pulvermetallurgische Verfahren zeigen bei denjenigen Targetmaterialien Probleme, die höhere Anteile an reaktionsempfindlichen Komponenten enthalten, und z. B. mit dem Luftsauerstoff reagieren. Wegen der großen spezifischen Oberfläche der Pulver und der daraus resultierenden Reaktionsfreudigkeit lassen sich keine hochwertigen Targetqualitäten mit niedrigem Sauerstoffgehalt pulvermetallurgisch herstellen.

Außerdem weisen pulvermetallurgisch hergestellte Produkte meist eine zum Teil offene Restporosität auf, die bei sauerstoffempfindlichen Targetmaterialien wegen der möglichen Oxidation des gesamten Targets nicht tolerierbar ist.

Diese Herstellungsschwierigkeiten treten insbesondere bei Targets auf, die überwiegend Seltene Erden und Übergangsmetalle enthalten.

Aus der DE-OS 35 37 191 ist ein pulvermetallurgisches Verfahren zur Herstellung von Targets aus Seltenen Erden und den Übergangsmetallen Eisen, Kobalt und Nickel bekannt, bei dem ein Pulvergemisch aus Seltenen Erden und den genannten Übergangsmetallen unter vermindertem Druck und unter Inertgas bei Temperaturen unterhalb des eutektischen Punktes einer Warmverformung unterzogen wird, wobei sich eine spröde intermetallische Verbindung an der Grenzfläche zwischen dem Seltenen Erdmetall und dem Übergangsmetall ausbildet. Dieses Verfahren hat den Nachteil, daß die Warmverformung aufwendig mindestens zwei Stunden lang erfolgen muß, wodurch sich bemerkbare Mengen an spröden intermetallischen Phasen bilden, daß die Pulverherstellung bei den Seltenen Erden mit Schwierigkeiten verbunden ist und der Sauerstoffgehalt im Pulver der Seltenen Erden noch relativ hoch ist.

Es war daher Aufgabe der vorliegenden Erfindung, ein Verfahren zur Herstellung von hochreinen, mechanisch stabilen und dichten Targets für Kathodenzerstäubungs- und Bedampfungsanlagen aus Seltenen Erden und den Übergangsmetallen Eisen, Kobalt und/oder Nickel durch Einwirkung von Druck und Temperatur auf entsprechende Pulvergemische in Inertgasatmosphäre oder unter Vakuum zu entwickeln, das ohne großen technischen Aufwand ein Produkt liefert, das geringe Anteile und eine feine Verteilung an spröden Phasen und möglichst wenig Sauerstoff enthält.

Diese Aufgabe wird erfindungsgemäß dadurch gelöst, daß die Seltenen Erden in Form von Vorlegierungen mit den entsprechenden Übergangsmetallen eingesetzt werden.

Vorzugsweise werden Vorlegierungen mit eutektischer Zusammensetzung eingesetzt.

Die reaktionsempfindlichen Seltenen Erden werden nicht als solche, sondern als Vorlegierungen aus Seltenen Erden und den entsprechenden Übergangsmetallen, die im Vergleich zu den reinen Komponenten niedrigere Schmelzpunkte aufweisen, zu Pulvern und/oder Spänen verarbeitet, mit Pulvern und/oder Spänen der Übergangsmetalle gut durchmischt und zu Targets kompaktiert. Die Kompaktierung dieser Mischung kann durch Sintern, Warmwalzen, Heißpressen, Warmschmieden, Heißisostatische Preßverfahren oder Kombinationen daraus im Vakuum oder unter Inertgas erfolgen.

Die Kompaktierungsbedingungen (Temperatur, Druck, Zeit, Umformungsgrad) werden so gewählt, daß ein mechanisch stabiles Verbundmaterial entsteht, dessen Gefüge als Ungleichgewichtszustand nur geringe Anteile an spröden Gleichgewichtsphasen aufweist, die durch Diffusion an den Grenzflächen der Seltenen Erdphasen und der Übergangsmetallphasen gebildet wer-

den. Durch die Verwendung von Vorlegierungen zwischen Seltenen Erden und Übergangsmetallen können relativ niedrige Kompaktierungstemperaturen und kurze Kompaktierungszeiten ausgewählt werden. Das führt zu einem verminderten technischen Aufwand und zu einer geringen Ausbildung von Diffusionszonen aus spröden Ungleichgewichtsphasen an den Berührungstellen der Pulverkörner und in den Körnern.

Es hat sich überraschenderweise gezeigt, daß aus Vorlegierungen der Seltenen Erden mit den entsprechenden Übergangsmetallen wesentlich einfacher, hochreine und sauerstoffarme Pulver oder Späne herstellbar sind als aus den reinen Seltenen Erden. Aus diesen Vorlegierungen lassen sich z. B. durch spanende Bearbeitung, wie Fräsen oder Feilen, unter Schutzgas Späne und Pulver erzeugen, was bei den reinen Seltenen Erd-Metallen auf Grund ihrer Duktilität auch mit nachfolgendem Mahlen nicht ohne Schwierigkeiten möglich ist. Die spröden Phasenanteile des äußerst feinen Gefüges der Vorlegierungen bewirken beim Zerkleinerungsvorgang die Bildung von Partikeln mit geeigneter Kornform und Korngrößenverteilung, so daß eine Pulverherstellung mit guter Ausbeute und sehr niedrigem Sauerstoffgehalt (< 500 ppm) möglich ist.

Ebenso werden bei der Pulverherstellung durch Inertgasverdüsen aus den niedriger schmelzenden Vorlegierungen aufgrund der bei den tieferen Prozeßtemperaturen geringeren Reaktionsgeschwindigkeiten mit dem Tiegelmateriale und mit der Atmosphäre und aufgrund der kürzeren Verweilzeit in der Anlage, niedrigere Werte an Verunreinigungen und geringere Sauerstoffgehalte im Pulver erzielt. Desweiteren lassen sich hier die Verdüsungparameter (Überhitzung, Abkühlrate, Prozeßgasdruck usw.) in weiten Maßen optimieren, so daß die Pulverausbeute und die Kornfraktion im Vergleich zur Verdüsung der reinen Seltenen Erden günstiger eingestellt werden können.

Die Verwendung von Vorlegierungen aus Seltenen Erden und Übergangsmetallen führt im Vergleich zur Verwendung der reinen Seltenen Erden überraschenderweise zu wesentlich kürzeren Kompaktierungszeiten, was einen bedeutenden wirtschaftlichen Vorteil dieses Verfahrens bedeutet.

Die Kompaktierungszeiten liegen in der Regel unterhalb einer Stunde, meist unterhalb von 30 Minuten. Darüber hinaus zeigen die erfindungsgemäß hergestellten Targets im Vergleich zu Targets, die pulvermetallurgisch aus den reinen Metallen hergestellt wurden, günstigere mechanische und magnetische Eigenschaften. Dies beruht, wie sich gezeigt hat, auf einer vorteilhaften Verteilung der im Gefüge vorhandenen Anteile an freiem Eisen, Kobalt und/oder Nickel, an den geringen Mengen intermetallischer Phasen und an dem aus den Vorlegierungen stammenden Resteutektikum.

Die folgenden Beispiele sollen das erfindungsgemäße Verfahren näher erläutern:

1) Zur Herstellung von hochreinen, kompakten, mechanisch stabilen Sputtertargets wird zuerst eine Vorlegierung aus 80 Atom% Terbium und 20 Atom% Eisen in einem Vakuuminduktionsofen (10^{-5} Pa) hergestellt. Die anschließende Pulverisierung und Weiterverarbeitung erfolgt ausschließlich unter Argon-Schutzgas. Mit Hilfe eines Wellenfräasers wird der Gußblock zu grobem Pulver zerspannt. Durch anschließendes Mahlen in einer Kugelmühle erhält man ein Terbium-Eisenpulver mit einer mittleren Korngröße ≤ 105 μm und mit einer Ausbeute

von 70–80%. Die gewünschte Targetzusammensetzung von z. B. 67 Atom% Eisen, 25 Atom% Terbium und 8 Atom% Kobalt wird durch 20 minütiges Mischen der entsprechenden Eisen-, Kobalt- und Vorlegierungspulvermengen in einem Taumelmischer eingestellt.

Zum Kompaktieren der Pulvermischung zu Targets wurden die Verfahren des Heißisostatischen Pressens (HIP) des Warmwalzens und des Heißschmiedens in einer Kapsel angewendet.

Beim Heißisostatischen Pressen werden als Prozeßparameter 820°C Endtemperatur, 200 MPa Druck und 20 min Haltezeit bei Endtemperatur gewählt. Als Behälter wurden Stahlblechkannen verwendet, die vor Einbringen in die Anlage bei 300°C über 3 Stunden evakuiert wurden. Man erhält auf diese Weise mechanisch stabile, bearbeitbare Formkörper, die ein Ungleichgewichtsgefüge aus Eisenkörnern, Zonen von intermetallischen Phasen und Resteutektikum aufweisen. Die Dichte ist $> 99\%$ der theoretischen Dichte, der Sauerstoffgehalt liegt unterhalb 1200 ppm. Längere Haltezeiten von z. B. 1–2 Stunden bei 820°C haben eine weitgehende Ausbildung von großflächigen Zonen der intermetallischen Gleichgewichtsphasen zur Folge, was eine unerwünschte Versprödung des Gefüges bedingt. Es sind daher möglichst kurze Haltezeiten einzuhalten.

Für das Kompaktieren durch Warmwalzen wird das Pulvergemisch in eine Eisenblechkapsel eingefüllt, die evakuiert und zugeschweißt wird. Die Umformung erfolgt bei 650°C bis zu einem Umformgrad von ca. 60% in mehreren Stichen in einem Zeitraum von wenigen Minuten. Nach Entfernen des Kapselmateriale ergibt sich ein stabiles, hochreines Target mit einer Dichte von $\approx 99\%$ der theoretischen Dichte und einem Sauerstoffgehalt von 1200 ppm. Das Gefüge ist ähnlich ausgebildet wie bei der Kompaktierung durch Heißisostatisches Pressen.

Beim Heißschmieden wird die Pulvermischung ebenfalls in eine Blechkanne gefüllt und evakuiert. Der Schmiedevorgang kann bei 800°C in einer 200 t Schmiede durchgeführt werden. Die erzielbare Dichte beträgt bei 60% Umformung $\approx 99\%$ der theoretischen Dichte.

2. Eine andere Art der Pulverherstellung ist das Verdüsen einer Schmelze im Inertgasstrom. Bei Benutzung von niedrigschmelzenden Vorlegierungen der Zusammensetzung 66 Atom% Gadolinium, 18 Atom% Eisen und 16 Atom% Kobalt mit einer Schmelztemperatur von ca. 620°C kann eine Verdüsungstemperatur von 700–800°C gewählt werden. Diese vergleichsweise sehr niedrige Verdüsungstemperatur ermöglicht bei den extrem reaktionsfreudigen Seltenen Erd-Materialien die Herstellung von sauerstoffarmen, sehr reinen Vorlegungspulvern, da die Reaktion der Schmelze mit dem Tiegelmateriale und der Atmosphäre vergleichsweise gering ist. Des weiteren zeigt die Vorlegungsschmelze bei dieser Temperatur eine günstige Viskosität und Oberflächenspannung, so daß die verdüsten Pulver gute Ausbeuten von z. B. 80–90% bei einer Korngröße ≤ 88 μm aufweisen. Mit Hilfe der in Beispiel 1 beschriebenen Misch- und Kompaktierungsverfahren erhält man ebenfalls hochreine, kompakte, stabile Formkörper mit Sauerstoffgehalten < 1000 ppm. Die Ungleichge-

wichtsgefüge bestehen wiederum aus Eisenkörnern, Zonen von intermetallischen Phasen und Resteutektikum.

Da die intermetallischen Phasen in den Vorlegierungen fein verteilt vorliegen, und das Gefüge nicht sehr verspröden, lassen sich die Mengenanteile an freiem Eisen im Vergleich zu den intermetallischen Phasen durch Variation der Zusammensetzung der Vorlegierungen reduzieren. Die magnetischen Eigenschaften der Targets, die für das Magnetronsputtern wichtig sind, lassen sich hierdurch verbessern.

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Conducting transparent coatings and method for their production

Publication number: EP1092689
Publication date: 2001-04-18
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Classification:
 - international: **C03C17/36; C03C17/36;** (IPC1-7): C03C17/36
 - european: C03C17/36
Application number: EP20000119591 20000907
Priority number(s): DE19991048839 19991011

Also published as:

 US6905776 (B1)
 DE19948839 (A1)

Cited documents:

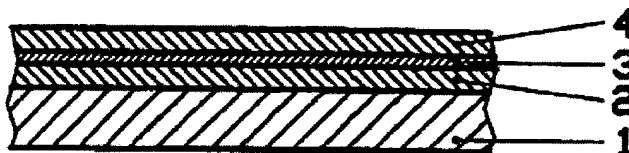
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Abstract of EP1092689

Conducting transparent layer system comprises a silver layer (3) between two oxide layers (2, 4) on a substrate (1) and has a quantity coefficient of more than 0.0855 Ohm $\langle -1 \rangle$ for the wavelengths 435, 545 and 610 nm at a flat resistance R_s of less than 2.9 preferably less than 2.5 Ohm sq. An Independent claim is also included for a process for the production of a conducting transparent layer system comprising applying the second oxide layer (4) using a pulsed direct current (DC) sputtering process or an alternating current (AC)-superimposed DC sputtering process.

Fig.1



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(12) **EUROPÄISCHE PATENTANMELDUNG**

(43) Veröffentlichungstag:
 18.04.2001 Patentblatt 2001/16

(51) Int. Cl.⁷: **C03C 17/36**

(21) Anmeldenummer: 00119591.6

(22) Anmeldetag: 07.09.2000

(84) Benannte Vertragsstaaten:
AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU
MC NL PT SE
 Benannte Erstreckungsstaaten:
AL LT LV MK RO SI

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(30) Priorität: 11.10.1999 DE 19948839

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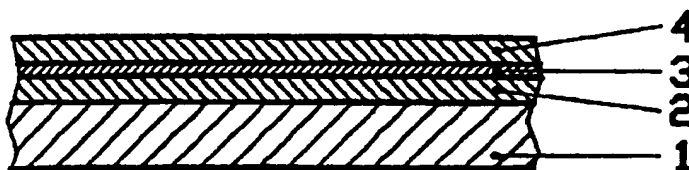
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(54) **Leitende transparente Schichten und Verfahren zu ihrer Herstellung**

(57) Auf einem Substrat aus Glas (1) ist eine Grundschicht (2) aus Indium-Cer-Oxid und darauf eine dünne Kupfer enthaltende Silberschicht (3), beide hergestellt durch DC-Zerstäubung, aufgebracht. Darauf befindet sich eine weitere Indium-Cer-Oxidschicht (4), welche durch AC-überlagerte DC-Zerstäubung hergestellt wird.

Dieses Schichtsystem weist sehr niedere Flächenwiderstände bei gleichzeitig hoher Durchlässigkeit im sichtbaren Spektralbereich, also einen hohen Haackeschen Gütefaktor auf.

Fig.1



EP 1 092 689 A1

Beschreibung

[0001] Die Erfindung betrifft leitende transparente Schichten nach dem Oberbegriff des Patentanspruches 1 sowie ein Verfahren zur Herstellung dieser leitenden transparenten Schichten nach Patentanspruch 6.

[0002] Leitende transparente Schichten werden heute in der Displaytechnik, in der Optoelektronik sowie als Architekturglas vielfältig eingesetzt. Dabei wird einerseits eine möglichst hohe Transmission im sichtbaren Spektralbereich und andererseits eine möglichst hohe Leitfähigkeit beziehungsweise ein möglichst niedriger Flächenwiderstand angestrebt. Als Mass für die Güte leitender transparenter Schichten kann der Haakesche Gütefaktor $\Phi_{TC} = T^{10}/R_S$, definiert in Journal of Applied Physics, Vol. 47, Seite 4086 bis 4089 (1976), verwendet werden. Dabei bedeuten T die optische Transmission der Schicht (als Bruchteil der auffallenden Strahlung) und R_S den Flächenwiderstand in Ω_{sq} . So besitzt beispielsweise eine Schicht mit einer Transmission von 90% und einem Flächenwiderstand von $3 \Omega_{sq}$ einen Haakeschen Gütefaktor von $0,116 \Omega^{-1}$. Eine Schicht mit einer Transmission von 80% und einem Flächenwiderstand von $5 \Omega_{sq}$ besitzt einen Gütefaktor von $0,021 \Omega^{-1}$.

[0003] Eine weitere wichtige Eigenschaft eines solchen Schichtsystems ist seine Ätzbarkeit. Diese hängt von seiner chemischen Zusammensetzung und seiner Dicke ab. Für eine kurze Ätzzeit und gute Kantenschärfe ist es wichtig, dass die Schichtdicke möglichst klein ist, d.h. unter 100 nm beträgt.

[0004] Zur Erzielung hoher Gütefaktoren ist es vorteilhaft, Schichtsysteme aus oxidischen und metallischen Schichten zu kombinieren. So ist es bekannt, sehr dünne Silberschichten zwischen dünne Oxidschichten einzulagern. Durch die Einlagerung zwischen Oxidschichten wird die Silberschicht einerseits stabilisiert und geschützt, andererseits wird gleichzeitig ihre Reflexion vermindert und dadurch die Transmission erhöht. Diese Schichtkombinationen besitzen ferner den Vorteil einer geringen Gesamtschichtdicke, nämlich 100 nm oder weniger, verglichen mit einer Schicht aus Indium-Zinn-Oxid mit vergleichbarem Flächenwiderstand, die eine Dicke von über 500 nm aufweist (S. H. Shin und Koautoren, Thin Solid Films 341 (1999) 225 - 229). Damit können Ätzprozesse, wie sie bei der Herstellung von Displays üblich sind, schneller und mit geringerer Unterätzung hergestellt werden.

[0005] Solche Schichtsysteme sind z. B. beschrieben in: EP 0 599 071 A1, JP 10062602 A und im Artikel von K. K. Choi und Koautoren, Thin Solid Films 341 (1999) 152 - 155.

[0006] In der EP 0 599 071 A1 wird ein Schichtsystem mit der Schichtfolge Indium-Zinn-Oxid, Silber bzw. verschiedene Silberlegierungen, Indium-Zinn-Oxid beschrieben. Durch einstündige Temperung bei 300°C lassen sich Schichten mit einem Flächenwiderstand von $3,2 \Omega_{sq}$ und gleichzeitig guter Transmission im

sichtbaren Bereich herstellen. Für die Wellenlängen 435, 545 und 610 nm ergibt sich ein gemittelter Haakescher Gütefaktor von 0,066. Nachteilig ist jedoch die für Displayanwendungen nötige nachträgliche Temperaturbehandlung, da diese einen zusätzlichen Arbeitsschritt bedeutet.

[0007] In der JP 10062602 A wird ein ähnliches Schichtsystem beschrieben. Hier wird eine dünne Silberschicht mit mindestens 1,5 At.-% Goldbeimengung zwischen Oxidschichten, bestehend aus Zinnoxid und Indiumoxid sowie geringen Beimengungen anderer Oxide, eingebettet. Damit werden Schichten mit einem Flächenwiderstand von $4 - 20 \Omega_{sq}$ und hoher Durchlässigkeit bei 550 nm erhalten. Die erhöhten Kosten durch die Goldbeimengung und der relativ hohe Flächenwiderstand müssen als Nachteile angesehen werden.

[0008] In Thin Solid Films 341 beschreiben K. K. Choi und Koautoren ein Schichtsystem bestehend aus Indium-Zinn-Oxid gefolgt von einer Silberschicht und als Deckschicht wiederum Indium-Zinn-Oxid. Zur Verbesserung der Leitfähigkeit werden die Schichten aus Indium-Zinn-Oxid bei 200°C, die Silberschicht jedoch bei Raumtemperatur abgeschieden. Doch durch die Erwärmung vor Abscheidung der zweiten Schicht aus Indium-Zinn-Oxid werden die Eigenschaften der Silberschicht bezüglich optischer Transmission und elektrischer Leitfähigkeit ungünstig beeinflusst. Im besten Fall wurden Schichten mit einem Flächenwiderstand von $4 \Omega_{sq}$ und einer Transmission von 90% bei 550 nm erzielt.

[0009] Es ist weiterhin bekannt, dass bei spezieller Wahl der Materialien und Beschichtungsparameter transparente leitende Schichtsysteme mit $2,93 \Omega_{sq}$ und Transmissionswerten (gegen Luft gemessen) von 89,2 % bei 435 nm, 92,4 % bei 545 nm und 82,2 % bei 610 nm mit einer Gesamtschichtdicke von 86,5 nm hergestellt werden können. Dieser transparente Leiter besitzt für die drei genannten Wellenlängen einen mittleren Haakeschen Gütefaktor von $0,104 \Omega^{-1}$.

[0010] Im Displaybereich für grossflächige flache LCD-Displays oder Computermonitore mit Bildformaten vorzugsweise über 17" werden nun transparente Elektroden mit noch niedrigerem Flächenwiderstand bei gleichzeitig hoher Durchlässigkeit im sichtbaren Bereich, d. h. einem hohem Haakeschem Gütefaktor, benötigt. Dies ist durch die Bildgrösse, die hohe Auflösung und Pixelzahl sowie die höhere Geschwindigkeit dieser Displays bedingt. Diese Anforderungen können mit den bisher bekannten Verfahren nicht mehr erfüllt werden.

[0011] Die vorliegende Erfindung macht sich zur Aufgabe, die Nachteile des Standes der Technik zu beheben, insbesondere einen noch niedrigeren Flächenwiderstand bei einem hohen Haakeschen Gütefaktor zu erreichen.

[0012] Diese Aufgabe wird gelöst durch ein Schichtsystem nach Anspruch 1 sowie ein Verfahren nach Anspruch 6. Die abhängigen Patentansprüche beschreiben weitere bevorzugte Ausführungen der

Erfindung.

[0013] Ein erfindungsgemässes Schichtsystem nach Anspruch 1 umfasst mindestens 2 Oxidschichten und eine dazwischen gelagerte Silberschicht und weist einen Flächenwiderstand von weniger als $2,9 \Omega_{sq}$, vorzugsweise $2,5 \Omega_{sq}$ und geringer auf, bei einem über die Wellenlängen 435, 545 und 610 nm gemittelten Haackeschen Gütefaktor von grösser als $0,085 \Omega^{-1}$.

[0014] Dabei ist es aus Gründen der Farbneutralität günstig, wenn bei einem Flächenwiderstand von $2,5 \Omega_{sq}$ die optische Transmission bei 435 nm mindestens 89 %, bei 545 nm mindestens 88 % und bei 610 nm mindestens 75 % beträgt. Damit ist gewährleistet, dass die Beschichtung in Durchsicht möglichst neutral erscheint.

[0015] Besonders gute Ergebnisse werden erzielt, wenn die Dicke der beiden Oxidschichten vorteilhafterweise unter 50 nm, vorzugsweise zwischen 30 und 40 nm, und die Dicke der Silberschicht unter 20 nm, vorzugsweise bei 15 nm, gewählt wird.

[0016] Die Entspiegelungswirkung der Oxidschichten wird besonders gut, wenn die Oxidschicht neben Indium 5 bis 10 At.-% Cer enthält.

[0017] Die Stabilität der Silberschicht wird durch Beigabe von bis zu 10 Gew.-% Kupfer erhöht. Besonders wirksam zeigten sich Beigaben von 0,5 bis 3 % und insbesondere 0,5 bis 1 %.

[0018] Bei der Herstellung des beschriebenen Schichtsystems ist es entscheidend, wie in Anspruch 6 und weiteren abhängigen Ansprüchen beschrieben, dass die Aufbringung der zweiten Oxidschicht nicht mit reiner DC-Zerstäubung, sondern mit einer gepulsten DC-Zerstäubung oder mit einer AC-überlagerten DC-Zerstäubung erfolgt. Die AC-Überlagerung wird beispielsweise dadurch erzeugt, dass das Ausgangssignal über ein Filter auf die mit einer DC-Stromversorgung gespeiste Sputterquelle eingekoppelt wird. Eine weitere Möglichkeit besteht beispielsweise auch darin, die DC-Stromversorgung entsprechend zu modulieren oder zu lasten (choppern). Es sind also verschiedene Modulationen möglich.

[0019] Die AC-Frequenz sollte zwischen 1 und 50 MHz, vorzugsweise zwischen 10 und 20 MHz, liegen, um besonders gute Ergebnisse zu erreichen.

[0020] Im Weiteren wird mit Vorteil der AC-Anteil, definiert durch das Verhältnis der eingespeisten DC- und AC-Leistung, zwischen 10 und 90 %, vorzugsweise zwischen 30 und 50 %, eingestellt.

[0021] Besonders geeignet erwies sich eine totale Leistungsdichte (AC und DC) von 1 bis 3 W/cm^2 , vorzugsweise von 2 bis $2,2 \text{ W/cm}^2$.

[0022] Als Zerstäubungsmethode wird Magnetronspattern bevorzugt.

[0023] Die Vorteile dieses Verfahrens können wie folgt zusammengefasst werden:

[0024] Durch die Erhaltung der guten Leitfähigkeit der dünnen Silberschicht durch die Art der Aufbringung der zweiten Oxidschicht kann die optische Transmission

hoch gehalten werden. Ohne das erfindungsgemässe Vorgehen müsste zur Erzielung dieser Leitfähigkeit die Dicke der Silberschicht erhöht werden, was unvermeidlich zu einer deutlicheren Verringerung der Transmission und damit zu einer wesentlichen Verschlechterung des Haackeschen Gütefaktors führen würde.

[0025] Die Herstellung solcher Schichten an Hand des erfindungsgemässen Verfahrens soll nun an dem nachfolgenden Beispiel beschrieben werden.

[0026] Die Glas-Substrate aus herkömmlichem dünnen Floatglas oder Maschinenglas werden in herkömmlicher Weise gereinigt und dann in eine Zerstäubungsanlage eingebracht. Die Vakuumkammer wird abgepumpt und nach Erreichung des nötigen Vakuums mit der Aufstäubung der ersten Oxidschicht aus Indium- und Ceroxid begonnen. Diese Oxidschicht wird teilreaktiv von einem Oxidtarget abgestäubt, d. h. in einer Argonatmosphäre von ca. $2,2 \times 10^{-3} \text{ hPa}$ mit einer Beimischung von Sauerstoff von maximal 5 %. Dieser Zerstäubungsprozess ist ein reiner DC-Prozess. Typische Zerstäubungsraten sind 5 bis $8 \text{ nm} \times \text{min} \times \text{cm}^2 \text{ W}$. Anschliessend erfolgt als reiner nicht-reaktiver DC-Prozess das Aufstäuben der Silberschicht. Hier liegen die typischen Zerstäubungsraten bei 12 bis $15 \text{ nm} \times \text{min} \times \text{cm}^2 \text{ W}$. Ihm schliesst sich das Aufstäuben der zweiten Oxidschicht mit einer AC-überlagerten DC-Zerstäubung an. Dabei liegt der AC-Anteil, definiert durch das Verhältnis der eingespeisten DC- und AC-Leistung, zwischen 30 und 50 %. Die AC-Frequenz liegt bei 13,56 MHz. Nach Beendigung des Zerstäubungsprozesses werden die beschichteten Gläser durch eine Schleuse oder durch Fluten der Kammer an Luft ausgebracht. In einem anschliessenden Ätzprozess werden die Substrate dann strukturiert und zu Displays weiterverarbeitet.

[0027] Im folgenden ist die Erfindung in den Figuren 1 und 2 an Hand von Ausführungsbeispielen erläutert.

Fig. 1 zeigt schematisch und im Querschnitt ein erfindungsgemässes Schichtsystem.

Fig. 2 zeigt die im sichtbaren Spektralbereich gegen Luft gemessene optische Transmission eines erfindungsgemässen Schichtsystems mit einem Flächenwiderstand von $2,5 \Omega_{sq}$.

[0028] In der Fig. 1 bedeuten 1 das Glassubstrat, auf welches das erfindungsgemässe Schichtsystem aufgebracht wird, 2 eine Indium-Cer-Oxidschicht, 3 eine Kupfer-dotierte Silber-Schicht, und 4 eine abschliessende Indium-Cer-Oxidschicht.

[0029] Das Glassubstrat 1 ist z. B. ein handelsübliches Floatglas mit 1,1 mm Dicke. Es können aber auch andere Glasdicken und andere Gläser, z. B. Maschinenglas, benützt werden.

[0030] Darauf wird durch teilreaktive DC-Zerstäubung von einem Oxidtarget, bestehend aus vorzugsweise 90 bis 95 At.-% Indium und 5 bis 10 At.-% Cer,

eine Oxidschicht 2 mit der geometrischen Dicke von 30 bis 37 nm abgeschieden.

[0031] Auf diese Oxidschicht 2 wird eine Silberschicht 3 mit 0,5 bis 10 % Kupfer-Beimengung, vorzugsweise 0,5 bis 3% und insbesondere 0,5 bis 1% Kupfer, in einem reinen DC-Zerstäubungsprozess in einer Argonatmosphäre in einer Dicke von 15 nm aufgebracht.

[0032] Auf die Schicht 3 aus Silber/Kupfer wird direkt eine zweite Indium-Cer-Oxidschicht 4, ebenfalls mit der geometrischen Dicke von 30 bis 37 nm, abgeschieden. Dies erfolgt jedoch mit einem AC-überlagerten DC-Zerstäubungsprozess. Dabei liegt der AC-Anteil, definiert durch das Verhältnis der eingespeisten DC- und AC-Leistung, zwischen 10 und 90 %, vorzugsweise zwischen 30 und 50 %. Die AC-Frequenz liegt zwischen 1 und 50 MHz, vorzugsweise zwischen 10 und 20 MHz.

[0033] Optional kann nach der Silber/Kupfer-Schicht eine Schutzschicht aus Oxiden von Titan- oder Nickellegierungen mittels DC-Magnetron-Zerstäubung aufgebracht werden.

[0034] In der Fig. 2 ist die optische Durchlässigkeit (gemessen gegen Luft) eines erfindungsgemässen Schichtsystems mit einem Flächenwiderstand von $2,5 \Omega_{sq}$ in Abhängigkeit von der Wellenlänge im Spektralbereich 400 bis 800 nm dargestellt. Bei 435 nm werden 89,8 %, bei 545 nm 88,4 % und bei 610 nm 75,4 % erreicht. Der über diese drei Wellenlängen gemittelte Haackesche Gütefaktor beträgt $0,092 \Omega^{-1}$.

Patentansprüche

1. Leitendes transparentes Schichtsystem mit zwei Oxidschichten (2,4) und einer dazwischen gelagerten Silber-Schicht (3) auf einem Substrat (1), **dadurch gekennzeichnet**, dass bei einem Flächenwiderstand R_s von $< 2,9 \Omega_{sq}$, vorzugsweise $< 2,5 \Omega_{sq}$ und weniger, der mittlere Haackesche Gütefaktor des Schichtsystems für die Wellenlängen 435, 545 und 610 nm $(\Phi_{TC} = T^{10}/R_s) > 0,085 \Omega^{-1}$ beträgt.
2. Schichtsystem nach Anspruch 1, **dadurch gekennzeichnet**, dass bei einem Flächenwiderstand von $2,5 \Omega_{sq}$ die Durchlässigkeit T bei 435 nm mindestens 89 %, bei 545 nm mindestens 88 % und bei 610 nm mindestens 75% beträgt.
3. Schichtsystem nach einem der vorangehenden Ansprüche, **dadurch gekennzeichnet**, dass die Dicke des Schichtsystems < 100 nm, vorzugsweise 80 bis 90 nm beträgt, wobei die Dicke der Silberschicht (3) bei < 20 nm, vorzugsweise bei 15 nm, und die Dicke der beiden Oxidschichten (2,4) bei < 50 nm, vorzugsweise zwischen 30 und 40 nm, liegt.
4. Schichtsystem nach einem der vorangehenden

Ansprüche, **dadurch gekennzeichnet**, dass die Oxidschichten (2,4) Indium und Cer enthalten, vorzugsweise 90 bis 95 At.-% Indium und 5 bis 10 At.-% Cer.

5. Schichtsystem nach einem der vorangehenden Ansprüche, **dadurch gekennzeichnet**, dass die Silber-Schicht (3) bis zu 10 Gew.-% Kupfer enthält, vorzugsweise im Bereich 0,5 bis 3 % und insbesondere 0,5 bis 1 %.
6. Verfahren zur Herstellung eines leitenden transparenten Schichtsystems nach einem der vorangehenden Ansprüche, **dadurch gekennzeichnet**, dass bei der Aufbringung der zweiten Oxidschicht (4) eine gepulste DC-Zerstäubung oder eine AC-überlagerte DC-Zerstäubung verwendet wird.
7. Verfahren nach Anspruch 6, **dadurch gekennzeichnet**, dass eine AC-Überlagerung mit einer Frequenz zwischen 1 und 50 MHz, vorzugsweise zwischen 10 und 20 MHz vorgenommen wird.
8. Verfahren nach Anspruch 6 und 7, **dadurch gekennzeichnet**, dass der AC-Anteil, definiert durch das Verhältnis der eingespeisten DC- und AC-Leistung, zwischen 10 und 90 %, vorzugsweise zwischen 30 und 50 % liegt.
9. Verfahren nach Anspruch 6, 7 und 8, **dadurch gekennzeichnet**, dass die totale Leistungsdichte (AC und DC) im Bereich 1 bis 3 W/cm^2 , vorzugsweise aber bei 2 bis 2.2 W/cm^2 liegt.
10. Verfahren nach Anspruch 6 bis 9, **dadurch gekennzeichnet**, dass als Zerstäubungsverfahren Magnetronzerstäubung gewählt wird.
11. Leitendes transparentes Schichtsystem nach Anspruch 1 bis 5, **dadurch gekennzeichnet**, dass es nach dem Verfahren nach den Ansprüchen 6 bis 10 gefertigt wurde.
12. Leitendes transparentes Schichtsystem als transparente Elektroden für grossflächige Displays nach Anspruch 1 bis 5, **dadurch gekennzeichnet**, dass es nach dem Verfahren nach den Ansprüchen 6 bis 10 gefertigt wurde.

Fig.1

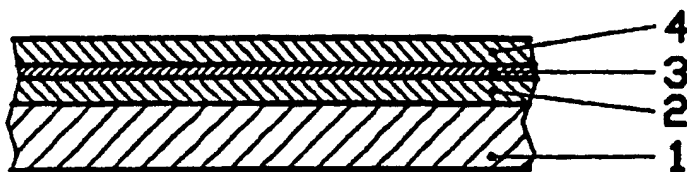
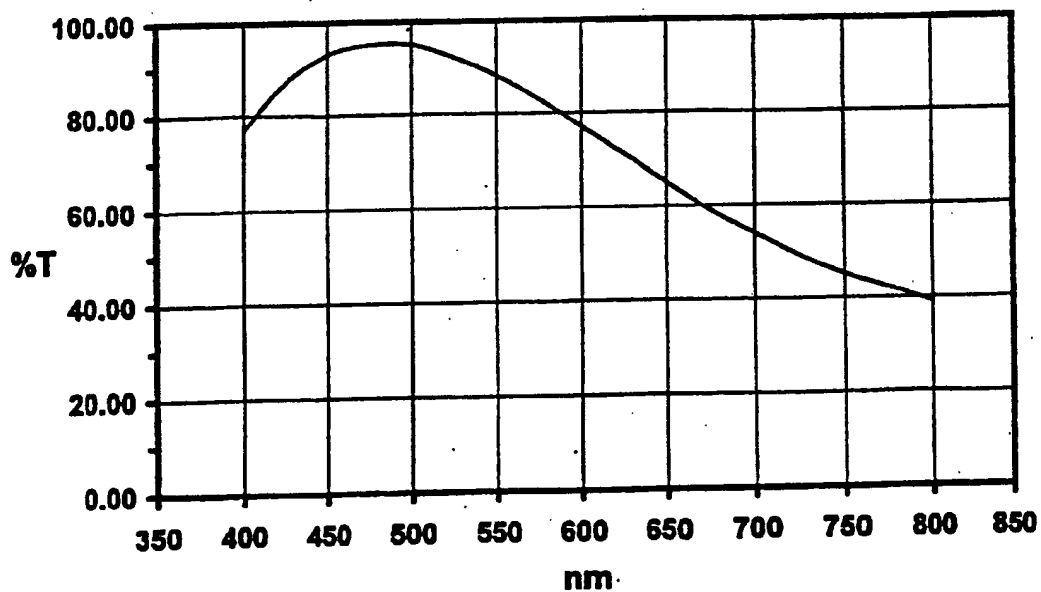


Fig.2





Europäisches
Patentamt

EUROPÄISCHER TEILRECHERCHENBERICHT

der nach Regel 45 des Europäischen Patent-
Übereinkommens für das weitere Verfahren als
europäischer Recherchenbericht gilt

Nummer der Anmeldung

EP 00 11 9591

EINSCHLÄGIGE DOKUMENTE			KLASSIFIKATION DER ANMELDUNG (Int.Cl.7)
Kategorie	Kennzeichnung des Dokuments mit Angabe, soweit erforderlich der maßgeblichen Teile	Betrifft Anspruch	
X	US 4 565 719 A (HIGGINS PATRICK K ET AL) 21. Januar 1986 (1986-01-21) * Anspruch 1; Tabelle 3 *	4	C03C17/36
X	GB 2 126 256 A (TOYODA CHUO KENKYUSHO KK) 21. März 1984 (1984-03-21) * Ansprüche 1-15; Beispiel 1 *	4-12	
A	DATABASE WPI Section Ch, Week 199015 Derwent Publications Ltd., London, GB; Class A32, AN 1990-113453 XP002159490 & JP 02 066158 A (UNITIKA LTD), 6. März 1990 (1990-03-06) * Zusammenfassung *	4	
P.A	GB 2 344 600 A (SONY CORP) 14. Juni 2000 (2000-06-14) * Ansprüche 1-4 *	4,5	
			RECHERCHIERTE SACHGEBIETE (Int.Cl.7)
			C03C
UNVOLLSTÄNDIGE RECHERCHE			
<p>Die Recherchenergebnisse sind der Auffassung, daß ein oder mehrere Ansprüche, den Vorschriften des EPU in einem solchen Umfang nicht entsprechen bzw. entsprechen, daß sinnvolle Ermittlungen über den Stand der Technik für diese Ansprüche nicht, bzw. nur teilweise, möglich sind.</p> <p>Vollständig recherchierte Patentansprüche:</p> <p>Unvollständig recherchierte Patentansprüche:</p> <p>Nicht recherchierte Patentansprüche:</p> <p>Grund für die Beschränkung der Recherche:</p> <p>Siehe Ergänzungsblatt C</p>			
Forschungsort		Abschlußdatum der Recherche	Prüfer
DEN HAAG		6. Februar 2001	Reedijk, A
<p>KATEGORIE DER GENANNTE DOKUMENTEN</p> <p>X : von besonderer Bedeutung allein betrachtet Y : von besonderer Bedeutung in Verbindung mit einer anderen Veröffentlichung derselben Kategorie A : technologischer Hintergrund O : nichtschriftliche Offenbarung P : Zwischenliteratur</p> <p>T : der Erfindung zugrunde liegende Theorien oder Grundsätze E : älteres Patentdokument, das jedoch erst am oder nach dem Anmeldedatum veröffentlicht worden ist D : in der Anmeldung angeführtes Dokument L : aus anderen Gründen angeführtes Dokument</p> <p>& : Mitglied der gleichen Patentfamilie, übereinstimmendes Dokument</p>			

EPO FORM 1503 03 92 (P04C06)



Vollständig recherchierte Ansprüche:
4-12

Nicht recherchierte Ansprüche:
1-3

Grund für die Beschränkung der Recherche:

Die geltenden Patentansprüche 1-3 sind auf ein Produkt, das (u.a.) mittels folgender Parameter definiert wird, zu beziehen:

PI: Haackesche Gütefaktor bei einem Flächenwiderstandsbereich.

Die Verwendung dieser Parameter muss im gegebenen Zusammenhang als Mangel an Klarheit im Sinne von Art. 84 EPÜ erscheinen. Es ist unmöglich, die vom Anmelder gewählten Parameter mit dem zu vergleichen, was der Stand der Technik hierzu offenbart. Der Mangel an Klarheit ist dergestalt, daß er eine sinnvolle vollständige Recherche unmöglich macht. Daher wurde die Recherche beschränkt auf die Teile mit Bezug auf Ausführungsbeispiele, wie sie in der Beschreibung auf Seite 4 und Patentansprüche 4-12 erwähnt sind.

**ANHANG ZUM EUROPÄISCHEN RECHERCHENBERICHT
 ÜBER DIE EUROPÄISCHE PATENTANMELDUNG NR.**

EP 00 11 9591

In diesem Anhang sind die Mitglieder der Patentfamilien der im obengenannten europäischen Recherchenbericht angeführten Patentedokumente angegeben.
 Die Angaben über die Familienmitglieder entsprechen dem Stand der Daten des Europäischen Patentamts am
 Diese Angaben dienen nur zur Unterrichtung und erfolgen ohne Gewähr.

06-02-2001

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EPO FORM P0401

Für nähere Einzelheiten zu diesem Anhang : siehe Amtsblatt des Europäischen Patentamts, Nr.12/82

MACHINE-ASSISTED TRANSLATION (MAT):

<p>(19) 【発行国】 日本国特許庁 (J P)</p> <p>(12) 【公報種別】 公開特許公報 (A)</p> <p>(11) 【公開番号】 特開平 5-230642</p> <p>(43) 【公開日】 平成 5 年 (1 9 9 3) 9 月 7 日</p> <p>(54) 【発明の名称】 スパッタ・ターゲット</p> <p>(51) 【国際特許分類第 5 版】 C23C 14/34 8414-4K H01J 37/08 9069-5E 37/30 9172-5E</p> <p>【審査請求】 未請求</p> <p>【請求項の数】 2</p> <p>【全頁数】 3</p> <p>(21) 【出願番号】 特願平 4-69981</p>	<p>(19)[ISSUING COUNTRY] Japan Patent Office (JP)</p> <p>(12)[GAZETTE CATEGORY] Laid-open Kokai Patent (A)</p> <p>(11)[KOKAI NUMBER] Unexamined Japanese Patent Heisei 5-230642</p> <p>(43)[DATE OF FIRST PUBLICATION] September 7, Heisei 5 (1993. 9.7)</p> <p>(54)[TITLE OF THE INVENTION] Sputter target</p> <p>(51)[IPC 5] C23C 14/34 8414-4K H01J 37/08 9069-5E 37/30 Z 9172-5E</p> <p>[REQUEST FOR EXAMINATION] No</p> <p>[NUMBER OF CLAIMS] 2</p> <p>[NUMBER OF PAGES] 3</p> <p>(21)[APPLICATION NUMBER] Japanese Patent Application Heisei 4-69981</p>
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(22) 【出願日】 (22)[DATE OF FILING]
平成4年(1992)2月21日 February 21, Heisei 4 (1992. 2.21)
日

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(57) 【要約】

(57)[ABSTRACT OF THE DISCLOSURE]

【目的】

[PURPOSE]

ターゲット材の加熱による割れを防止すること。

Prevent the crack by heat of a target material.

【構成】

[CONSTITUTION]

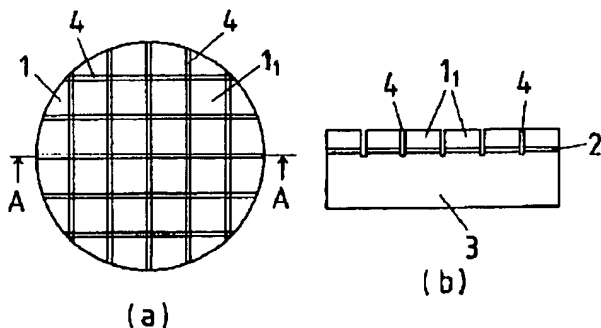
所要の元素を含むターゲット材 1 はボンディング材 2 によってバックングプレート 3 に固着されている。ターゲット材 1 にスリ割り溝（或いは切り込み、カッティングによる溝）4 を設け、ターゲット材 1 を多数の小区画 1₁ に分割する。ターゲット材の厚さ方向位置での伸びの差等が小さくなり、ターゲット材の割れを防ぐことができる。予め作っておいたターゲット材の小片をバックングプレートに貼り付けても良い。

The target material 1 containing a required element adheres to the backing plate 3 by the bonding material 2.

It provides the offset rate slot (or an incision, the slot by cutting) 4 in a target material 1, and partitions a target material 1 into many subsection 1₁.

The difference of the elongation in the thickness direction position of a target material etc. becomes smaller, it can prevent the crack of a target material.

It is sufficient to bond on a backing plate the fragment of the target material made beforehand.



【特許請求の範囲】**[CLAIMS]****【請求項 1】**

バックングプレートに固着されたターゲット材がスリ割り溝によって多数の小区画に分割されているか、又はターゲット材の多数の小片がタイル貼り状にバックングプレートに固着されていることを特徴とするスパッタ・ターゲット。

[CLAIM 1]

A sputter target, in which the target material which adhered to the backing plate is partitioned into many subsections by the offset rate slot, or many fragments of a target material adhere to the backing plate in the shape of a tile sticking.

【請求項 2】

請求項 1 のスパッタ・ターゲットを備えていることを特徴とするスパッタ型イオン源。

[CLAIM 2]

A sputter type ion source, which has the sputter target of Claim 1.

【発明の詳細な説明】**[DETAILED DESCRIPTION OF THE INVENTION]****【0001】****[0001]****【産業上の利用分野】**

本発明は、加熱によるターゲット材の割れを防いだスパッタ型イオン源等に用いるスパッタ・ターゲットに関する。

[INDUSTRIAL APPLICATION]

This invention relates to the sputter target which is used for the sputter type ion source which prevents the crack of the target material by heat.

【0002】**[0002]****【従来技術】**

スパッタ型イオン源、イオンビームスパッタリング装置等におけるスパッタ・ターゲットは、

[PRIOR ART]

As shown in the front elevation of FIG.4(a), and sectional drawing of said (b) in the A-A, the sputter target in a sputter type ion source, an

図4 (a) の正面図、そのA-A線での同 (b) の断面図に示すように、プラズマイオン、イオンビームでスパッタされる所要の元素を含む板状のターゲット材1をボンディング材2によってバックングプレート3に固着して構成されている。ターゲット材1はスパッタ時、周囲のプラズマとプラズマイオン、或いはイオンビームの衝撃により加熱されるから、その冷却のためにバックングプレート3は冷却ホルダーに取り付けられている。

ion-beam-sputtering apparatus, etc. adheres to a backing plate 3, and the bonding material 2 comprises the tabular target material 1 containing the required element by which a sputter is carried out from a plasma ion and an ion beam.

A target material 1 is heated by the shock of the surrounding plasma, a plasma ion, or an ion beam at the time of a sputter, therefore

The backing plate 3 is attached to the cooling holder for the cooling.

【0003】

【発明が解決しようとする課題】

かかるスパッタ・ターゲットのサイズが直径50mm程度の大きさのものになると、ターゲット材1が熱伝導の悪い材質の場合には、スパッタ時、ターゲット材1の厚み方向位置での熱膨張による伸びに大きな差が生じる等の理由により、ターゲット材が割れてしまうことがある。

[0003]

[PROBLEM TO BE SOLVED BY THE INVENTION]

When the size of this sputter target becomes the size which is diameter 50 mm degree and a target material 1 is the bad material of a heat conduction, a target material may break for the reasons of a big difference arising on the elongation by the thermal expansion in the thickness direction position of a target material 1 at the time of a sputter.

【0004】

本発明は、プラズマやイオンによる加熱で、ターゲット材が割れることを防止したスパッタ・ターゲットの提供を目的とするものである。

[0004]

This invention aims to provide the sputter target which prevents the crack of a target material by heating by the plasma or an ion.

【0005】

【課題を解決するための手段】
本発明は、スパッタ・ターゲットにおいて、バックングプレートに固着されたターゲット材がスリ割り溝によって多数の小区画に分割されているか、又は、ターゲット材の多数の小片がタイル貼り状にバックングプレートに固着されていることを主たる特徴とするものであり、そして、かかるスパッタ・ターゲットをスパッタ型イオン源に用いたことを特徴とするものである。

【0006】

【作用】
ターゲット材がスリ割り或いは小片のタイル貼り形式により、小さく分割されているから、各分割域における加熱による熱膨張が他の分割域に波及することがないから、ターゲット材の厚み方向位置での伸びの差等が小さく抑えられ、スパッタ・ターゲットの割れが生じない。そして、かかるターゲットをスパッタ型イオン源に用いることにより、イオンビームが安定して引出せる。

【0007】

[0005]

[MEANS TO SOLVE THE PROBLEM]

As for this invention, in a sputter target, the target material which adhered to the backing plate is partitioned into many subsections by the offset rate slot, or many fragments of a target material adhere to a backing plate in the shape of a tile sticking.

And this sputter target is used for the sputter type ion source.

It is characterized by the above-mentioned.

[0006]

[OPERATION]

The target material is small partitioned according to an offset rate or the tile sticking form of a fragment, therefore

Since the thermal expansion by the heat in each partition region does not affect another partition region, the difference of the elongation in the thickness direction position of a target material etc. is restrained small, the crack of a sputter target does not arise.

And it can pull out an ion beam with stability by using this target for a sputter type ion source.

[0007]

【実施例】

本考案の実施例について図面を参照して説明する。図1 (a)、(b) はスリ割り形式によるスパッタ・ターゲットの正面図及びそのA-A線での断面図である。バックングプレート3にボンディング材2で固着されたターゲット材1に、基盤の目状にスリ割り溝（スリ割らないし切り込み、カッティングによる溝）4を設け、ターゲット材1を一辺が10mm程度の多数の小区画1₁に小分割する。スリ割り溝4は、少なくともターゲット材部分に形成されていれば充分であるが、図1 (b) ではバックングプレート3にまで達しているものを示している。

【0008】

図2 (a)、(b) はタイル貼り形式のスパッタ・ターゲットの正面図とそのA-A線での断面図であり、予めターゲット材を一辺が10mm程度の小片1₂に分割しておき、かかる多数のターゲット材の小片1₂をボンディング材2によって、タイル貼り形式でバックングプレート3に固着する。

【0009】

このように、ターゲット材1は、何れも一辺が10mm程度の小区画ないしは小片1₁、1₂に分

[EXAMPLES]

With reference to drawing, it demonstrates the Example of this design.

FIG. 1 (a), (b) is the front elevation of the sputter target by offset rate form, and sectional drawing in the A-A.

It provides the offset rate slot (an offset rate or an incision, slot by cutting) 4 in the target material 1 which adhered to the backing plate 3 by the bonding material 2 in a grid pattern, and one side small-partitions a target material 1 into many subsection 1₁ which are 10 mm level.

If the offset rate slot 4 is formed in the target material part at least, it is enough.

However, FIG.1(b) shows what has reached even the backing plate 3.

[0008]

FIG. 2 (a), (b) is the front elevation and sectional drawing in an A-A of the sputter target of tile sticking form.

One side partitions the target material into fragment 1₂ which is 10 mm level beforehand, and it adheres fragment 1₂ of the target material of these many to a backing plate 3 in tile sticking form by the bonding material 2.

[0009]

Thus, the target material 1 is partitioned into the subsection whose one side is all 10 mm level or fragment 1₁, and 1₂, therefore

割されているから、ターゲット材1のスパッタ時に発熱、加熱が生じて、ターゲット材等の熱膨張は、各小区画、小片1₁、1₂内に限定されて、他の部分には波及せず、各小区画、小片1₁、1₂の厚み方向位置での伸びの差は小さく抑えられるから、ターゲット材に割れが発生しない。

【0010】

プラズマ・スパッタ型負イオン源では、スパッタ・ターゲット表面で発生した負イオンをイオン源の出口にビームとして集束させるために、スパッタ・ターゲットの表面を球面状にえぐった形とする場合がある。このようにすると、ターゲット材の周辺部では、その厚みは5mm程度に達し、スパッタ材が割れる可能性が高くなる。図3(a)及び(b)は、かかる負イオン源のスパッタ・ターゲットにスリ割りを実施したものの正面図と、そのA-A線での断面図である。図1に示したものと同様に、表面が球面状にえぐられたターゲット材1をボンディング材2によってバックングプレート3に固着しておき、スリ割り溝4を形成することによって、ターゲット材1を多数の小区画に分割する。加熱によるスパッタ・ターゲットの割れが防止で

Even if heat generation and heat arise at the time of the sputter of a target material 1, thermal expansion, such as a target material, is limited in each subsection, fragment 1₁, and 1₂, it does not affect the other part but the difference of the elongation in the thickness direction position of each subsection, fragment 1₁, and 1₂ is restrained small, therefore

A crack does not occur in a target material.

[0010]

In the source of a plasma sputter type negative ion, in order to converge as a beam the negative ion generated on the sputter target surface on the outlet of an ion source, it may make the surface of a sputter target into the form scooped out to the spherical shape.

If it does in this way, in the periphery part of a target material, the thickness will amount to 5 mm level, possibility that a sputter material will break becomes higher.

Although FIG.3(a) and (b) implemented the offset rate at the sputter target of this source of a negative ion, they are a front elevation and sectional drawing in the A-A.

It partitions a target material 1 into many subsections by the surface's adhering to the backing plate 3 the target material 1 scooped out by the spherical shape by the bonding material 2 like what was shown in FIG. 1, and forming the offset rate slot 4.

It can prevent the crack of the sputter target by heat, therefore

It becomes possible to pull out a beam more stably than an ion source.

きるから、イオン源より安定にビームを引出すことが可能になる。図2と同様に、タイル貼り形式を実施してもよい。

It is sufficient to implement tile sticking form like FIG. 2.

【0011】

[0011]

【発明の効果】

[ADVANTAGE OF THE INVENTION]

本発明は以上説明したように、ターゲット材が小さく分割されているから、スパッタ時に、ターゲット材における厚み方向の熱膨張による伸びの差等を小さく抑えることができ、ターゲット材の割れを防止することができる。

As for this invention, as explained above, the target material is partitioned small, therefore At the time of a sputter, it can restrain small the difference of the elongation by the thermal expansion of the thickness direction in a target material etc., and can prevent the crack of a target material.

【0012】

[0012]

そして、本発明によるスパッタ・ターゲットをイオン源に用いることにより、イオンビームを安定に引出すことができ、特に表面が球面状にえぐられたスパッタ・ターゲットの場合に効果的である。

And in the case of the sputter target with which it could pull out the ion beam stably, and particularly the surface was scooped out by the spherical shape, it is effective by using the sputter target by this invention for an ion source.

【図面の簡単な説明】

[BRIEF DESCRIPTION OF THE DRAWINGS]

【図1】

[FIG. 1]

本発明の実施例の正面図及び断面図である。

It is the front elevation and sectional drawing of an Example of this invention.

【図2】

[FIG. 2]

他の実施例の正面図及び断面図である。

It is the front elevation and sectional drawing of another Example.

【図 3】

更に他の実施例の正面図及び断面図である。

[FIG. 3]

Furthermore, it is the front elevation and sectional drawing of another Example.

【図 4】

従来のスパッタ・ターゲットの正面図及び断面図である。

[FIG. 4]

It is the front elevation and sectional drawing of a sputter target of the past.

【符号の説明】

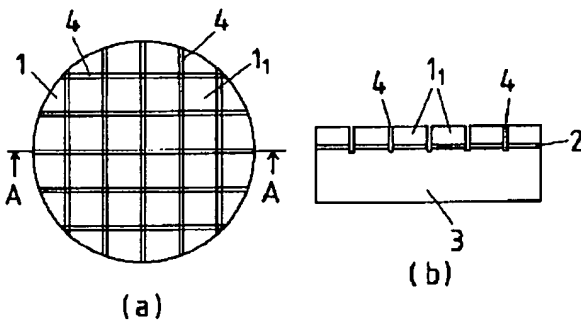
- 1,1₁,1₂ ターゲット材
- 2 ボンディング材
- 3 バックングプレート
- 4 スリ割り溝

【DESCRIPTION OF SYMBOLS】

- 1,1₁,1₂ target material
- 2 Bonding material
- 3 Backing plate
- 4 Offset rate slot

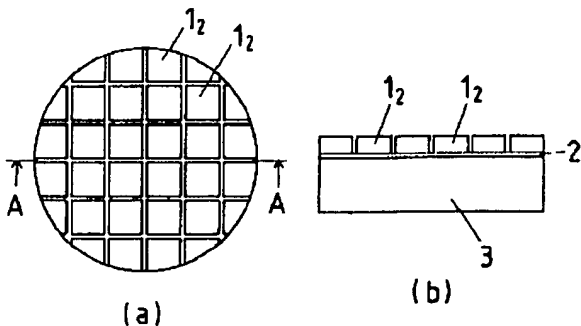
【図 1】

[FIG. 1]



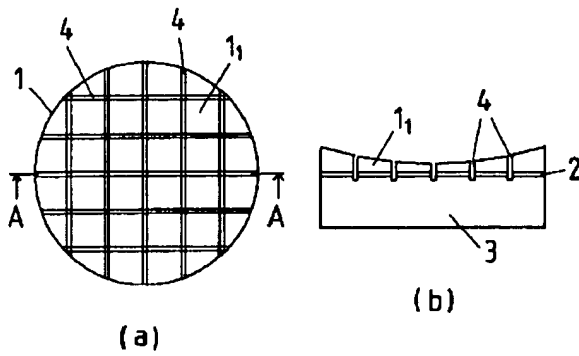
【図 2】

[FIG. 2]



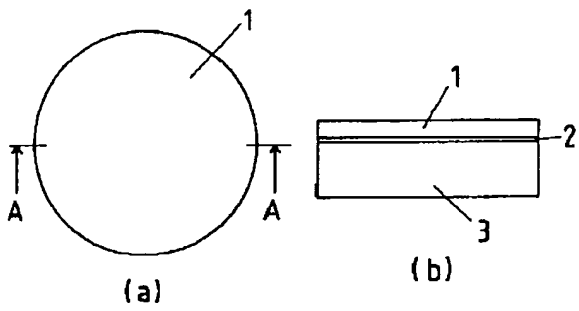
【図 3】

[FIG. 3]



【図 4】

[FIG. 4]



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SPUTTERING TARGET

Publication number: JP5230642

Publication date: 1993-09-07

Inventor: BABA TAKASHI; TAKEYAMA KUNIIHIKO

Applicant: NISSIN HIGH VOLTAGE CO LTD

Classification:

- international: **C23C14/34; H01J37/08; H01J37/30; C23C14/34; H01J37/08; H01J37/30; (IPC1-7): C23C14/34; H01J37/08; H01J37/30**

- european:

Application number: JP19920069981 19920221

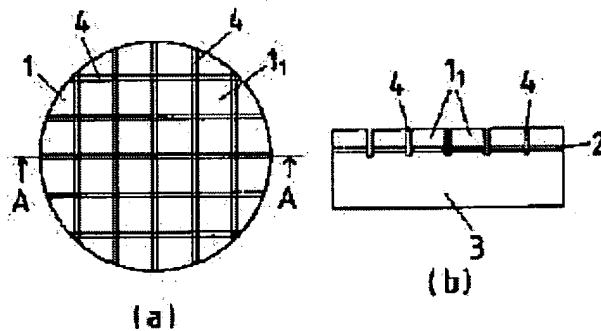
Priority number(s): JP19920069981 19920221

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Abstract of JP5230642

PURPOSE: To prevent the cracks of a target material caused by heating.

CONSTITUTION: A target material 1 containing required elements is fixed to a backing plate 3 by a bonding material 2. The target material 1 is provided with slit grooves (or grooves by notching or grooves by cutting) 4, and the target material 1 is divided into many small divisions 11. The difference of the elongation in the position in the thickness direction of the target material or the like are reduced, by which the cracks of the target material can be prevented. The small pieces of the target material which has previously been made may be adhered to the backing plate.



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(19) 日本国特許庁 (J P)

(12) 公開特許公報 (A)

(11) 特許出願公開番号

特開平5-230642

(43) 公開日 平成5年(1993)9月7日

(51) Int.Cl. ⁵	識別記号	庁内整理番号	F I	技術表示箇所
C 2 3 C 14/34		8414-4K		
H 0 1 J 37/08		9069-5E		
37/30	Z	9172-5E		

審査請求 未請求 請求項の数2(全3頁)

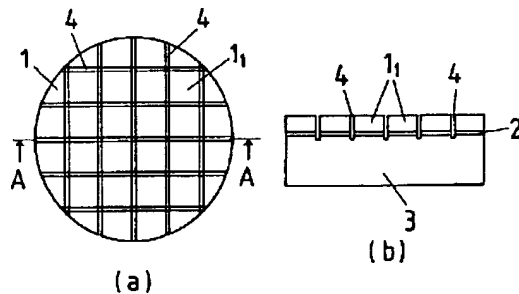
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		(74) 代理人	弁理士 成田 擴其

(54) 【発明の名称】 スパッタ・ターゲット

(57) 【要約】

【目的】 ターゲット材の加熱による割れを防止すること。

【構成】 所要の元素を含むターゲット材1はボンディング材2によってバックングプレート3に固着されている。ターゲット材1にスリ割り溝(或いは切り込み、カッティングによる溝)4を設け、ターゲット材1を多数の小区画1₁に分割する。ターゲット材の厚さ方向位置での伸びの差等が小さくなり、ターゲット材の割れを防ぐことができる。予め作っておいたターゲット材の小片をバックングプレートに貼り付けても良い。



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【特許請求の範囲】

【請求項1】 バックリングプレートに固着されたターゲット材がスリ割り溝によって多数の小区画に分割されているか、又はターゲット材の多数の小片がタイル貼り状にバックリングプレートに固着されていることを特徴とするスパッタ・ターゲット。

【請求項2】 請求項1のスパッタ・ターゲットを備えていることを特徴とするスパッタ型イオン源。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明は、加熱によるターゲット材の割れを防いだスパッタ型イオン源等に用いるスパッタ・ターゲットに関する。

【0002】

【従来の技術】スパッタ型イオン源、イオンビームスパッタリング装置等におけるスパッタ・ターゲットは、図4(a)の正面図、そのA-A線での同(b)の断面図に示すように、プラズマイオン、イオンビームでスパッタされる所要の元素を含む板状のターゲット材1をボンディング材2によってバックリングプレート3に固着して構成されている。ターゲット材1はスパッタ時、周囲のプラズマとプラズマイオン、或いはイオンビームの衝撃により加熱されるから、その冷却のためにバックリングプレート3は冷却ホルダーに取り付けられている。

【0003】

【発明が解決しようとする課題】かかるスパッタ・ターゲットのサイズが直径50mm程度の大きさのものになると、ターゲット材1が熱伝導の悪い材質の場合には、スパッタ時、ターゲット材1の厚み方向位置での熱膨張による伸びに大きな差が生じる等の理由により、ターゲット材が割れてしまうことがある。

【0004】本発明は、プラズマやイオンによる加熱で、ターゲット材が割れることを防止したスパッタ・ターゲットの提供を目的とするものである。

【0005】

【課題を解決するための手段】本発明は、スパッタ・ターゲットにおいて、バックリングプレートに固着されたターゲット材がスリ割り溝によって多数の小区画に分割されているか、又は、ターゲット材の多数の小片がタイル貼り状にバックリングプレートに固着されていることを主たる特徴とするものであり、そして、かかるスパッタ・ターゲットをスパッタ型イオン源に用いたことを特徴とするものである。

【0006】

【作用】ターゲット材がスリ割り或いは小片のタイル貼り形式により、小さく分割されているから、各分割域における加熱による熱膨張が他の分割域に波及することがないから、ターゲット材の厚み方向位置での伸びの差等が小さく抑えられ、スパッタ・ターゲットの割れが生じない。そして、かかるターゲットをスパッタ型イオン源

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に用いることにより、イオンビームが安定して引出せる。

【0007】

【実施例】本考案の実施例について図面を参照して説明する。図1(a)、(b)はスリ割り形式によるスパッタ・ターゲットの正面図及びそのA-A線での断面図である。バックリングプレート3にボンディング材2で固着されたターゲット材1に、基盤の目状にスリ割り溝(スリ割りないし切り込み、カッティングによる溝)4を設け、ターゲット材1を一辺が10mm程度の多数の小区画1₁に小分割する。スリ割り溝4は、少なくともターゲット材部分に形成されていれば充分であるが、図1(b)ではバックリングプレート3にまで達しているものを示している。

【0008】図2(a)、(b)はタイル貼り形式のスパッタ・ターゲットの正面図とそのA-A線での断面図であり、予めターゲット材を一辺が10mm程度の小片1₂に分割しておき、かかる多数のターゲット材の小片1₂をボンディング材2によって、タイル貼り形式でバックリングプレート3に固着する。

【0009】このように、ターゲット材1は、何れも一辺が10mm程度の小区画ないしは小片1₁、1₂に分割されているから、ターゲット材1のスパッタ時に発熱、加熱が生じて、ターゲット材等の熱膨張は、各小区画、小片1₁、1₂内に限定されて、他の部分には波及せず、各小区画、小片1₁、1₂の厚み方向位置での伸びの差は小さく抑えられるから、ターゲット材に割れが発生しない。

【0010】プラズマ・スパッタ型負イオン源では、スパッタ・ターゲット表面で発生した負イオンをイオン源の出口にビームとして集束させるために、スパッタ・ターゲットの表面を球面状にえぐった形とする場合がある。このようにすると、ターゲット材の周辺部では、その厚みは5mm程度に達し、スパッタ材が割れる可能性が高くなる。図3(a)及び(b)は、かかる負イオン源のスパッタ・ターゲットにスリ割りを実施したものの正面図と、そのA-A線での断面図である。図1に示したものと同様に、表面が球面状にえぐられたターゲット材1をボンディング材2によってバックリングプレート3に固着しておき、スリ割り溝4を形成することによって、ターゲット材1を多数の小区画に分割する。加熱によるスパッタ・ターゲットの割れが防止できるから、イオン源より安定にビームを引出すことが可能になる。図2と同様に、タイル貼り形式を実施してもよい。

【0011】

【発明の効果】本発明は以上説明したように、ターゲット材が小さく分割されているから、スパッタ時に、ターゲット材における厚み方向の熱膨張による伸びの差等を小さく抑えることができ、ターゲット材の割れを防止することができる。

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【0012】そして、本発明によるスパッタ・ターゲットをイオン源に用いることにより、イオンビームを安定に引出すことができ、特に表面が球面状にえぐられたスパッタ・ターゲットの場合に効果的である。

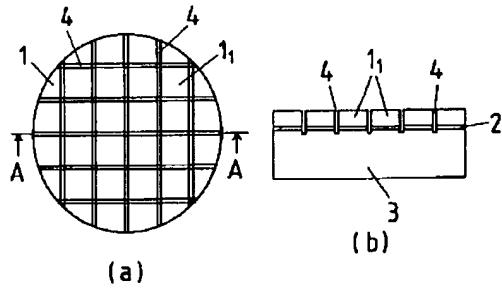
【図面の簡単な説明】

【図1】本発明の実施例の正面図及び断面図である。

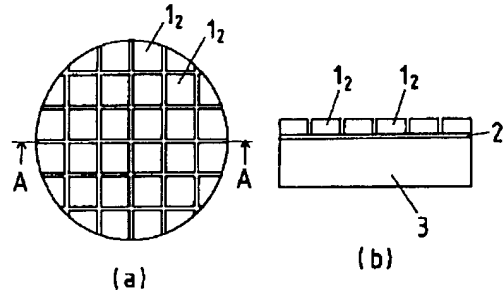
【図2】他の実施例の正面図及び断面図である。

【図3】更に他の実施例の正面図及び断面図である。

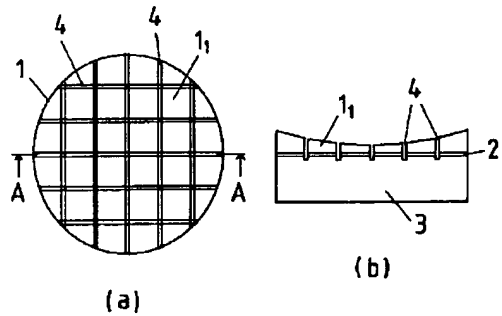
【図1】



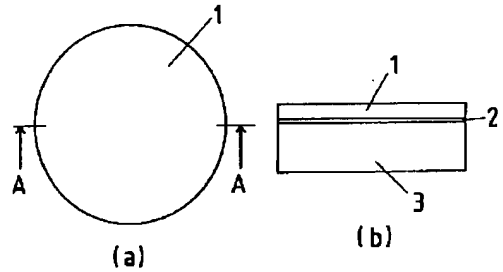
【図2】



【図3】



【図4】



【図4】従来のスパッタ・ターゲットの正面図及び断面図である。

【符号の説明】

- 1, 1₁, 1₂ ターゲット材
- 2 ボンディング材
- 3 バッキングプレート
- 4 スリ割り溝



PATENT ABSTRACTS OF JAPAN

(11) Publication number: 07224379 A

(43) Date of publication of application: 22.08.95

(51) Int. Cl. C23C 14/34

(21) Application number: 06017653

(22) Date of filing: 14.02.94

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ISHIKAWA MICHIO
OTA YOSHIFUMI
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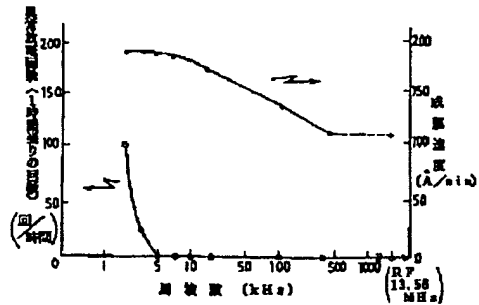
(54) SPUTTERING METHOD AND DEVICE THEREFOR

(57) Abstract:

PURPOSE: To form a film at a high rate without generating an abnormal discharge for a long time at the time of sputtering a conductive target in a gaseous reactant atmosphere by DC sputtering by applying a positive potential on a negative-potential target in the form of a pulse.

CONSTITUTION: A substrate and a conductive target are opposed in a vacuum treating chamber, and the target is sputtered in a gaseous reactant atmosphere by DC sputtering to form a thin film on the substrate. In this case, a device with a power source to apply a positive potential on a negative potential connected to the target is used, and a positive potential is applied on the negative-potential target in the form of a pulse at the frequency of 5-400kHz to conduct sputtering. The electron in the plasma is attracted by the positive potential to neutralize the plus ion accumulated on the insulator and high-resistance film, and the abnormal discharge due to an arc discharge is not generated.

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(51)Int.Cl. ⁶	識別記号	庁内整理番号	F I	技術表示箇所
C 2 3 C 14/34		R 8414-4K		
		A 8414-4K		
		M 8414-4K		

審査請求 未請求 請求項の数 4 O L (全 5 頁)

(21)出願番号 特願平6-17653
 (22)出願日 平成6年(1994)2月14日



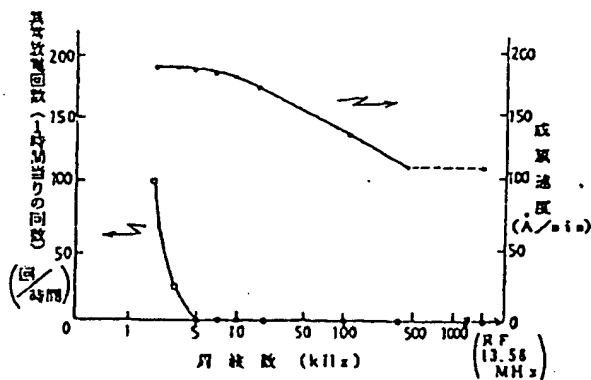
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 最終頁に続く

(54)【発明の名称】 スパッタ方法およびそのスパッタ装置

(57)【要約】

【目的】 反応ガス雰囲気中で直流スパッタ法により導電性ターゲットにスパッタリングを行っても高速成膜が可能であり、かつ、長時間スパッタリングを行っても異常放電のないスパッタ成膜が出来るスパッタ方法。

【構成】 反応ガス雰囲気中で直流スパッタ法により導電性ターゲットにスパッタリングを行って基板上に薄膜を形成するスパッタ法において、負電位の導電性ターゲットに正電位を一定の周波数でパルス状に印加しながらスパッタリングする。



PTO 99-4732
 S.T.I.C. Translations Branch

【特許請求の範囲】

【請求項1】 反応ガス雰囲気中で直流スパッタ法により導電性ターゲットにスパッタリングを行って基板上に薄膜を形成するスパッタ法において、負電位の導電性ターゲットに正電位を周波数5～400kHzでパルス状に印加しながらスパッタリングすることを特徴とするスパッタ方法。

【請求項2】 前記導電性ターゲットはSi、Al、Ta、Ti、C、ITO、ZnO、SnO₂またはこれらの合金であり、また、反応ガスはN₂、O₂、H₂、NH₃、CO、CO₂、CH₄、C₂H₂、H₂Oのいずれか1つまたは2つ以上の混合ガスであることを特徴とする請求項第1項に記載のスパッタ方法。

【請求項3】 真空処理室内に基板と導電性ターゲットを対向させて設け、反応ガス雰囲気中で直流スパッタ法により導電性ターゲットにスパッタリングを行って基板上に薄膜を形成するスパッタ装置において、負電位に正電位を周波数5～400kHzでパルス状に印加する電源を導電性ターゲットに接続したことを特徴とするスパッタ装置。

【請求項4】 前記導電性ターゲットはSi、Al、Ta、Ti、C、ITO、ZnO、SnO₂またはこれらの合金であり、また、反応ガスはN₂、O₂、H₂、NH₃、CO、CO₂、CH₄、C₂H₂、H₂Oのいずれか1つまたは2つ以上の混合ガスであることを特徴とする請求項第3項に記載のスパッタ装置。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明はスパッタ方法およびそのスパッタ装置に関し、更に詳しくは、反応ガス雰囲気中で導電性ターゲットにスパッタリングを行うスパッタ方法およびそのスパッタ装置に関する。

【0002】

【従来の技術】従来から、反応ガス雰囲気中で導電性ターゲットにスパッタリングを行って基板上に誘電体膜、光学膜、保護膜等の膜を成膜するスパッタ法に用いられるスパッタ装置としては、真空処理室内に基板とターゲットカソードを対向して設け、該ターゲットカソードに導電性ターゲットを装着し、真空処理室内を所定の雰囲気とした状態でターゲットに所定電圧を印加してターゲットにスパッタリングを行う装置が知られている。

【0003】そして、スパッタリング時の電源としては直流電源、または例えば13.56MHzの高周波電源を用いている。

【0004】

【発明が解決しようとする課題】前記従来のスパッタ装置を用いて基板上に誘電体膜、光学膜、保護膜等の膜を成膜する際、高周波電源を用いるRFスパッタリングは長時間異常放電なしに成膜することが可能であるが、成膜速度が遅いという問題がある。

【0005】また、前記従来のスパッタ装置を用いて基板上に誘電体膜、光学膜、保護膜等の膜を成膜する際、直流電源を用いるDCスパッタリングは成膜速度は速いが、スパッタリングの経時と共に、ターゲット表面に絶縁物や高抵抗膜が堆積するため、異常放電を起こすという問題がある。

【0006】スパッタリング中に異常放電が発生すると欠陥のない均質な膜を成膜する上で致命的な悪影響を及ぼす。

10 【0007】従って、従来のスパッタ装置では通常RFスパッタリングを行っているが、高速成膜を必要とする場合は、DCスパッタリングを行い、ターゲット表面に絶縁物や高抵抗膜が堆積しないようにターゲット全面に亘ってエロージョン化するようにしているが、異常放電を十分に除去することが出来ない。

20 【0008】本発明はかかる問題点を解消し、反応ガス雰囲気中でスパッタリングを行っても高速成膜が可能であり、かつ、長時間スパッタリングを行っても異常放電のないスパッタ成膜の可能なスパッタ方法およびそれに用いるスパッタ装置を提供することを目的とする。

【0009】

【課題を解決するための手段】本発明のスパッタ方法は、反応ガス雰囲気中で直流スパッタ法により導電性ターゲットにスパッタリングを行って基板上に薄膜を形成するスパッタ法において、負電位の導電性ターゲットに正電位を周波数5～400kHzでパルス状に印加しながらスパッタリングすることを特徴とする。

【0010】また、前記導電性ターゲットはSi、Al、Ta、Ti、C、ITO、ZnO、SnO₂またはこれらの合金とし、また、反応ガスはN₂、O₂、H₂、NH₃、CO、CO₂、CH₄、C₂H₂、H₂Oのいずれか1つまたは2つ以上の混合ガスとしてもよい。

【0011】本発明のスパッタ装置は、真空処理室内に基板と導電性ターゲットを対向させて設け、反応ガス雰囲気中で直流スパッタ法により導電性ターゲットにスパッタリングを行って基板上に薄膜を形成するスパッタ装置において、負電位に正電位を周波数5～400kHzでパルス状に印加する電源を導電性ターゲットに接続したことを特徴とする。

【0012】また、前記導電性ターゲットはSi、Al、Ta、Ti、C、ITO、ZnO、SnO₂またはこれらの合金とし、また、反応ガスはN₂、O₂、H₂、NH₃、CO、CO₂、CH₄、C₂H₂、H₂Oのいずれか1つまたは2つ以上の混合ガスとしてもよい。

【0013】

【作用】反応ガス雰囲気中で導電性ターゲットに直流電源より直流電圧を印加し、スパッタリングを行うとターゲットはスパッタされて基板上に薄膜が形成される。

【0014】長時間連続してスパッタリングを行うと、ターゲット上に堆積した絶縁物や高抵抗膜上に不活性ガスまたは反応ガスのプラス(+)イオンが蓄積される。このプラス(+)イオンの電荷がターゲット間、エロージョン部、アース電極等とアーク放電を引き起こして異常放電の原因となる。

【0015】この異常放電でターゲット材、絶縁物、高抵抗膜が粒子状となって飛散し、基板上に付着し、成膜された薄膜の欠陥となる。

【0016】ターゲットに直流電圧を印加してDCスパッタリングを行う際、負電位のターゲットに正電位を一定の周波数でパルス状に印加すると、正電位によりプラズマ中の電子を引き寄せ、ターゲット上に堆積した絶縁物、高抵抗膜上に蓄積するプラス(+)イオンの電荷を中和し、アーク放電による異常放電を防止する。

【0017】その際、ターゲットに印加する正電位の印加時間は負電位の時間に比べて極めて短くとも効果があるため、成膜速度は直流電流のみによる成膜速度より数%の減少となる程度であり、この成膜速度は高周波13.56MHzスパッタ時の成膜速度よりも高い。

【0018】

【実施例】以下添付図面に従って本発明の実施例について説明する。

【0019】図1は、スパッタ装置の1例を示すもので、図中、1は真空処理室を示す。

【0020】真空処理室1を外部のクライオポンプのような真空ポンプ等の真空排気系2にバルブ3を介して接続し、該真空処理室1内の真空度を調節自在とすると共に、真空処理室1内に基板4とターゲットカソード5とを対向に配置し、該ターゲットカソード5の前面に導電性ターゲット6をロウ材でボンディングするようにした。また、該真空処理室1内にガス導入管7を介してガスポンプ等のガス供給源8よりスパッタガスを導入するようにした。図示例ではガス導入管7を分岐し、一方の分岐管9aにガス圧調節弁10aを介して不活性ガス(例えばアルゴンガス)のガス供給源8aを接続し、他方の分岐管9bにガス圧調節弁10bを介して反応ガス(例えば窒素ガス)のガス供給源8bを接続した。

【0021】また、ターゲットカソード5に直流電源11をパルスユニット12を介して接続し、パルスユニット12を調節して導電性ターゲット6に負電位と正電位を所定の周波数でパルス状に印加するようにした。

【0022】また、ターゲットカソード5の背面側にマグネトロンスパッタのためのマグネット13を配置して、ターゲットカソード5に取り付けられた導電性ターゲット6の表面にマグネトロンスパッタに必要な磁場を与えるようにした。

【0023】次に、図1装置を用いて本発明のスパッタ方法の具体的実施例について説明する。

【0024】実施例1

先ず、真空処理室1内に基板4と、ターゲットカソード5に導電性ターゲット6としてケイ素(Si)製ターゲットを装着した。

【0025】続いて、真空処理室1内を真空排気系2により 6.7×10^{-4} Pa (5×10^{-3} Torr)に排気した後、真空処理室1内にガス供給源8aからのアルゴン(Ar)ガスと、ガス供給源8bからの窒素(N₂)ガスとから成る反応ガスをガス導入管7を介して導入して、該真空処理室1内のスパッタ圧が 6.7×10^{-4} Pa (5×10^{-3} Torr)となるようにした。次に基板4上に形成される窒化ケイ素(SiN_x)の屈折率が2.03となるようにArガスとN₂ガスの流量をそれぞれ100sccmと50sccmに調整した後、DCマグネトロンスパッタ法により導電性ターゲット6に直流電源11より直流電力3kWを印加し、負電位のターゲット6に正電位をパルスユニット12より周波数を2kHzから400kHzに変化させながらパルス状(図2参照)に印加し、スパッタリングを行って基板4上に膜厚900Åの窒化ケイ素(SiN_x)膜を形成した。尚、負電位のターゲットにパルス状に印加する正電位の印加時間は周波数10kHzまでの場合は10μsecとし、周波数10kHzを超えて100kHzまでの場合は5μsecとし、周波数100kHzを超えた場合は1μsecとした。また、DCマグネトロンスパッタ時の磁場強度を250Oeとした。

【0026】そして異常放電回数と成膜速度を負電位の導電性ターゲット6に印加する正電位の周波数毎に測定した。得られた測定結果を図3に示す。

【0027】図3から明らかなように負電位に印加する正電位の周波数の増加に伴い、異常放電回数は減少し、周波数が5kHz以上ではその回数はほとんど0になり、これ以上の周波数で正電位を負電位のターゲットに印加すれば異常放電が発生しないことが分かる。また、負電位に印加する正電位の周波数が400kHz(時間1μsec)の場合は、通常の高周波電源(13.56MHz)とほぼ同じ成膜速度となり、400kHz以上の周波数では正電位の印加は意味がない。従って、負電位のターゲットに印加する正電位の周波数範囲は5~400kHzであることが確認された。

【0028】また、周波数範囲5~400kHzでの成膜速度は高周波電源(13.56MHz)よりも高い成膜速度である。

【0029】反応ガス雰囲気中で直流スパッタ法により導電性ターゲットにスパッタリングを行うと、ターゲット上の非エロージョン部分に絶縁物または高抵抗膜が堆積し、従来の直流電源でターゲットに負の電位を放電し続けると、絶縁物上にプラス(+)の電荷が蓄積し、ターゲット、エロージョン部、アース電極との間でアーク放電を起こし、電荷を放出する。その結果異常放電が発生することになるが、本発明では図2に示すように負電位に正電位を周期的に、即ち一定の周波数で印加することにより前記の電荷を補償して異常放電の発生を防止するようにしている。

【0030】実施例2

反応ガスとして酸素(O₂)ガスを用い、スパッタ圧を6.7×10⁻¹Pa(5×10⁻³Torr)とし、直流電力を2kWとし、基板上に形成される酸化ケイ素(SiO_x)の屈折率が1.46となるように酸素流量を調整した以外は前記実施例1と同様の方法でDCマグネトロンスパッタ法によりターゲットにスパッタリングを行って基板上に膜厚1000Åの酸化ケイ素(SiO_x)膜を形成した。

【0031】そして異常放電回数を負電位のターゲットに印加する正電位の周波数毎に測定した。得られた測定結果を図4に示す。

【0032】図4から明らかなように負電位に印加する正電位の周波数は約5kHzで異常放電をほぼ防止し得ることが分かる。

【0033】尚、図4におけるSiN_xの測定結果(黒丸印)は前記実施例1の測定結果を参考のために記載したものである。

【0034】実施例3

導電性ターゲットとしてグラファイト(C)を用い、反応ガスとしてメタン(CH₄)ガスを用い、スパッタ圧を6.7×10⁻¹Pa(5×10⁻³Torr)とし、直流電力を2kWとした以外は前記実施例1と同様の方法でDCマグネトロンスパッタ法によりターゲットにスパッタリングを行って基板上に膜厚200Åのダイヤモンドライクカーボン(DLC)膜を形成した。

【0035】そして異常放電回数を負電位のターゲットに印加する正電位の周波数毎に測定した。得られた測定結果を図4に示す。

【0036】図4から明らかなように負電位に印加する正電位の周波数は約5kHzで異常放電をほぼ防止し得ることが分かる。

【0037】前記実施例1, 2, 3の結果から、導電性ターゲット材料、放電状態によりプラス(+)の電荷の蓄積量が異なるため、異常放電はターゲットに印加する周波数に大きく依存する。従って、周波数5kHz以上であれば、種々のターゲット材料でもスパッタリング中の異常放電を大幅に減少することが出来ることになる。

【0038】実施例4

導電性ターゲットとしてアルミニウム(Al)、チタン(Ti)、タンタル(Ta)、またはこれらの合金例えばAl-2at%Tiを用い、反応ガスとして窒素(N₂)、酸素(O₂)、水素(H₂)、一酸化炭素(CO)、二酸化炭素(CO₂)、アンモニア(NH₃)、水(H₂O)を単独、或いは2種以上を用い、これらを種々組み合わせし、その他の条件を前記実施例1と同様にしてDCマグネトロンスパッタ法により基板上に酸化物、窒化物、炭化物のいずれかの薄膜を形成したが、負電位に印加する正電位が周波数5kHz以上ではスパッタリング中に異常放電の発生は見られなかった。

【0039】また、前記実施例1では導電性ターゲットにSiを用い、反応ガスとして窒素ガスを用いて基板上に窒化ケイ素(SiN_x)膜を形成したが、導電性ターゲットとしてSiの代わりにIn₂O₃、SnO₂、焼結体(ITO)、酸化亜鉛(ZnO)、酸化スズ(SnO₂)を用い、反応ガスとして窒素ガスの代わりに酸素(O₂)、一酸化炭素(CO)、二酸化炭素(CO₂)、水(H₂O)等の酸素を含むガス、またはそのガスに水素ガス(H₂)混合したガスを用い、負電位のターゲットに印加する正電位の周波数を5~400kHzとした直流スパッタ法(DCマグネトロンスパッタ法)により基板上に透明導電膜であるITO、ZnO、SnO₂の薄膜を形成することが出来る。

【0040】図1装置では負電位に一定の周波数で正電位をパルス状に印加する電源装置として直流電源とパルス電源の組み合わせた装置としたが、これに限定されるものではなく、図2に示すような負電位に正電位が周期的(パルス状)に印加される一体型の電源装置としてもよい。

【0041】

【発明の効果】本発明のスパッタ方法によるときは、反応ガス雰囲気中で、負電位の導電性ターゲットに正電位を一定の周波数でパルス状に印加しながらスパッタリングを行うようにしたので、スパッタリング中にターゲット上に堆積した絶縁物、高抵抗膜上に蓄積するプラスイオンの電荷を中和することが出来て、アーク放電による異常放電を防止しながら長時間に亘って速い成膜速度で基板上に欠陥のない均質な薄膜を形成することが出来る効果がある。

【0042】また、本発明のスパッタ装置によるときは、導電性ターゲットに負電位に正電位を一定の周波数でパルス状に印加する電源を接続するようにしたので、アーク放電による異常放電を防止しながら長時間に亘って速い成膜速度で基板上に欠陥のない均質な薄膜を形成することが出来るスパッタ装置を提供する効果がある。

【図面の簡単な説明】

【図1】本発明のスパッタ方法を実施するためのスパッタ装置の1例の概略断面側面図。

【図2】図1装置による導電性ターゲットに印加される電位のモデル図。

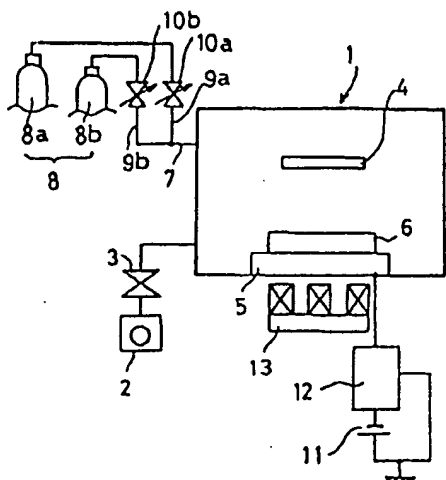
【図3】本発明スパッタ方法の1実施例における成膜時の周波数と異常放電回数との関係、並びに周波数と成膜速度との関係を示す特性線図。

【図4】本発明スパッタ方法の他の実施例における成膜時の周波数と異常放電回数との関係を示す特性線図。

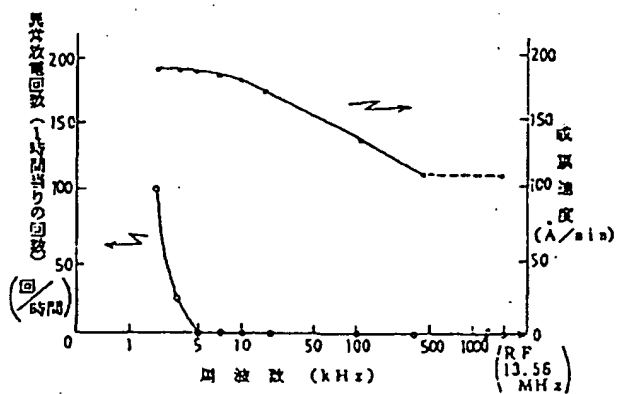
【符号の説明】

- 1 真空処理室、 2 真空排気系、 4 基板、 6 導電性ターゲット、 7 ガス導入管、 8 スパッタガス供給源、 11 直流電源、 12 パルスユニット。

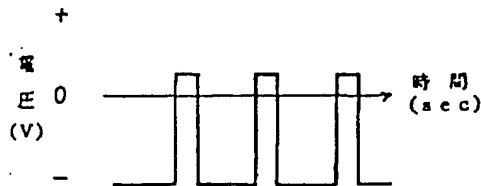
【図1】



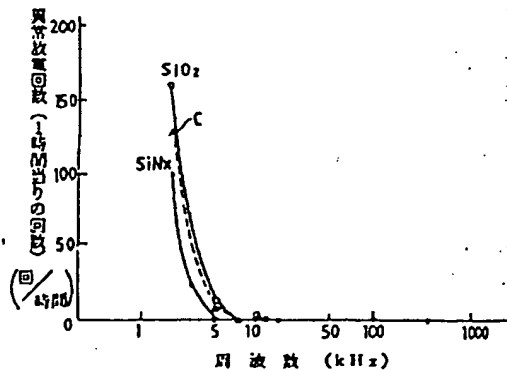
【図3】



【図2】



【図4】



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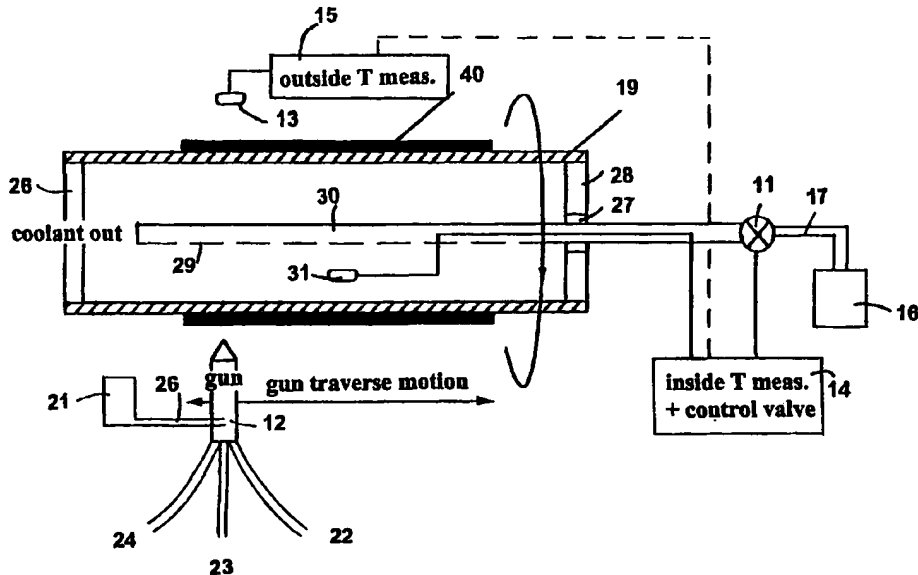
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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification ⁶ : C23C 4/10, 4/12, 14/34</p>	<p>A1</p>	<p>(11) International Publication Number: WO 99/61674 (43) International Publication Date: 2 December 1999 (02.12.99)</p>
<p>(21) International Application Number: PCT/EP99/03599 (22) International Filing Date: 26 May 1999 (26.05.99) (30) Priority Data: 98870120.7 26 May 1998 (26.05.98) EP (71) Applicant (for all designated States except US): UNIVER-SITEIT GENT [BE/BE]; St. Pietersnieuwstraat 25, B-9000 Gent (BE). (72) Inventors; and (75) Inventors/Applicants (for US only): HOSTE, Serge [BE/BE]; Gaverse steenweg 579, B-9280 Merelbeke (BE). PERSYN, Frans [BE/BE]; Oude boomgaardlaan 23, B-9080 Lochrist (BE). VAN DRIESSCHE, Isabel [BE/BE]; Wareslagstraat 94, B-9250 Waasmunster (BE). (74) Agents: BIRD, William et al.; Bird Goën & Co., Termerestraat 1, B-3020 Winksele (BE).</p>		<p>(81) Designated States: JP, US, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published With international search report.</p>

(54) Title: SPRAYING METHOD TO FORM A THICK COATING AND PRODUCTS OBTAINED



(57) Abstract

A method and an apparatus for spraying materials onto a substrate to produce a coating thereon is described which allows very thick layers of complex metal oxides to be produced. The apparatus and method are particularly suitable for producing superconducting coatings.

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SPRAYING METHOD TO FORM A THICK COATING AND PRODUCTS OBTAINED

The present invention relates an apparatus and a method of spraying to form a coating on flat or curved substrates, for example, either as part of the direct
5 formation of metallic or ceramic coatings such as superconductive or piezo-
electric layers or for the production of targets for sputtering magnetrons having
coatings which are precursors of such layers.

TECHNICAL BACKGROUND

10 From EP-A-286 135 it is known to flame spray complex ceramic materials
onto a substrate such as a tape to form a superconducting layer. It is suggested to
pre-heat the substrate to temperatures above 540°C and to cool the coating
slowly. It is further recommended to treat the coating in an atmosphere containing
one of the components of the superconducting ceramic. An oxy-acetylene flame is
15 used for the flame spraying. Thickness of up to 3 mm are described.

It is also known from US 5,196,400 to plasma spray a coating onto a target
for use in a sputtering magnetron to sputter a Y-Ba-CuO superconductor coating.
Deposition of only a thin target coating of 0.5 mm is reported.

The production of superconducting powders using flame spraying is
20 reported in US 5,140,005. An oxy-acetylene flame is used. It is tacitly accepted
that the high temperature of the flame changes the stoichiometric ratios of the
components and that this has to be compensated by increasing the more volatile
components in the original mixtures. US 5,045,365 describes a method of cooling
a oxy-acetylene flame-sprayed substrate with water. Without special precautions,
25 water cooling is unsuitable for superconductors due to the water vapour produced.

EP-A-355 736 describes production of flat targets with metal oxides up to
a layer thickness of 3 mm. WO 98/0833 describes the production of < 20 micron
thick layers of superconducting metal oxide mixtures.

The article by Murakami et. al. "Rapidly Solidified Thick Deposit Layers
30 of Fe-C-Mo Alloys by Flame Spraying" describes up to 1.5 mm thick rapidly

cooled thick layers of Fe-C-Mo alloys by flame spraying. Special precautions were taken to produce dense layers, e.g. direct application of cryogenic gas on the coating during application.

EP-A-586 809 describes the metal spraying application of a layer of
5 relatively homogeneous material (nickel coated silicon) which is much easier to handle than the heterogeneous oxide mixtures contemplated by the present invention. Layer thicknesses of up to 8 mm are described but 3 to 5 mm is preferred. Various layers are proposed including a Ni-Al layer for improving adhesion between the deposited layer and the substrate. A Ni-Al adhesion
10 promoter is known from DE-A-33 18 828.

Plasma spraying of superconducting materials is described in EP-A-288711 up to a thickness of 250 micron.

It is an object of the present invention to provide an apparatus and a method of spraying heterogeneous metal oxides to form a ceramic coating on flat
15 or curved substrates.

It is a further object of the present invention to provide an apparatus and a method of spraying heterogeneous metal oxides to form a thick walled ceramic coating on flat or curved substrates which is structurally sound.

It is a further object of the present invention to provide an apparatus and a
20 method of spraying to form a thick walled coating of a superconducting ceramic material.

It is still a further object of the present invention to provide an apparatus and a method of spraying suitable for forming a thick walled ceramic coating on flat or curved targets to be used in a sputtering magnetron.

25 It is still another object of the present invention to provide a method of producing a (magnetron) vacuum sputtering target as well as the target itself with improved thermal and electrical conductivity and high mechanical strength using a spraying process employing dedicated powder formulations.

30 SUMMARY OF THE INVENTION

One aspect of the present invention is to provide a substrate with a coating of a combination of metal oxides having a thickness greater than 3 mm more preferably greater than 5 mm and most preferably greater than 8 mm. Preferably, the coating is deposited by spraying, e.g. flame or plasma spraying. Preferably, the substrate is cylindrical and is more preferably is suitable as a cylindrical target substrate for a sputtering magnetron. The combination of oxides preferably comprises at least a superconductive precursor or a superconductor. The thermal conductivity of the deposited material is preferably between 1 and $5 \text{ Wm}^{-1}\text{K}^{-1}$. When deposited on a steel substrate the thermal conductivity of the composite preferably lies within the range 25 to $125 \text{ Wm}^{-1}\text{K}^{-1}$. These values are particularly preferred for $\text{YBa}_2\text{Cu}_3\text{O}_7$ coatings. Preferably, an adhesion promoter layer is applied onto the substrate before application of the coating of the metal oxide combination. The adhesion promoter may be a layer of Ni-Al or a layer of an In-alloy, for example. The deposited coating is preferably impact resistant, e.g. withstands impact of a 0.036 kg steel ball from a height of 2 metres. Preferably, about 20% or up to 30% of a noble metal is included in the oxide material to improve electrical and thermal properties of the deposited layer. The noble metal is preferably silver. The noble metal may be included as a salt or oxide, e.g. silver nitrate or silver oxide, in the material to be sprayed. Preferably, the electrical resistivity of the deposited layer is lower than $15 \times 10^{-6} \text{ Ohm.m}$, more preferably lower than 10×10^{-6} and most preferably less than $5 \times 10^{-6} \text{ Ohm.m}$. Values below $1 \times 10^{-6} \text{ Ohm.m}$ can be achieved. Up to 30% of a noble metal such as silver may be added to lower the resistivity. These values are particularly preferred for $\text{YBa}_2\text{Cu}_3\text{O}_7$ coatings.

The electrical, thermal and mechanical properties of the coating deposited in accordance with the present invention should be sufficient that the deposited layer can be applied to a suitable substrate by means of a sputtering magnetron preferably at a static sputtering deposition speed of at least 5 nm/minute, more preferably, at 20 nm/minute and most preferably at at least 40 nm/minute.

When a superconductor precursor or a superconductive material is

deposited, at least 10% of the coating is in the superconducting phase, more preferably 15%. This may be assisted by a subsequent limited thermal treatment, e.g. 3 hours and 940°C, after deposition.

5 The present invention also includes a method of depositing by spraying a superconductor precursor layer onto a cylindrical target for a sputtering magnetron, the layer having a thickness of at least 3 mm, and at least 10% of the layer being in a superconductive phase. The present invention also includes a method of depositing by spraying a layer onto a substrate, the layer having a thickness of at least 5 mm, and the coating comprising metal oxides.

10 In accordance with one aspect of the present invention a flame spraying apparatus is provided for depositing a metal oxide combination onto a substrate to produce a coating thereon, comprising: a burner for producing a flame; an inlet for feeding material to be sprayed through the flame, the flame imparting a temperature to the material to be sprayed of 1500°C or less, preferably 1200°C or less. Preferably the temperature imparted may be a little higher than the melting
15 point of the powder to be sprayed, e.g. 600 to 1000°C for some metal oxides. Preferably, the thickness of the deposited coating is greater than 3 mm more preferably greater than 5 mm and most preferably greater than 8 mm.

Another aspect of the present invention is to provide a flame spraying
20 apparatus for depositing a metal oxide combination onto a substrate to produce a coating thereon, comprising: a flame spraying gun; and a cooling system for the substrate, the cooling system including a device for bringing a cryogenic fluid into contact with the substrate. Preferably, the thickness of the deposited coating is greater than 3 mm more preferably greater than 5 mm and most preferably greater
25 than 8 mm. The input material for the sprayer may be a liquid solution of soluble compounds (e.g. nitrates) which decompose thermally into ceramic component oxides, liquid slurries of the ceramic components or metal powders, or dry metal or ceramic powders or precursors of the ceramic components, e.g. nitrates, of such powders.

30 The present invention may provide a method of flame spraying a

combination of metal oxide materials onto a substrate to produce a coating thereon, comprising: generating a flame; feeding the material to be sprayed through the flame, the flame imparting a temperature to the material to be sprayed of 1500°C or less, preferably 1200°C or less. Preferably the temperature imparted
5 may be a little higher than the melting point of the powder to be sprayed, e.g. 600 to 1000°C for some metal oxides.

The present invention may also provide a method of flame spraying metal oxide combinations onto a substrate to produce a coating thereon, comprising: generating a flame for spraying the materials; and cooling the substrate by
10 bringing a cryogenic fluid into contact with the substrate.

The present invention may also provide a method of flame spraying a superconducting ceramic material or a precursor thereof onto a substrate to produce a coating thereon, comprising: generating a flame for spraying the ceramic material; depositing the coating on the substrate; and during deposition of
15 the coating, cooling the substrate so that the solidified coating thereon has a temperature between room temperature (~25°C) and 150°C, preferably room temperature (~25°C) and 100°C. Water or cryogenic fluid cooling are particularly preferred.

One linking concept between the above methods and apparatus is control
20 of the total heat energy into the spraying/coating system. This can be achieved by careful control of parameters which influence the energy input such as spraying distance, spray head traverse speed, rotation speed of a cylindrical substrate, powder dwell time in the hot exit plume from the spray head, particle velocity exiting the spray head, cooling method and rate of cooling the substrate during
25 coating deposition.

The present invention also includes a method of reconditioning a target for a sputtering magnetron by flame spraying or atmospheric plasma spraying as well as a reconditioned target as made in accordance with the method. The target material or coating is preferably a ceramic coating, in particular a
30 superconducting or superconductor precursor coating.

The final coating is preferably a metallic or ceramic layer, in particular a superconducting or piezo-electric layer or a precursor thereof. The present invention includes a method of spray drying a liquid to form a powder suitable for flame spraying. The spray dried powder may be sintered. The present invention
5 also includes a manufacturing method for depositing a coating on a substrate comprising the steps of: spray drying a precursor liquid to form a powder and flame spraying the powder to form a coating on a substrate. The substrate may be a target for a sputtering magnetron and the final coating may be sputtered onto a final substrate in the sputtering magnetron. The ceramic powder may be sintered after
10 the spray drying step. The flame of the flame spray gun preferably imparts a temperature to the powder to be sprayed of 1500°C or less, preferably 1200°C or less. Preferably the temperature imparted may be a little higher than the melting point of the powder to be sprayed, e.g. 600 to 1000°C for some metal oxides. During flame spraying the target is preferably cooled by bringing a cryogenic
15 fluid into contact with the target. In particular the cooling device should maintain the solidified coating at a temperature between room temperature (~25°C) and 150°C, more preferably between room temperature (~25°C) and 100 °C.

The present invention includes an apparatus for spray drying a liquid to form a powder suitable for flame spraying. The present invention may also
20 include an apparatus for depositing a coating on a substrate comprising: a spray drier for drying a precursor liquid to a powder, and a flame sprayer for flame spraying the powder to form a coating on a substrate. The substrate may be a target for a magnetron. Additionally, a sputtering magnetron for sputtering the final coating onto the final substrate using the target may be provided. The flame
25 of the flame spray gun preferably imparts a temperature to the powder to be sprayed of slightly above the melting point of the sprayed material. Preferably the temperature imparted is 1500°C or less, preferably 1200°C or less. Temperatures of 600 to 850 °C may be suitable for some metal oxides. In the flame sprayer a cooling system for the target is preferably provided, the cooling system including
30 a device for bringing a cryogenic fluid into contact with the target. In particular

the cooling device should maintain the solidified coating at a temperature between room temperature ($\sim 25^{\circ}\text{C}$) and 150°C , more preferably between room temperature ($\sim 25^{\circ}\text{C}$) and 100°C .

The above methods may be used, for example, either as part of the direct
5 formation of superconductive or piezo-electric layers on the substrate, e.g. a tape, or for the production of coatings on targets for use in a sputtering magnetron to sputter a superconducting layer onto a final substrate. The present invention may provide oxide sputtering targets supporting very high power dissipation thus enabling high sputter deposition rates of at least 50 nm/min.

10 The dependent claims describe additional individual embodiments of the present invention. The present invention will now be described with reference to the following drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

15 Fig. 1 is a schematic representation of a flame spraying apparatus in accordance with one embodiment of the present invention.

Fig. 2 is a schematic representation of a flame spraying apparatus in accordance with another embodiment of the present invention.

20 Fig. 3 is a schematic representation of a spray drying apparatus in accordance with another embodiment of the present invention.

DESCRIPTION OF THE ILLUSTRATIVE EMBODIMENTS

The present invention will be described with reference to certain specific
embodiments and with reference to certain specific drawings but the invention is
25 not limited thereto but only by the claims. In particular, the present invention will mainly be described with reference to the deposition of a superconductor precursor or superconductive coatings but the invention is not limited thereto but may be used advantageously with other heterogeneous coatings such as ceramic coatings, particularly those having special properties such as piezo-electric
30 coatings and in particular coatings which contain components which can be

degraded by high temperatures or which are more volatile than other components. More particularly the present invention will be described with reference to the manufacture of $\text{YBa}_2\text{Cu}_3\text{O}_7$ superconducting powders and coatings but the invention is not limited thereto but only by the claims. Further one way of

5 carrying out the present invention will be described with reference to low temperature flame spraying but the present invention is not limited thereto. By carrying out the invention in accordance with the processing details and principles described below thick layer (greater than 3 mm, more preferably greater than 5 mm and most preferably greater than 8 mm) metal oxide combination coatings

10 suitable for use as a sputtering magnetron target have been applied by oxy-acetylene flame spraying with water cooling or by atmospheric pressure or low-pressure plasma spraying to substrates including cylindrical substrates used in rotating cathode magnetrons. During plasma spraying gasses may be used such as argon or mixtures of argon and other gasses to shield the plasma spray. Also the

15 present invention will mainly be described with reference to an input to the flame spraying head of spray dried powder. The present invention is not limited thereto but includes other forms of input materials such as a mixture of the metal oxides, including slurries thereof or mixtures of precursors of metal oxides such as metal nitrates as well as slurries and solutions thereof.

20 Fig. 1 is a schematic diagram of the flame spraying apparatus 10 in accordance with a first embodiment of the present invention. A flame spraying gun is represented schematically at 12. The gun 12 may be a commercially available flame spraying gun as for instance available from Sulzer Metco, Westbury, NY, USA or a high velocity oxy-fuel spraying gun available from the

25 same company. The gun 12 may be provided with an air pincher. The gun 12 may be fed with fuel gas in pipe 22, oxygen in pipe 23 and gun cooling air in pipe 24. Additional gases may be supplied to the gun 12 as described for instance in US 5,273,957 or EP-A-413 296. Material to be coated is fed in powder or liquid form, e.g. a dry powder, a slurry of the powder and a liquid or in solution, to the

30 gun via conduit 26 from hopper 21. Gun 12 is mounted on a drive (not shown)

which provides the necessary movements of the gun 12 to coat the substrate 19. When substrate 19 is a cylindrical target, for instance, for a rotating cathode magnetron, this may be rotated and the movements of the gun 12 may be simple reciprocating movements parallel to the axis of the target 19. If the substrate 19 is a flat rectangular or circular plate, the movements may be provided by a suitable robot and may be complex, e.g. including rotational cycloidal motions. For rapid deposition several guns 12 may spray the same substrate 19 at the same time.

The fuel gas for the gun 12 may be selected from one of acetylene, propylene, hydrogen or similar fuels but the present invention is not necessarily limited thereto. Particularly preferred in one embodiment of the present invention is a fuel with a lower calorific value such as one of ethylene, natural or town gas, butane or propane as these provide a lower temperature flame than acetylene and butane is particularly preferred as it gives a stable easily controllable flame and is considered safer than acetylene if powders containing copper compounds are used. It is generally accepted that oxy-acetylene flames have temperatures of 2000 °C and more. It is preferred in accordance with an embodiment of the present invention if the flame of the flame spraying gun 12 imparts a temperature only sufficient to just melt the powder to be sprayed. Temperatures of 1500 °C or less and preferably 1200 °C or less are preferred and temperatures between 600 and 1000 °C may be more preferable. These low flame temperatures minimise decomposition of the ceramic powder components during flame spraying. Moreover, they limit the impact of evaporation of the materials to be flame sprayed and allow a deposition efficiency of more than 80%, i.e. more than 80% of the solid mass originally introduced into the gun 12, becomes attached to the substrate 19. Mechanically stable, scratch resistant flame sprayed coatings are produced with these low temperatures.

The gun 12 is preferably held at 7 to 15 cm from the substrate 19 to be coated but this depends upon the size of the flame. Similar coatings have been obtained using both oxy-acetylene flame spraying and plasma spraying. Attention must be paid to the energy taken up by the sprayed particles during the spraying

and the transfer of this energy to the substrate. Intensive cooling of the substrate is preferred which may be on the side of the substrate remote from the deposited layer and/or on the same side. By altering the velocity of the particles in the flame or plasma the dwell time therein may be altered, thus limiting the energy uptake
5 by the particles.

The material of substrate 19 preferably has a high melt temperature and a high thermal conductivity and when the substrate 19 is to be used as a target for a sputtering magnetron a good electrical conductivity is preferable. It is also preferred if the thermal expansion of the substrate material is similar to that of the
10 ceramic coating to be applied. In accordance with embodiments of the present invention low temperature flame spraying and/or intense cooling of the substrate 19 allows the use of substrates 19 with a thermal expansion coefficient up to at least twice or down to at least a half of the thermal expansion coefficient of the ceramic coating. A non-limiting list of suitable materials may be steel, iron,
15 stainless steel, copper or copper alloys, however the low temperature flame spraying process in accordance with the present invention, either independently or in combination with intense cryogenic cooling of the substrate 19, allows other materials to be used such as paper, cardboard or polymeric materials. Preferably, the substrate 19 should be free of grease and dry before deposition. Preferably,
20 the outer surface of metals is sand blasted and then lapped with abrasive materials. Buffer layers between the substrate and the sprayed coating may be used such as Ni-Al or an In-alloy. These may be applied by flame or plasma spraying before application of the metal oxide coating. .

Where the substrate 19 is rigid it may be mounted in a suitable jig. For
25 example, a cylindrical substrate 19 is preferably mounted in a rotating device such as a lathe. The substrate 19 may be held by rotatable chucks at each end thereof. The temperature of the solidified flame sprayed coating 40 on the surface of the substrate 19 is preferably measured by a temperature sensor 13, 15. The sensor head 13 is preferably a remote sensing optical head which is not in contact with
30 the surface 40 of the flame sprayed coating. The temperature to be measured is of

the solidified coating 40 and not that of the coating immediately on impacting the substrate 19 which may have a higher temperature. Hence, the temperature sensor 13 is preferably mounted so that it lags behind the impact position of the flame sprayed materials a little. In addition a temperature sensor 31 may be provided
5 inside the substrate 19 for further control of the deposition process. Control of deposition temperature is an important aspect of the present invention. Control of temperature affects the amount of thermal stress in the coating, a low stress reducing the possibility of cracks forming in the coating.

In accordance with one embodiment of the present invention a means for
10 intense cooling of the substrate 19 is provided. This is preferably a cryogenic cooler comprising a supply 16 of cryogenic fluid and a delivery system 11, 14, 17, 29, 30. The delivery system may be adapted to the form of the substrate 19. For example, for a cylindrical substrate 19 the cooling device may be a conduit 17 for supplying the cryogenic fluid to a control valve 11, a conduit 30 with regularly
15 spaced holes 29 for distribution of the cryogenic fluid inside the substrate 19 and a control device 14 for receiving the output of the temperature sensor 13, 15 and for controlling the operation of the control valve 11 so as to maintain the surface temperature of the solidified coating 40 to within a certain range. Particularly preferred is a temperature range from room temperature (25 to 30°C) to 150 °C
20 and more preferably room temperature to 100 °C. These low temperatures avoid thermal stresses between the coating 40 and the substrate 19 providing a good bond and good coating density, hardness and scratch resistance thus helping to ensure the long term stability of such a coating. Using a cryogenic fluid such as liquid nitrogen (77 °K) is quite advantageous and economical as it does not
25 require the complication of perfectly sealed rotating inlets and outlets to the substrate 19 when water or other liquid coolants are used. Additionally, cryogenic fluids such as liquid nitrogen produce large temperature gradients, thus increasing the thermal sink-effect. Other liquid coolants such as water are not excluded from the present invention.

30 The cylindrical substrate 19 may be sealed by a seal 26 at one end and with

a rotating seal 28 at the other. The seal 28 may be provided with a sealed feedthrough 27 for the supply of cryogenic fluid. If water cooling is used, rotating seals at both ends of the cylindrical substrate are considered very important to prevent escape of water vapour into the deposition environment. In accordance with an embodiment of the present invention it is preferred if the ends 26, 27 allow escape of a cryogenic fluid which then forms a shield gas around substrate 19 during the spraying process. Particularly preferred cryogenic fluids are liquid nitrogen, liquid oxygen and liquid air. With some complex ceramic materials, one or more components may be reduced in the spraying process. For such materials it may be advantageous to use a shield gas including oxygen, e.g. liquid air or liquid oxygen, which may help to reoxidise the reduced component. On the other hand with other complex ceramics it may be advantageous to reduce the contact time with oxygen at high temperatures, under which conditions liquid nitrogen would be preferred, or a reducing gas may be included such as hydrogen. It is preferable to control the atmosphere in the vicinity of the substrate 19 during coating deposition to prevent the presence of excessive water vapour and in particular to prevent condensation of water on the substrate 19. This may be achieved by generally air conditioning the air around the substrate 19 to reduce its dew point.

It is preferred if the deposition rate is selected in order to maintain the substrate surface temperatures mentioned above. Assuming the cylindrical substrate as shown in Fig. 1, the rotation speed of the substrate 19, the linear speed of the gun 12 and the rate of material exiting the gun 12 may be controlled to achieve the temperatures specified above. For instance, it has been found that when using cylindrical substrates made of stainless steel of 15 cm diameter and up to 40 cm long, a powder delivery of 5-10 g/min was suitable to produce 3 -10 mm coatings when depositing a $\text{YBa}_2\text{Cu}_3\text{O}_7$ layer. The rotational speed of the substrate 19 may be in the range 10 to 100 RPM with a surface speed in the range 1 to 40 m/min and the longitudinal feed of the gun 12 in the range 1-3 m/min, typically 2 m/min. The deposition rate per reciprocating pass of the gun 12 may be 10 to 50 micron thickness of the coating. About 10% to 15% of the deposited

coating had maintained the lattice structure of the powder and exhibited superconducting properties. It will be appreciated by the skilled person that increasing the deposition speed, deposition thickness per pass or the flame temperature or reducing the thermal conductivity of the substrate material will increase the thermal load on the cooling system and adjustments of one or more of these parameters may be necessary to obtain satisfactory coatings. The thermal conductivity of the deposited material is preferably between 1 and 5 $\text{Wm}^{-1}\text{K}^{-1}$. When deposited on a steel substrate the thermal conductivity preferably lies within the range 25 to 125 $\text{Wm}^{-1}\text{K}^{-1}$. These values are particularly preferred for $\text{YBa}_2\text{Cu}_3\text{O}_7$ coatings. Preferably, an adhesion promoter layer is applied onto the substrate before application of the coating of the metal oxide combination. The adhesion promoter may be a layer of Ni-Al or a layer of an In-alloy, for example. The deposited coating is preferably impact resistant, e.g. withstands impact of a 0.036 kg steel ball from a height of 2 metres. Preferably, about 20% or up to 30% of a noble metal is included in the oxide material to improve electrical and thermal properties of the deposited layer. The noble metal is preferably silver. The noble metal may be included as a salt or oxide, e.g. silver nitrate or silver oxide, in the material to be sprayed. Preferably, the electrical resistivity of the deposited layer is lower than 15×10^{-6} Ohm.m, more preferably lower than 10×10^{-6} and most preferably less than 5×10^{-6} Ohm.m. Values below 1×10^{-6} Ohm.m can be achieved. Up to 30% of a noble metal such as silver may be added to lower the resistivity. These values are particularly preferred for $\text{YBa}_2\text{Cu}_3\text{O}_7$ coatings.

Fig. 2 is a schematic representation of a further embodiment of the flame spraying process and apparatus in accordance with the present invention.

Components in Fig. 2 with the same reference numbers as in Fig. 1 refer to equivalent items. The substrate 19 in accordance with this embodiment is a foil or sheet of metal, plastic or other flexible material which is wound from a pay-off spool 32 to a take-up spool 36. Where the final coating 40 cannot be spooled, the foil with coating 14 may be drawn linearly from the pay-off spool 32 and cut into lengths. The coating 40, which may be a superconducting layer, is flame sprayed

with a flame spray gun 12 similar to the one described with respect to Fig. 1. In particular it is preferable to use a fuel with a lower calorific value than acetylene such as natural or town gas, butane or propane. Preferably, the temperature of the flame of the gun 12 imparts a temperature of 1500 °C or less, more preferably 5 1200 °C or less to the material being sprayed through the flame. This material may be in the form of powder either of finished components of the coating 40, e.g. oxides, or precursors thereof, e.g. nitrates, or may be in the form of a slurry of powders, e.g. oxides, or a solution, e.g. of nitrates. Gun 12 may be controlled by hand or more preferably by a robot to provide zigzag motions across the width 10 of foil 19 thus applying an even coating 40. Preferably a layer of 10 to 50 micron thickness is applied in each pass.

The temperature of the coating 40 may be monitored by one or more optical sensors 13, 15. The temperature of the foil 19 is regulated by means of a cryogenic fluid supplied from a container 16 to a series of holes or jets 29 via 15 conduit 17, a controllable valve 11 and a conduit 30. The valve 11 is controlled by a controller 14 to maintain the temperature of the foil as determined by the sensor 13, 15 to less than 400 °C, preferably less than 150 °C and most preferably between 50 and 100 °C. Such low temperatures allow a wide range of materials for substrate 19 including polymeric materials, cellulosic materials as well as 20 metals. Although only one controller 14 is shown the present invention includes several controllers each with its own controllable cryogenic cooling device 11, 29, 30 for individually controlling the temperature of different parts of the foil 19 or coating 40. Optionally, an optical encoder 34 may be attached to a roller 35. The optical encoder may be read with an optical sensor 37, 38, the pulse frequency 25 generated in the sensor 37, 38 being proportional to the linear speed of the foil 19. This value may also be used by the controller 14 to control the complete process to maintain the temperatures and coating thicknesses mentioned above.

When producing superconducting coatings 40, it is preferred if there is no condensation of water onto the coating 40 nor onto the foil 19 so it is preferred if 30 the atmosphere around the deposition equipment is air conditioned to reduce the

dew point to below ambient temperature. Preferably the coated substrates in accordance with this invention are preferably stored for long periods in a plastic bag filled with a dry inert gas such as dry nitrogen. One aspect of the present invention is the flame spraying of powders which already have superconducting properties in the powder form. Using the methods in accordance with the present invention it is possible to flame spray such coatings and retain 10% to 15% of superconducting property of the coating 40 produced without extensive post-heat treatments.

The superconducting and/or ceramic powder and/or metallic powder to be used for flame spraying is preferably homogeneous, exhibits the appropriate rheological properties and correct stoichiometry to generate the required properties in the final coating. Typical preferred densities for superconducting powders may lie in the range 4 to 5 g/cm³. A non-limiting list of suitable materials which may be flame sprayed as powders, slurries or liquid solutions in accordance with the present invention are: superconducting materials such as R₁Ba₂Cu₃O_y where R is Y, La, Nd, Sm, Eu, Gd, Dy, Ho, Er, Tm, Yb, Lu; or Bi₂-_xPb_xSr₂Ca_{n-1}Cu_nO_y, Tl₂Ba₂Ca_{n-1}Cu_nO_{2n+3}, HgBa₂Ca_{n-1}Cu_nO_{2n+2+δ}; or Ba₂Ca_{n-1}O_{2n+2}, or CaBa₂Ca_{n-1}Cu_nO_{2n+δ}; or cuprate high temperature superconductors of the general formula A_mE₂R_{n-1}Cu_nO_{2n+m+2} where A, E, R are selected from various cations such as A = Bi, Tl, Hg, Pb, Cu or a lanthanide element, E = Ba or Sr and R = Ca or rare earth element; or piezo-electric ceramics, for example, with the general formula M(Zr_xTi_{1-x})O₃ where M = Pb, Ba or Sr; or refractory ceramic oxides, nitrides, carbides or phosphates, e.g. Al₂O₃, MgO, Zr_xO_y; or metals and their alloys.

In accordance with a further embodiment of the present invention a method is provided for production of suitable ceramic powders. By starting from aqueous solutions containing the salts of the metals in the correct proportions a reactive precursor powder can be obtained using commercially available spray drying equipment in batches of kilograms. The type of salt (mostly nitrates) should preferably be compatible with thermal decomposition to oxides in further

processes such as sintering or flame spraying. In accordance with the present invention spray dried nitrate powders may be flame sprayed directly or the powders may first be sintered and then flame sprayed.

A spray drying system 50 in accordance with an embodiment of the present invention for the delivery of powder suitable for subsequent flame spraying is shown schematically in Fig. 3. The input liquid is drawn from a suitable source 53 via a peristaltic pump 54 to a spray head 71. Pressurised air 51 is drawn in through an air dryer and optional pre-heater 52 to the spray head 71 by a suction device such as a fan 63 at the end of the generally closed system. The liquid from source 53 enters the spray head 71 which is cooled by any suitable means 55 to prevent clogging due to early evaporation of the liquid. The liquid is atomised in a co-current two fluid nozzle 71 by the dry pressurised air 51 and discharged it into a chamber 56 where it dries to a powder. The liquid from source 53 may be a solution of suitable nitrates or a slurry of the relevant oxides with the addition of other agents such as binders.

Air 65 is drawn in by fan 63 over a heater 64 and introduced into chamber 56 via a ring orifice 72 which surrounds the outlet of the spray head 71. The air 65 also heats the spray head 71. The circumferential air flow 65 guides the evaporating liquid in chamber 56 and helps to prevent the powder sticking to the walls of the chamber 56. The air throughput of fan 63 is chosen so that powder of the correct grain size is swept from chamber 36 through an optional heater section 58 into a powder collector 59. Heavier particles settle out in trap 57 and are removed from the bottom of chamber 56.

The powder collector 59 may be any suitable device such as a cyclone, a bag filter or an electrostatic filter although a cyclone is preferred. The cyclone discharges the powder into a removable container 60 sealed to the bottom of the cyclone 59. Spent air is removed via the trap 61 and scrubbed in scrubber 12 to remove impurities. The final clean air is exhausted to atmosphere by the fan 63 which controls air flow through the system.

The control system 66 - 70 for the process functions as follows. The

rotational speed of the centrifugal air pump 53, the temperature of the heating element 64 and the flow of the atomised air are set with controller 67, 70. Air flow is measured by gauge 68. The temperature of the hot air 65 and the air in the tube leading from the chamber 56 to the optional heater 58 is monitored using
5 thermocouples 66, whereas final powder temperature is monitored by temperature sensor 69.

After spray drying, the powder may be sintered in a single step. For example, to produce a superconducting powder of the general formula $\text{YBa}_2\text{Cu}_3\text{O}_7$ with optional Ag, the required nitrates are dissolved in water in the
10 correct stoichiometric proportions and spray dried as indicated above. The nitrates are then reduced to oxides by sintering at 920 - 960 °C for 40 to 60 hours. Optionally the nitrates may first be reduced by heating in air at 780 °C for 10 hours before sintering at the above temperatures and times. The $\text{YBa}_2\text{Cu}_3\text{O}_7$ powder produced by this procedure is superconducting. On aspect of the present
15 invention is to produce powders with superconducting properties by spray drying and optional sintering and then to flame spray these superconductive powders at the lowest flame temperatures necessary to obtain melting of the powder and coating formation on the substrate while cooling the coating in the fastest possible way. By this procedure the crystal structure present in the superconducting
20 powder is disturbed as little as possible by the flame spraying process. Of course, melting the powder during flame spraying causes complete loss of crystal organisation if the time in the melt is long. By lowering the flame temperature and shortening the time in the melt phase by cooling the coating very rapidly in accordance with the present invention, some local crystal organisation is kept in
25 the final flame sprayed coating, e.g. about 10% of the final coating is in the superconducting phase, thus providing a coating on the substrate with an optimum starting condition for further heat processing to develop full superconducting properties. The addition of the metallic silver enhances the thermal and mechanical properties in later flame spraying and magnetron sputtering.

30 Alternatively, the powder for flame spraying may be spray dried from

slurries of the relevant oxides in the correct stoichiometric proportions with the optional addition of silver in the above apparatus in accordance with the present invention. For instance, in the manufacture of a ceramic material the mixture of oxides may be produced by individually sieving them to 40 micron and then
5 mixing in the correct proportions to obtain the stoichiometric proportions in the final coating. A quantity of deionised water is added of about 60% by weight of the powder as well as a quantity of an organic binder such as PVA (polyvinyl acetate) equal to about 2% by weight of the powder and then mixed into a slurry. The slurry is then spray dried as described above resulting in powder with a grain
10 size from 30 to 50 micron. Generally, spray dried oxide slurries do not require sintering before flame spraying. The organic binder may be burnt out during flame spraying or in a special sintering step.

Spray drying of 10% by weight nitrate solutions generally produce grain sizes of 3 micron on average with at least 90% of the grains between 0.5 and 5
15 microns. In order to obtain the required grain size it is preferable to sinter as mentioned above. Light grinding and sieving of this sintered powder may produce a mass fraction of more than 80% with grain sizes between 40 and 80 micron. By the variation of appropriate concentrations of the solution of the aqueous media
53, and/or the addition of binders and/or the spray drying of slurries rather than
20 solutions, allows control of the grain size in the final powder to between 2 and 100 microns. For example, the present invention includes the addition of organic binders such as polyvinyl acetate (PVA) to the liquid to be spray dried to control grain size in the final powder. Such binders may be burnt out in a later high
temperature process such as sintering. An average grain size of 40 to 80 microns
25 is preferred for good flame spray deposition. The final powder may be lightly milled and sieved to be improve the homogeneity of grain sizes.

One aspect of the present invention is the inclusion of silver metal in the final superconducting ceramic coating. This is achieved as mentioned above by inclusion of about 20% to 30% by weight of the ceramic materials of silver nitrate
30 when nitrate solutions are spray dried and the flame sprayed or by addition of

Ag₂O powder in an oxide slurry which is then spray dried and flame sprayed. The addition of silver in the flame sprayed material is beneficial for the inter-grain adhesion and heat dispersal during flame spraying thus yielding a strong and dense coating. The silver improves the thermal and electrical conductivity of the flame sprayed coating which is beneficial to the sputtering process when the substrate is used as a sputtering target. The improved conductivities allow higher power throughput for the magnetron than targets not containing silver.

The flame spraying process in accordance with the present invention allows the reconditioning of targets for sputtering magnetrons. It is well known that the presence of a static race-track plasma on a magnetron target during sputtering results in an erosion groove and poor target utilisation. Using the flame spraying process of the present invention such a worn target may be reconditioned by spraying the appropriate target material into the erosion groove and building up the target to its former thickness in these regions. By providing the intensive cryogenic cooling described above, the general target temperature may be kept below 400 °C, preferably below 150°C and most preferably between room temperature (~25°C) and 100°C. These low temperatures result in little damage to the existing target material while still providing a mechanically strong coating in the old erosion grooves. Such a process is particularly economic when the target material is expensive such as superconducting materials. The flame spraying gun 12 described above may be hand held and the contour of the erosion groove in the used target followed building up the lost material slowly, preferably 10 to 50 micron per pass. Preferably the gun 12 is controlled by a robot which is programmed to execute the correct motions with the gun 12 to fill up the erosion groove in the target.

While the invention has been shown and described with reference to preferred embodiments, it will be understood by those skilled in the art that various changes or modifications in form and detail may be made without departing from the scope and spirit of this invention as defined in the attached claims.

CLAIMS

1. A composite comprising: a substrate and a coating deposited on said substrate, the coating being deposited by spraying, the thickness of the coating being at least 5 mm, more preferably greater than 8mm, the coating comprising metal oxides.
5
2. The composite according to claim 1, wherein the coating comprises a superconductor precursor and at least 10% of the coating is in a superconductive phase
- 10 3. The composite according to claim 1 or 2, wherein the composite is a target for a sputtering magnetron.
4. The composite according to claim 3, wherein the target is cylindrical.
- 15 5. The target for a sputtering magnetron comprising: a cylindrical substrate and a coating deposited on said substrate, the coating being deposited by spraying, the thickness of the coating being at least 3mm, more preferably at least 5 mm, most preferably greater than 8mm, the coating comprising a superconductor precursor and at least 10% of the coating is in a superconductive phase.
- 20 6. The target or a composite in accordance with any previous claim, wherein the coating has a thermal conductivity of between 1 and 5 $\text{Wm}^{-1}\text{K}^{-1}$.
7. The target or a composite in accordance with any previous claim, wherein the
25 thermal conductivity of the composite or the target through the substrate and the coating is in the range 25 to 125 $\text{Wm}^{-1}\text{K}^{-1}$.
8. The target or a composite in accordance with any previous claim, wherein the
30 coating has an electrical resistivity of lower than 15×10^{-6} Ohm.m, more preferably lower than 10×10^{-6} and most preferably less than 5×10^{-6} Ohm.m.

9. The target or a composite in accordance with any previous claim, wherein the coating can withstand impact of a 0.036 kg steel ball from a height of 1 metre, preferably from 1.5 metre.

5

10. The target or a composite in accordance with any previous claim, wherein the spraying is one of plasma spraying and flame spraying.

11. A method of depositing by spraying a superconductor precursor layer onto a cylindrical target for a sputtering magnetron, the layer having a thickness of at least 3 mm, and at least 10% of the layer being in a superconductive phase.

12. A method of depositing by spraying a layer onto a substrate, the layer having a thickness of at least 5 mm, and the coating comprising metal oxides.

15

13. The method according to claim 11 or 12, wherein the spraying step is one of flame spraying and low-pressure or atmospheric pressure plasma spraying.

14. The method according to claim 13, wherein the spraying step includes spraying a material through a spraying head, the material being in the form of a powder, a slurry or a solution.

15. A method of reconditioning a used target for a sputtering magnetron having an erosion groove in the target material, comprising the step of: flame or atmospheric pressure plasma spraying target material into the erosion groove.

16. A reconditioned target for a sputtering magnetron, comprising:
an erosion groove in the target material; and target material flame sprayed or atmospheric plasma sprayed into said groove to restore the thickness of the target material to that of the unused material.

30

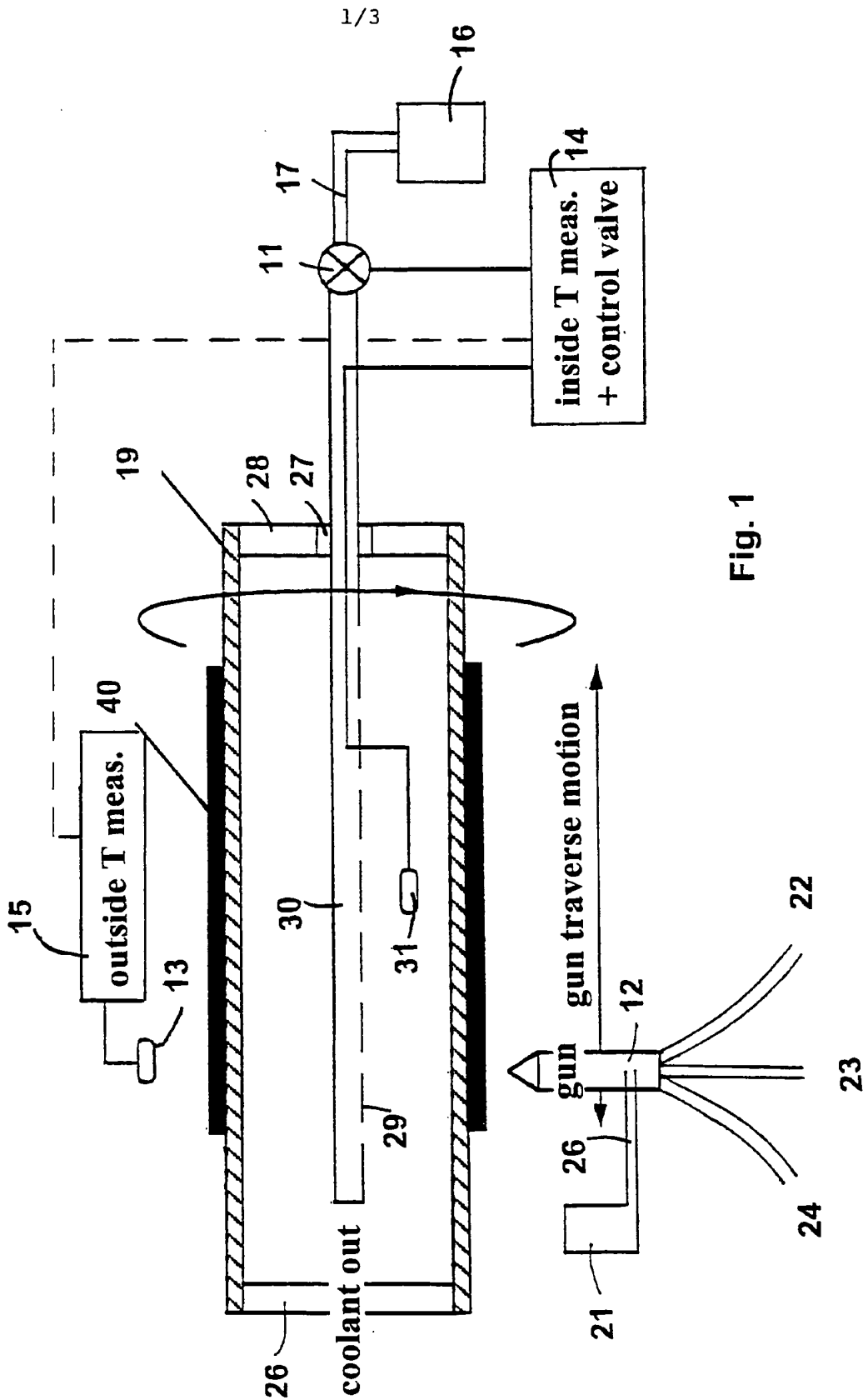


Fig. 1

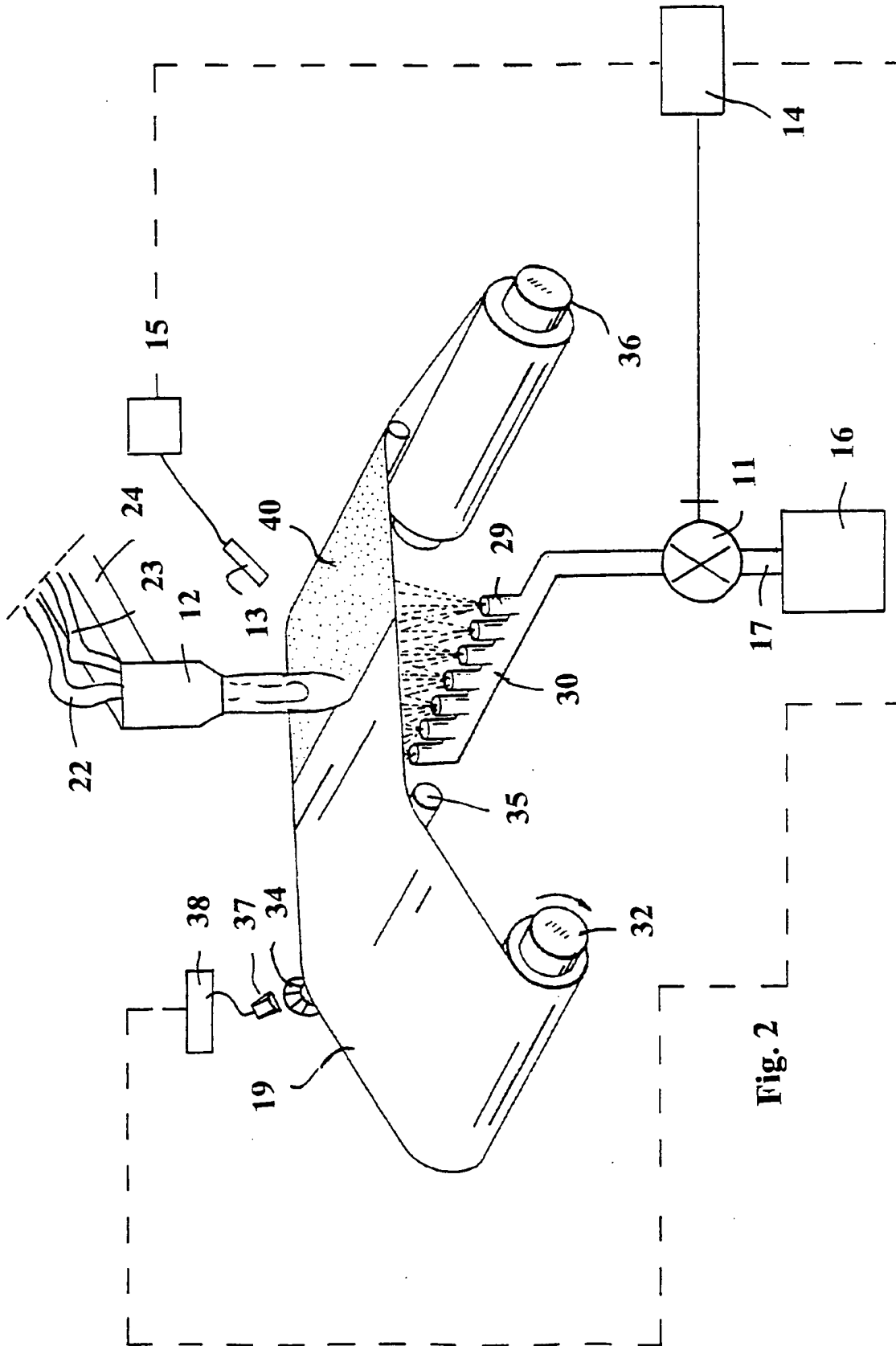


Fig. 2

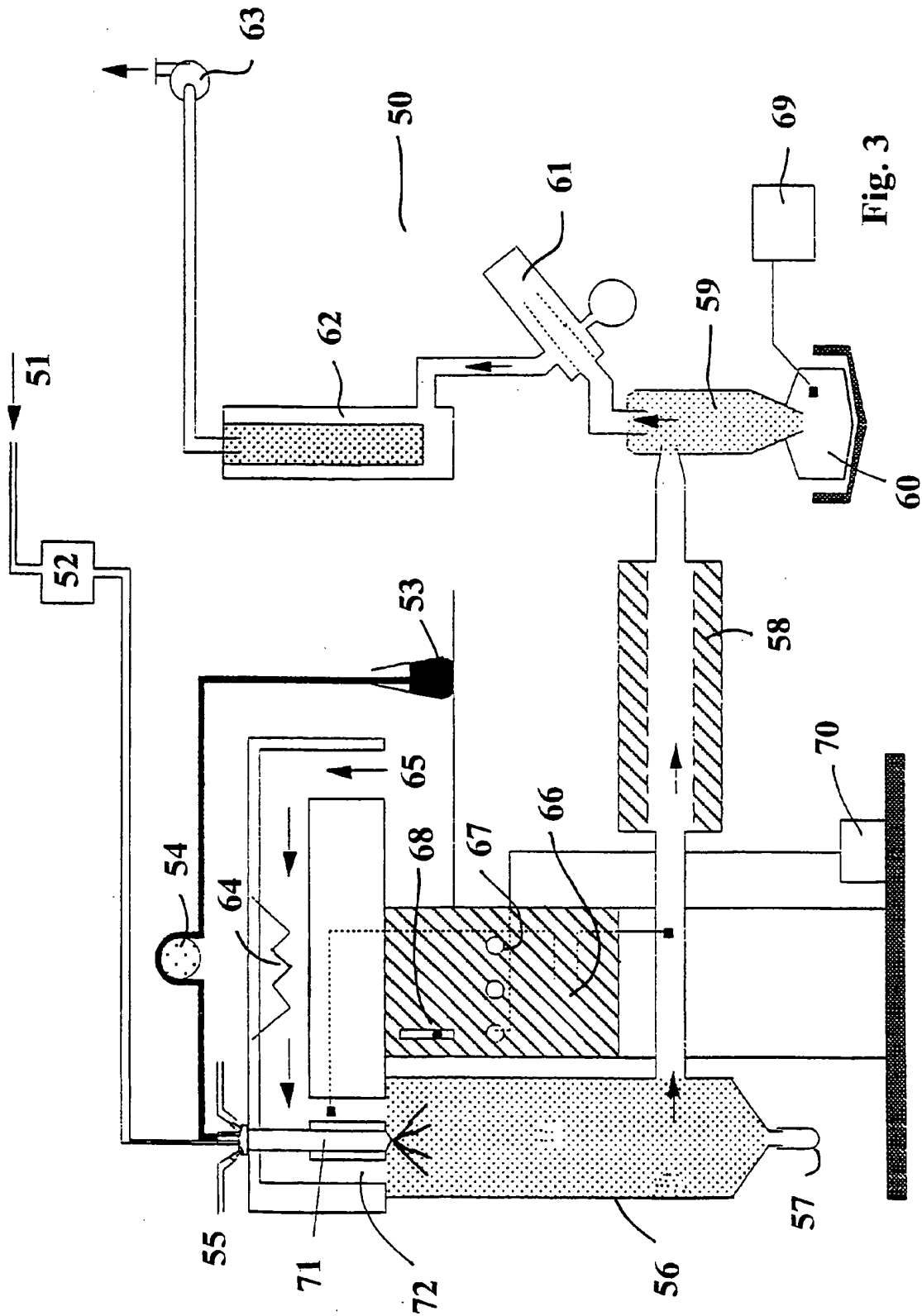


Fig. 3

INTERNATIONAL SEARCH REPORT

International Application No

PL/EP 99/03599

A. CLASSIFICATION OF SUBJECT MATTER
 IPC 6 C23C4/10 C23C4/12 C23C14/34

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED
 Minimum documentation searched (classification system followed by classification symbols)
 IPC 6 C23C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 196 400 A (CHEN CHIOU T ET AL) 23 March 1993 (1993-03-23) cited in the application column 3, line 52 - column 4, line 16 ---	1-5, 10-14
X	EP 0 330 196 A (PERKIN ELMER CORP) 30 August 1989 (1989-08-30) page 5, line 20 - line 25; claims 1,8 ---	1,2,10, 12-14
X	CH 648 358 A (CASTOLIN SA) 15 March 1985 (1985-03-15) claim 1 ---	1,10, 12-14
X	DD 277 471 A (MANSFELD KOMBINAT W PIECK VEB) 4 April 1990 (1990-04-04) page 2, line 21 - line 23 ---	15,16
	-/--	

Further documents are listed in the continuation of box C. Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance
 "E" earlier document but published on or after the international filing date
 "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
 "O" document referring to an oral disclosure, use, exhibition or other means
 "P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
 "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
 "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
 "&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
9 September 1999	16/09/1999

Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer Patterson, A
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2

INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 99/03599

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 40 15 387 A (LEYBOLD AG) 21 November 1991 (1991-11-21) the whole document ---	15,16
A	EP 0 377 073 A (MITSUBISHI METAL CORP) 11 July 1990 (1990-07-11) page 3, line 41 - line 57; table 2 page 7, line 7 - line 23; table 4 -----	5-8

INTERNATIONAL SEARCH REPORT

International application No.

PCT/EP 99/ 03599

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. Claims Nos.: 9
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
SEE FURTHER INFORMATION SHEET

3. Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

SEE FURTHER INFORMATION SHEET

1. As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.

2. As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:

4. No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims: it is covered by claims Nos.:

Remark on Protest

- The additional search fees were accompanied by the applicant's protest.
- No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

International Application No. PCT/EP 99 03599

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. Claims: 1-8,10-14

Claims for a composite comprising an oxide-containing coating deposited by spraying and having a certain minimum thickness, a sputter target having a layer of a superconductor precursor, also deposited by spraying and having a minimum thickness, and methods for producing each of these products, respectively.

2. Claims: 15,16

Reconditioned sputter target and method for its manufacture by flame or plasma spraying material into the sputtered erosion groove.

The only common concept linking subjects 1 and 2 is the application of spraying for depositing layers of material. Since this feature is known from many prior art documents (see for example search report) there is effectively no common concept to connect the two groups of claims. Furthermore, while subject 1 addresses the problem of producing relatively thick deposits of oxides by means of spraying, subject 2 solves the unrelated problem of spraying an unspecified material to repair local damage on a target.

Therefore there is a lack of unity between the two subjects.

Nevertheless, since the EPO has already searched the subject-matter of claims 15 and 16 in connection with the priority application EP98870120, an International search report is issued for both subjects.

INTERNATIONAL SEARCH REPORT

International Application No. PCT/EP 99 03599

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box I.2

Claims Nos.: 9

Claim 9 relates to a product defined (inter alia) by reference to the following parameter:

the result of a test involving impact of a ball consisting of an unspecified steel, wherein both the weight of the ball and the height from which it is dropped are arbitrarily chosen.

The use of this parameter in the present context is considered to lead to a lack of clarity within the meaning of Article 6 PCT. It is impossible to compare the parameter the applicant has chosen to employ with what is set out in the prior art. The lack of clarity is such as to render a meaningful complete search impossible. Consequently, the search has been restricted to the remaining claims

The applicant's attention is drawn to the fact that claims relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 99/03599

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 5196400	A	23-03-1993	NONE	
EP 0330196	A	30-08-1989	CN 1036286 A JP 1309952 A	11-10-1989 14-12-1989
CH 648358	A	15-03-1985	NONE	
DD 277471	A	04-04-1990	NONE	
DE 4015387	A	21-11-1991	NONE	
EP 0377073	A	11-07-1990	JP 2156079 A JP 2157150 A KR 9507087 B US 5077269 A KR 9507088 B	15-06-1990 15-06-1990 30-06-1995 31-12-1991 30-06-1995

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
15 June 2006 (15.06.2006)

PCT

(10) International Publication Number
WO 2006/063308 A2

- (51) International Patent Classification:
B05D 5/12 (2006.01) H01M 10/38 (2006.01)
H01M 4/52 (2006.01) C23C 14/34 (2006.01)
- (21) International Application Number:
PCT/US2005/044781
- (22) International Filing Date:
7 December 2005 (07.12.2005)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:
60/634,818 8 December 2004 (08.12.2004) US
60/651,363 8 February 2005 (08.02.2005) US

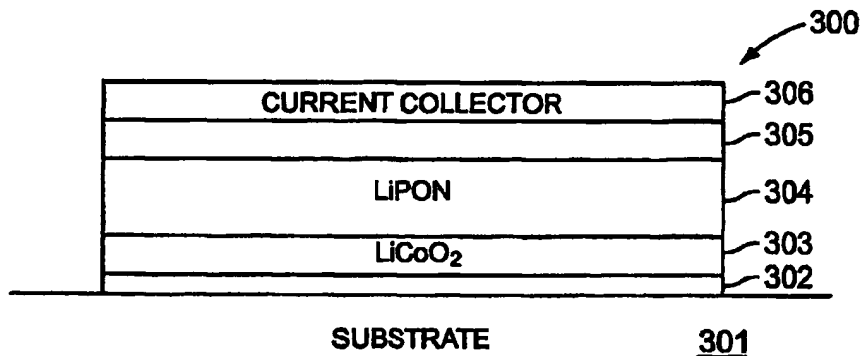
- (74) Agent: EDWARDS, Gary, J.; Finnegan, Henderson, Farabow, Garrett & Dunner LLP, 901 New York Avenue, Washington, D.C., District of Columbia 20001-4413 (US).
- (81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.
- (84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

- (71) Applicant (for all designated States except US): SYM-MORPHIX, INC. [US/US]; 1278 Reamwood Avenue, Sunnyvale, CA 94089-2233 (US).
- (72) Inventors; and
- (75) Inventors/Applicants (for US only): ZHANG, Hongmei [US/US]; 1330 Rodney Drive, San Jose, CA 95118 (US). DEMARAY, Richard, E. [US/US]; 190 Fawn Lane, Portola Valley, CA 94028 (US). SHAO, May [US/US]; 5401 Nectar Circle, Elk Grove, CA 95757 (US).

Published:
— without international search report and to be republished upon receipt of that report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: DEPOSITION OF LiCoO₂



WO 2006/063308 A2

(57) Abstract: In accordance with the present invention, deposition of LiCoO₂ layers in a pulsed-dc physical vapor deposition process is presented. Such a deposition can provide a low-temperature, high deposition rate deposition of a crystalline layer of LiCoO₂ with a desired <101> or <003> orientation. Some embodiments of the deposition addresses the need for high rate deposition of LiCoO₂ films, which can be utilized as the cathode layer in a solid state rechargeable Li battery. Embodiments of the process according to the present invention can eliminate the high temperature (>700°C) anneal step that is conventionally needed to crystallize the LiCoO₂ layer. Some embodiments of the process can improve a battery utilizing the LiCoO₂ layer by utilizing a rapid thermal anneal process with short ramp rates.

DEPOSITION OF LiCoO_2

RELATED APPLICATION

[001] The present application claims priority to Provisional Application No. 60/651,363, filed on February 8, 2005, by Hongmei Zhang and Richard E. Demaray, and to Provisional Application No. 60/634,818, filed on December 8, 2004, by the same inventors, each of which are herein incorporated by reference in their entirety.

BACKGROUND

1. Field of the Invention

[002] The present invention is related to thin-film solid-state batteries and, in particular, the deposition of LiCoO_2 films and layers for battery manufacture.

2. Discussion of Related Art

[003] Solid-state thin-film batteries are typically formed by stacking thin films on a substrate in such a way that the films cooperate to generate a voltage. The thin films typically include current collectors, a cathode, an anode, and an electrolyte. The thin films can be deposited utilizing a number of deposition processes, including sputtering and electroplating. Substrates suitable for this application have conventionally been high temperature materials capable of withstanding at least one high temperature anneal process to at least 700 °C for up to about 2 hours in air so as to crystallize the LiCoO_2 film. Such a substrate can be any suitable material with appropriate structural and material properties, for example a semiconductor wafer, metallic sheet (e.g., titanium or zirconium), ceramic such as alumina, or other material capable of withstanding subsequent high temperature processing in

the presence of the LiCoO_2 , which can experience significant interfacial reactions with most materials utilized in a battery during these temperature cycles.

[004] Other lithium containing mixed metal oxides besides LiCoO_2 , including Ni, Nb, Mn, V, and sometimes also Co, but including other transition metal oxides, have been evaluated as crystalline energy storage cathode materials. Typically, the cathode material is deposited in amorphous form and then the material is heated in an anneal process to form the crystalline material. In LiCoO_2 , for example, an anneal at or above 700°C transforms the deposited amorphous film to a crystalline form. Such a high temperature anneal, however, severely limits the materials that can be utilized as the substrate, induces destructive reaction with the lithium containing cathode material and often requires the use of expensive noble metals such as gold. Such high thermal budget processes (i.e., high temperatures for extended periods of time) are incompatible with semiconductor or MEM device processing and limit the choice of substrate materials, increase the cost, and decrease the yield of such batteries. The inventors are unaware of a process disclosed in the art that allows production of cathodic lithium films for a battery structure where a post-deposition anneal process has a low enough thermal budget to allow production of functional structures on low temperature materials such as stainless steel, aluminum, or copper foil.

[005] It is known that crystallization of amorphous LiCoO_2 on precious metals can be achieved. An example of this crystallization is discussed in Kim et al., where a conventional furnace anneal at 700°C for 20 minutes of an amorphous layer of LiCoO_2 on a precious metal achieves crystallization of the LiCoO_2 material, as shown by x-ray diffraction data. Kim, Han-Ki and Yoon, Young Soo, "Characteristics of rapid-thermal-annealed LiCoO_2 cathode film for an all-solid-state thin film microbattery," J. Vac. Sci. Techn. A 22(4), Jul/Aug 2004. In Kim et al., the LiCoO_2 film was deposited on a platinum film that was deposited on a high-temperature MgO/Si substrate. In Kim et al, it was shown that such

a crystalline film is capable of constituting the Li⁺ ion containing cathode layer of a functional all solid-state Li⁺ ion battery. However, it is of continuing interest for the manufacture of solid state Li⁺ ion batteries to further reduce the thermal budget of the post deposition anneal, both in time and in temperature, so as to enable the manufacture of such batteries without the need for expensive precious metal nucleation, barrier layers, or expensive high-temperature substrates.

[006] There are many references that disclose an ion beam assisted process that can provide a LiCoO₂ film that demonstrates some observable crystalline composition by low angle x-ray diffraction (XRD). Some examples of these are found in U.S. Patent Applications 09/815,983 (Publication No. US 2002/001747), 09/815,621 (Publication No. US 2001/0032666), and 09/815,919 (Publication No. US 2002/0001746). These references disclose the use of a second front side ion beam or other ion source side-by-side with a deposition source so as to obtain a region of overlap of the flux of ions with the flux of LiCoO₂ vapor at the substrate surface. None of these references disclose film temperature data or other temperature data of the film during deposition to support an assertion of low temperature processing.

[007] It is very difficult to form a uniform deposition either by sputtering a material layer or by bombardment with an ion flux. Utilization of two uniform simultaneous distributions from two sources that do not occupy the same position and extent with respect to the substrate enormously increases the difficulties involved in achieving a uniform material deposition. These references do not disclose a uniform materials deposition, which is required for reliable production of thin-film batteries. A well understood specification for material uniformity for useful battery products is that a 5% one-sigma material uniformity is standard in thin film manufacturing. About 86% of the films with this uniformity will be found acceptable for battery production.

[008] It is even more difficult to scale a substrate to manufacturing scale, such as 200 mm or 300 mm. Indeed, in the references discussed above that utilize both a sputtering deposition and an ion beam deposition, only small area targets and small area substrates are disclosed. These references disclose a single feasibility result. No method for achieving a uniform distribution from two separate front side sources has been disclosed in these references.

[009] Further, conventional materials and production processes can limit the energy density capacity of the batteries produced, causing a need for more batteries occupying more volume. It is specifically desirable to produce batteries that have large amounts of stored energy per unit volume in order to provide batteries of low weight and low volume.

[010] Therefore, there is a need for a low temperature process for depositing crystalline material, for example LiCoO_2 material, onto a substrate.

SUMMARY

[011] In accordance with the present invention, deposition of LiCoO_2 layers in a pulsed-dc physical vapor deposition process is presented. Such a deposition can provide a low-temperature, high deposition rate deposition of a crystalline layer of LiCoO_2 with a desired $\langle 101 \rangle$ orientation. Some embodiments of the deposition address the need for high rate deposition of LiCoO_2 films, which can be utilized as the cathode layer in a solid state rechargeable Li battery. Embodiments of the process according to the present invention can eliminate the high temperature ($>700^\circ\text{C}$) anneal step that is conventionally needed to crystallize the LiCoO_2 layer.

[012] A method of depositing a LiCoO_2 layer according to some embodiments of the present invention includes placing a substrate in a reactor; flowing a gaseous mixture including argon and oxygen through the reactor; and applying pulsed-DC power to a target

formed of LiCoO_2 positioned opposite the substrate. In some embodiments, a LiCoO_2 layer is formed on the substrate. Further, in some embodiments the LiCoO_2 layer is a crystalline layer of orientation $\langle 101 \rangle$.

[013] In some embodiments, a stacked battery structure can be formed. The stacked battery structure includes one or more battery stacks deposited on a thin substrate, wherein each battery stack includes: a conducting layer, a crystalline LiCoO_2 layer deposited over the conducting layer, a LiPON layer deposited over the LiCoO_2 layer; and an anode deposited over the LiPON layer. A top conducting layer can be deposited over the one or more battery stacks.

[014] In some embodiments, a battery structure can be formed in a cluster tool. A method of producing a battery in a cluster tool includes loading a substrate into a cluster tool; depositing a conducting layer over the substrate in a first chamber of the cluster tool; depositing a crystalline LiCoO_2 layer over the conducting layer in a second chamber of the cluster tool; depositing a LiPON layer over the LiCoO_2 layer in a third chamber of the cluster tool; depositing an anode layer over the LiCoO_2 layer in a fourth chamber; and depositing a second conducting layer over the LiPON layer in a fifth chamber of the cluster tool.

[015] A fixture for holding a thin substrate can include a top portion and a bottom portion, wherein the thin substrate is held when the top portion is attached to the bottom portion.

[016] These and other embodiments of the invention are further discussed below with reference to the following figures. It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory only and are not restrictive of the invention, as claimed. Further, specific explanations or theories regarding the deposition or performance of certain layers during deposition processes or in the performance of devices incorporating those layers are presented for explanation only and

are not to be considered limiting with respect to the scope of the present disclosure or the claims.

BRIEF DESCRIPTION OF THE DRAWINGS

[017] Figures 1A and 1B illustrate a pulsed-DC biased reactive deposition apparatus that can be utilized in the methods of depositing according to the present invention.

[018] Figure 2 shows an example of a target that can be utilized in the reactor illustrated in Figures 1A and 1B.

[019] Figure 3 illustrates a thin-film battery design according to some embodiments of the present invention.

[020] Figures 4A and 4B show an x-ray diffraction analysis of and an SEM photograph of a LiCoO_2 film deposited according to embodiments of the present invention.

[021] Figures 5A through 5F show SEM photographs of LiCoO_2 films according to some embodiments of the present invention.

[022] Figure 5G shows x-ray diffraction data corresponding to the depositions shown in Figures 5B-5F.

[023] Figure 6A illustrates a layer of LiCoO_2 deposited according to some embodiments of the present invention on a thin substrate.

[024] Figure 6B illustrates a layer of LiCoO_2 deposited according to some embodiments of the present invention over a conducting layer on a thin substrate.

[025] Figures 7A, 7B, 7C, and 7D illustrate a thin substrate mount and mask arrangement that can be utilized in the deposition of LiCoO_2 layers deposited according to some embodiments of the present invention.

[026] Figure 8 illustrates a cluster tool that can be utilized to form batteries with LiCoO_2 layers deposited according to some embodiments of the present invention.

[027] Figures 9A and 9B illustrate examples of stacked batter structures with LiCoO₂ layers deposited according to some embodiments of the present invention.

[028] Figures 10A through 10D illustrate deposition and anneal steps for LiCoO₂ deposited over an iridium layer on a silicon wafer.

[029] Figures 11A through 11D illustrate a single layer battery formed over an iridium layer according to some embodiments of the present invention.

[030] Figures 12A through 12L illustrate deposition of a crystalline LiCoO₂ layer on a silicon or alumina substrate.

[031] Figures 13A through 13F illustrate rapid thermal anneal processes for LiCoO₂ layers deposited according to the present invention.

[032] Figures 14A through 14D illustrate several anneal processes utilized with a LiCoO₂ film deposited according to embodiments of the present invention.

[033] Figures 15A and 15B illustrate the effects of ramp-time in a rapid thermal anneal of LiCoO₂ films deposited according to the present invention.

[034] Figure 16 illustrates thickness uniformity of a LiCoO₂ film deposited according to some embodiments of the present invention.

[035] Figure 17 illustrates battery charge and discharge profiles of a battery formed utilizing a LiCoO₂ film according to some embodiments of the present invention.

[036] In the figures, elements having the same designation have the same or similar functions.

DETAILED DESCRIPTION

[037] In accordance with embodiments of the present invention, LiCoO₂ films are deposited on a substrate by a pulsed-dc physical vapor deposition (PVD) process. In contrast to, for example, Kim et al., LiCoO₂ films according to some embodiments of the present

invention provide a crystalline LiCoO_2 film as deposited on a substrate at a substrate temperature as low as about 220 °C during deposition, without the use of a metallic nucleation or barrier underlying film. The as-deposited crystalline LiCoO_2 films can be easily ripened to very high crystalline condition by anneal at about 700 °C for as little as 5 minutes without the use of an underlying precious metal film. In addition, the as deposited crystalline films, when positioned on a noble metal film can be annealed at much further reduced temperatures, for example as low as 400 to 500 °C, providing for deposition, annealing, and production of solid state batteries on lower temperature substrates.

[038] In the present application, a single, extended source is described which has been scaled to 400mm X 500mm for production achieving a LiCoO_2 uniformity of 3% one-sigma measured at 25 points at a deposition rate of 1.2 microns thickness an hour over an area of 2000 cm^2 , without the need for secondary front side ion source or ion assistance.

[039] In one example process, a LiCoO_2 film was deposited utilizing a conductive ceramic LiCoO_2 target as described herein, with pulsed-dc power of 4 kW, no bias, with 60 sccm Ar and 20 sccm O_2 gas flows. A 3000 Angstrom layer of crystalline LiCoO_2 was deposited on a substrate area of 400 X 500 mm. As demonstrated in Figure 16, film thickness uniformity was located at about 25 locations spaced uniformly across the substrate using a felt marker pen to lift off a portion of the film in each location. High precision white-light interferometry was utilized to measure the film thickness in each location by measuring the step height from the substrate to film surface. All 25 thickness measurements demonstrated a 3% one-sigma uniformity in the film thickness over 400 X 500 mm substrate area. As shown in Figure 16, a film was deposited with average thickness of about 2.96 μm with a maximum of 3.09 μm and a minimum of 2.70 μm and standard deviation of 0.093. Thickness data was taken at points spaced 0.65 mm apart on the surface of the film. The film thickness therefore showed 3% one-sigma uniformity over the shown surface area.

[040] On other depositions utilizing this process, a temperature measurement of the substrate during deposition showed that the substrate remained at less than 224 °C.

Temperature measurements were performed utilizing a temperature sticker purchased from Omega Engineering, Stamford, Ct (Model no. TL-F-390, active from 199-224 °C).

[041] Moreover, in some embodiments, films deposited according to the present invention can have a deposition rate of from about 10 to about 30 times higher than processes in conventional films. Deposition thicknesses and times of deposition for films deposited according to the present invention are illustrated in Table I. Furthermore, films according to the present invention can be deposited on wide area substrates having a surface area from 10 to 50 times the surface area of prior sputtering processes, resulting in much higher productivity and much lower cost of manufacture, thereby providing high-volume, low-cost batteries.

[042] Further, conventional deposition processes without ion sources are capable of depositing amorphous LiCoO_2 layers, but do not deposit crystalline LiCoO_2 layers. Surprisingly, depositions according to some embodiment of the present invention, deposit a LiCoO_2 layer with substantial crystallinity readily measured by x-ray diffraction techniques. In some embodiments, the crystallinity of the as-deposited LiCoO_2 layers is sufficient to be utilized in a battery structure with no further thermal processing. In some embodiments, crystallinity of the as-deposited LiCoO_2 layers are improved by thermal processes with low thermal budgets, which can be compatible with films deposited on low-temperature substrates.

[043] Further, as-deposited the stoichiometry of some LiCoO_2 layers deposited according to some embodiments of the present invention shows that this layer is sufficient for utilization in a battery. With the demonstrated ability to deposit a LiCoO_2 film with crystallinity and with sufficient stoichiometry, a battery utilizing as-deposited LiCoO_2 films

can be produced. Heat treating the LiCoO_2 layers may improve the crystallinity and lower the impedance.

[044] In some embodiments, a crystalline layer of LiCoO_2 with a $\langle 101 \rangle$ or a $\langle 003 \rangle$ crystalline orientation is deposited directly on the substrate. Deposition of crystalline material can eliminate or lessen the need of a subsequent high temperature anneal or precious-metal layers to crystallize and orient the film. Removing the high temperature anneal allows for formation of battery structures on light-weight and low temperature substrates such as stainless steel foil, copper foil, aluminum foil, and plastic sheet, reducing both the weight and the cost of batteries while retaining the energy density storage capabilities of Li-based batteries. In some embodiments, a crystalline LiCoO_2 layer can be deposited on a precious metal layer, such as platinum or iridium, resulting in a further significant lowering of the ripening thermal budget required to improve crystallinity.

[045] Deposition of materials by pulsed-DC biased reactive ion deposition is described in U.S. Patent Application Serial No. 10/101863, entitled "Biased Pulse DC Reactive Sputtering of Oxide Films," to Hongmei Zhang, et al., filed on March 16, 2002. Preparation of targets is described in U.S. Patent Application Serial No. 10/101,341, entitled "Rare-Earth Pre-Alloyed PVD Targets for Dielectric Planar Applications," to Vassiliki Milonopoulou, et al., filed on March 16, 2002. U.S. Patent Application Serial No. 10/101863 and U.S. Patent Application Serial No. 10/101,341 are each assigned to the same assignee as is the present disclosure and each is incorporated herein in their entirety. Deposition of oxide materials has also been described in U.S. Patent No. 6,506,289, which is also herein incorporated by reference in its entirety. Transparent oxide films can be deposited utilizing processes similar to those specifically described in U.S. Patent No. 6,506,289 and U.S. Application Serial No. 10/101863.

[046] Figure 1A shows a schematic of a reactor apparatus 10 for sputtering material from a target 12 according to the present invention. In some embodiments, apparatus 10 may, for example, be adapted from an AKT-1600 PVD (400 X 500 mm substrate size) system from Applied Komatsu or an AKT-4300 (600 X 720 mm substrate size) system from Applied Komatsu, Santa Clara, CA. The AKT-1600 reactor, for example, has three deposition chambers connected by a vacuum transport chamber. These AKT reactors can be modified such that pulsed DC power is supplied to the target and RF power is supplied to the substrate during deposition of a material film. Apparatus 10 can also be a Phoenix Gen III PVD cluster tool made by Symmorphix, which is specifically designed for pulsed-dc processes such as is described herein.

[047] Apparatus 10 includes target 12 which is electrically coupled through a filter 15 to a pulsed DC power supply 14. In some embodiments, target 12 is a wide area sputter source target, which provides material to be deposited on a substrate 16. Substrate 16 is positioned parallel to and opposite target 12. Target 12 functions as a cathode when power is applied to it from the pulsed DC power supply 14 and is equivalently termed a cathode. Application of power to target 12 creates a plasma 53. Substrate 16 is capacitively coupled to an electrode 17 through an insulator 54. Electrode 17 can be coupled to an RF power supply 18. A magnet 20 is scanned across the top of target 12.

[048] For pulsed reactive dc magnetron sputtering, as performed by apparatus 10, the polarity of the power supplied to target 12 by power supply 14 oscillates between negative and positive potentials. During the positive period, the insulating layer on the surface of target 12 is discharged and arcing is prevented. To obtain arc free deposition, the pulsing frequency exceeds a critical frequency that can depend on target material, cathode current and reverse time. High quality oxide films can be made using reactive pulse DC magnetron sputtering as shown in apparatus 10.

[049] Pulsed DC power supply 14 can be any pulsed DC power supply, for example an AE Pinnacle plus 10K by Advanced Energy, Inc. With this DC power supply, up to 10 kW of pulsed DC power can be supplied at a frequency of between 0 and 350 kHz. The reverse voltage can be 10% of the negative target voltage. Utilization of other power supplies can lead to different power characteristics, frequency characteristics, and reverse voltage percentages. The reverse time on this embodiment of power supply 14 can be adjusted between 0 and 5 μ s.

[050] Filter 15 prevents the bias power from power supply 18 from coupling into pulsed DC power supply 14. In some embodiments, power supply 18 can be a 2 MHz RF power supply, for example a Nova-25 power supply made by ENI, Colorado Springs, Co.

[051] In some embodiments, filter 15 can be a 2 MHz sinusoidal band rejection filter. In some embodiments, the band width of the filter can be approximately 100 kHz. Filter 15, therefore, prevents the 2 MHz power from the bias to substrate 16 from damaging power supply 14 and allow passage of the pulsed-dc power and frequency.

[052] Pulsed DC deposited films are not fully dense and may have columnar structures. Columnar structures can be detrimental to thin film applications such as barrier films and dielectric films, where high density is important, due to the boundaries between the columns. The columns act to lower the dielectric strength of the material, but may provide diffusion paths for transport or diffusion of electrical current, ionic current, gas, or other chemical agents such as water. In the case of a solid state battery, a columnar structure containing crystallinity as derived from processes according to the present invention is beneficial for battery performance because it allows better Li transport through the boundaries of the material.

[053] In the Phoenix system, for example, target 12 can have an active size of about 800.00 X 920.00 mm by 4 to 8 mm in order to deposit films on substrate 16 that have

dimension about 600 X 720 mm. The temperature of substrate 16 can be adjusted to between -50 °C and 500 °C. The distance between target 12 and substrate 16 can be between about 3 and about 9 cm (in some embodiments, between 4.8 and 6 cm are used). Process gas can be inserted into the chamber of apparatus 10 at a rate up to about 200 sccm while the pressure in the chamber of apparatus 10 can be held at between about .7 and 6 milliTorr. Magnet 20 provides a magnetic field of strength between about 400 and about 600 Gauss directed in the plane of target 12 and is moved across target 12 at a rate of less than about 20-30 sec/scan. In some embodiments utilizing the Phoenix reactor, magnet 20 can be a race-track shaped magnet with dimensions about 150 mm by 800 mm.

[054] Figure 2 illustrates an example of target 12. A film deposited on a substrate positioned on carrier sheet 17 directly opposed to region 52 of target 12 has good thickness uniformity. Region 52 is the region shown in Figure 1B that is exposed to a uniform plasma condition. In some implementations, carrier 17 can be coextensive with region 52. Region 24 shown in Figure 2 indicates the area below which both physically and chemically uniform deposition can be achieved, for example where physical and chemical uniformity provide refractive index uniformity, oxide film uniformity, or metallic film uniformity. Figure 2 indicates region 52 of target 12 that provides thickness uniformity, which is, in general, larger than region 24 of target 12 providing thickness and chemical uniformity to the deposited film. In optimized processes, however, regions 52 and 24 may be coextensive.

[055] In some embodiments, magnet 20 extends beyond area 52 in one direction, for example the Y direction in Figure 2, so that scanning is necessary in only one direction, for example the X direction, to provide a time averaged uniform magnetic field. As shown in Figures 1A and 1B, magnet 20 can be scanned over the entire extent of target 12, which is larger than region 52 of uniform sputter erosion. Magnet 20 is moved in a plane parallel to the plane of target 12.

[056] The combination of a uniform target 12 with a target area 52 larger than the area of substrate 16 can provide films of highly uniform thickness. Further, the material properties of the film deposited can be highly uniform. The conditions of sputtering at the target surface, such as the uniformity of erosion, the average temperature of the plasma at the target surface, and the equilibration of the target surface with the gas phase ambient of the process are uniform over a region which is greater than or equal to the region to be coated with a uniform film thickness. In addition, the region of uniform film thickness is greater than or equal to the region of the film which is to have highly uniform electrical, mechanical, or optical properties such as index of refraction, stoichiometry, density, transmission, or absorptivity.

[057] Target 12 can be formed of any materials that provide the correct stoichiometry for LiCoO_2 deposition. Typical ceramic target materials include oxides of Li and Co as well as metallic Li and Co additions and dopants such as Ni, Si, Nb, or other suitable metal oxide additions. In the present disclosure, target 12 can be formed from LiCoO_2 for deposition of LiCoO_2 film.

[058] In some embodiments of the invention, material tiles are formed. These tiles can be mounted on a backing plate to form a target for apparatus 10. A wide area sputter cathode target can be formed from a close packed array of smaller tiles. Target 12, therefore, may include any number of tiles, for example between 2 and 60 individual tiles. Tiles can be finished to a size so as to provide a margin of edge-wise non-contact, tile to tile, less than about 0.010" to about 0.020" or less than half a millimeter so as to eliminate plasma processes that may occur between adjacent ones of tiles 30. The distance between tiles of target 12 and the dark space anode or ground shield 19 in Figure 1B can be somewhat larger so as to provide non contact assembly or to provide for thermal expansion tolerance during process chamber conditioning or operation.

[059] As shown in Figure 1B, a uniform plasma condition can be created in the region between target 12 and substrate 16 in a region overlying substrate 16. A plasma 53 can be created in region 51, which extends under the entire target 12. A central region 52 of target 12 can experience a condition of uniform sputter erosion. As discussed further herein, a layer deposited on a substrate placed anywhere below central region 52 can then be uniform in thickness and other properties (i.e., dielectric, optical index, or material concentrations). In some embodiments, target 12 is substantially planar in order to provide uniformity in the film deposited on substrate 16. In practice, planarity of target 12 can mean that all portions of the target surface in region 52 are within a few millimeters of a planar surface, and can be typically within 0.5 mm of a planar surface.

[060] Figure 3 shows a battery structure with a LiCoO_2 layer deposited according to some embodiments of the present invention. As shown in Figure 3, a metallic current collection layer 302 is deposited on a substrate 301. In some embodiments, current collection layer 302 can be patterned in various ways before deposition of a LiCoO_2 layer 303. Also according to some embodiments, LiCoO_2 layer 303 can be a deposited crystalline layer. In some embodiments of the invention, layer 303 is crystalline without the necessity of a crystallizing heat treatment. Therefore, substrate 301 can be a silicon wafer, titanium metal, alumina, or other conventional high temperature substrate, but may also be a low temperature material such as plastic, glass, or other material which could be susceptible to damage from the high temperature crystallizing heat treatment. This feature can have the great advantage of decreasing the expense and weight of battery structures formed by the present invention. The low temperature deposition of the LiCoO_2 allows for successive depositions of battery layers, one upon another. Such a process would have the advantage that successive layers of battery structure would be obtained in a stacked condition without the inclusion of a substrate

layer. The stacked layered battery would provide higher specific energy density as well as low impedance operation for charging and discharging.

[061] In some embodiments, an oxide layer can be deposited on substrate 301. For example, a silicon oxide layer can be deposited on a silicon wafer. Other layers can be formed between conducting layer 302 and substrate 301.

[062] As further shown in Figure 3, a LiPON layer 304 ($\text{Li}_x\text{PO}_y\text{N}_z$) is deposited over LiCoO_2 layer 303. LiPON layer 304 is the electrolyte for battery 300 while LiCoO_2 layer 303 acts as the cathode. A metallic conducting layer 305 can be deposited over the LiPON layer 304 in order to complete the battery. Metallic conducting layer 305 can include lithium adjacent to LiPON layer 304.

[063] An anode 305 is deposited over LiPON layer 304. Anode 305 can be, for example an evaporated lithium metal. Other materials such as, for example, nickel can also be utilized. A current collector 306, which is a conducting material, is then deposited over at least a portion of anode 305.

[064] A Li based thin film battery operates by transport of Li ions in the direction from current collector 306 to current collector 302 in order to hold the voltage between current collector 306 and current collector 302 at a constant voltage. The ability for battery structure 300 to supply steady current, then, depends on the ability of Li ions to diffuse through LiPON layer 304 and LiCoO_2 layer 303. Li transport through bulk cathode LiCoO_2 layer 303 in a thin film battery occurs by the way of grains or grain boundaries. Without being restricted in this disclosure to any particular theory of transport, it is believed that the grains with their planes parallel to substrate 302 will block the flow of Li ions while grains oriented with planes perpendicular to substrate 301 (i.e., oriented parallel to the direction of Li ion flow) facilitate the Li diffusion. Therefore, in order to provide a high-current battery

structure, LiCoO₂ layer 303 should include crystals oriented in the <101> direction or <003> direction.

[065] In accordance with the present invention, LiCoO₂ films can be deposited on substrate 302 with a pulsed-DC biased PVD system as was described above. In addition, an AKT 1600 PVD system can be modified to provide an RF bias, which is available in the Phoenix system, and an Advanced Energy Pinnacle plus 10K pulsed DC power supply can be utilized to provide power to a target. The pulsing frequency of the power supply can vary from about 0 to about 350 KHz. The power output of the power supply is between 0 and about 10 kW. A target of densified LiCoO₂ tiles having a resistivity in the range of about 3 to about 10 kΩ can be utilized with dc-sputtering.

[066] In some embodiments, LiCoO₂ films are deposited on Si wafers. Gas flows containing Oxygen and Argon can be utilized. In some embodiments, the Oxygen to Argon ratio ranges from 0 to about 50% with a total gas flow of about 80 sccm. The pulsing frequency ranges from about 200 kHz to about 300 kHz during deposition. RF bias can also be applied to the substrate. In many trials, the deposition rates vary from about 2 Angstrom/(kW sec) to about 1 Angstrom/(kW sec) depending on the O₂/Ar ratio as well as substrate bias.

[067] Table I illustrates some example depositions of LiCoO₂ according to the present invention. XRD (x-Ray Diffraction) results taken on the resulting thin films illustrate that films deposited according to the present invention are crystalline films, often with highly textured grain sizes as large as about 150 nm. The dominant crystal orientation appears to be sensitive to the O₂/Ar ratio. For certain O₂/Ar ratios (~10%), as-deposited films exhibit a preferred orientation in the <101> direction or the <003> direction with poorly developed <003> planes.

[068] Figures 4A and 4B illustrate an XRD Analysis and SEM cross section, respectively, of the LiCoO₂ film deposited as Example 15 in Table I. Such a LiCoO₂ film was deposited on Si wafer with 2kW of target power, a frequency of 300 kHz, with 60 sccm Ar and 20 sccm of O₂ for a substrate with an initial temperature of about 30°C. As shown in the XRD analysis of Figure 4A, a strong <101> peak is indicated showing a strong orientation of LiCoO₂ crystals in the desired <101> crystallographic direction. The SEM cross section shown in Figure 4B further shows the columnar structure of the film having the <101> direction and the grain boundaries of the resulting LiCoO₂ crystals.

[069] Figures 5A through 5F show SEM cross sections of further example depositions of LiCoO₂ crystals according to the present invention. In each of the examples, deposition of the LiCoO₂ film was performed on a Si wafer with target power of about 2 kW and frequency of about 250 kHz. The LiCoO₂ film shown in Figure 5A corresponds to the example deposition Example 1 in Table I. In the deposition of the LiCoO₂ film shown in Figure 5A, no bias power was utilized with an argon flow rate of about 80 sccm and an oxygen flow rate of about 0 sccm. A deposition rate of about 1.45 μm/hr was achieved over the full substrate area of 400 X 500 mm. Further, as is indicated in the cross section shown in Figure 5A, a <101> orientation of the LiCoO₂ was achieved.

[070] The rate of deposition of the LiCoO₂ layer shown in Figure 5A is very high, likely due to the relatively high conductivity or low resistivity of the ceramic LiCoO₂ oxide sputter target. A target resistance of 10 kOhms was measured by means of an Ohm meter over a distance of about 4 cm on the surface of target 12. This high rate allows the manufacture of the 3 micron or thicker LiCoO₂ layer required for the battery at high rate over a wide area in short times, resulting in very high productivity and very low cost. Target resistance on the order of about 500 kΩ over the same distance by the same measurement technique or higher would not allow for such a high sputter efficiency or high rate of

deposition at such a low target power. The resistance of conventional target materials can be unmeasurably high. A resistance of 100 k Ω over about 4 cm of surface will result in high sputter efficiency and high rate of deposition. Further, because deposition rates typically scale nearly linearly with target power, a deposition at 6 kW will yield a deposition rate of approximately 3 $\mu\text{m/hr}$, which is a very desirable rate of deposition for manufacturability of Li-based thin-film solid-state batteries on a surface area of 400 X 500 mm².

[071] The LiCoO₂ layer shown in Figure 5B is deposited under the conditions listed as Example 7 in Table I. Again, no bias was utilized in the deposition. An argon flow rate of about 72 sccm and an oxygen flow rate of about 8 sccm was utilized. The deposition rate was significantly reduced to about 0.85 $\mu\text{m/hr}$. Further, although a <101> crystallinity can be discerned, that <101> crystallinity is not as pronounced as that exhibited in the deposition of the film shown in Figure 5A.

[072] The LiCoO₂ film shown in Figure 5C was deposited according to Example 3 in Table I. In this deposition, 100 W of bias power is applied to the substrate. Further, an argon flow rate of 72 sccm, and an oxygen flow rate of 8 sccm was utilized. The deposition rate was about 0.67 $\mu\text{m/hr}$. Therefore, the application of bias in comparison with the LiCoO₂ film shown in Figure 5B further reduced the deposition rate (from 0.85 $\mu\text{m/hr}$ of the example shown in Figure 5B to 0.67 $\mu\text{m/hr}$ of the example shown in Figure 5C). Further, the desired <101> directionality of formed crystals appears to be further degraded.

[073] The LiCoO₂ film shown in Figure 5D corresponds to Example 4 in Table I. In this deposition, the Ar/O₂ ratio was increased. As is shown in Figure 5D, increasing the Ar/O₂ ratio improves crystallinity. With respect to the example illustrated in Figure 5C, the deposition illustrated in Figure 5D was performed with an argon flow of about 76 sccm and an oxygen flow of about 4 sccm as well as retaining the 100 W bias to the substrate. The

LiCoO₂ deposition rate was improved to 0.79 μm/hr from a rate of 0.67 μm/hr illustrated in Figure 5C.

[074] In the example deposition illustrated in Figure 5E corresponding to Example 5 in Table I. The substrate temperature was set at about 200°C while the bias power remained at about 100 W. The argon flow rate was set at about 76 sccm and the oxygen flow rate was set at about 4 sccm. The resulting deposition rate for the LiCoO₂ layer was about 0.74 μm/hr.

[075] In the example deposition illustrated in Figure 5F, which corresponds with Example 6 of Table I, the argon flow rate was set at about 74 sccm and the oxygen flow rate was set at about 6 sccm, resulting in a LiCoO₂ deposition rate of about 0.67 μm/hr. Therefore, increasing both argon and oxygen flow rate over the deposition illustrated in Figure 5E resulted in a lower deposition rate.

[076] Figure 5G illustrates XRD data corresponding to Figures 5F, 5D, 5C, 5E, and 5B, respectively. As illustrated in Figure 5G, as-deposited crystalline LiCoO₂ is deposited in these processes.

[077] The data show clearly that an as-deposited crystalline film of LiCoO₂ can be obtained under several of the process conditions, as shown in Table II. In particular, very high rates of deposition with low power are obtained along with the oriented crystalline structure for the process conditions according to embodiments of the present invention.

[078] Figure 6A illustrates a layer of LiCoO₂ 602 deposited on a thin substrate 601 according to some embodiments of the present invention. Higher lithium-ion mobilities can be achieved utilizing crystalline LiCoO₂ cathode films 602 deposited on a thin substrate 601 that has thickness comparable to that of the battery stack itself, rather than a thickness many or tens of times that of the battery stack. Such a film can lead to faster charging and discharging rates. Substrate 601 can be formed of a thin metallic sheet (e.g., aluminum,

titanium, stainless steel, or other suitable thin metallic sheet), can be formed of a polymer or plastic material, or may be formed of a ceramic or glass material. As shown in Figure 6B, if substrate 601 is an insulating material, a conducting layer 603 can be deposited between substrate 601 and LiCoO_2 layer 602.

[079] Depositing materials on a thin substrate involves holding and positioning the substrate during deposition. Figures 7A, 7B, 7C, and 7D illustrate a reusable fixture 700 for holding a thin film substrate. As shown in Figure 7A, reusable fixture 700 includes a top portion 701 and a bottom portion 702 that snap together. Thin substrate 601 is positioned between top portion 701 and bottom portion 702. As shown in Figure 7B, top portion 701 and bottom portion 702 are such that substrate 601 is brought into tension and subsequently clamped as top portion 701 is closed into bottom portion 702. Substrate 601 can be easily held by fixture 700 so that substrate 601 can be handled and positioned. In some embodiments, the corners of substrate 601, areas 703, are removed so that substrate 601 is more easily stretched by avoiding "wrap-around" corner clamping effects when top portion 701 is closed into bottom portion 702.

[080] As shown in Figure 7C, a mask 712 can be attached to fixture 700. In some embodiments, fixture 700 includes guides in order to align fixture 700 with respect to mask 712. In some embodiments, mask 712 may be attached to fixture 700 and travel with fixture 700. Mask 712 can be positioned at any desired height above substrate 601 in fixture 700. Therefore, mask 712 can function as either a contact or proximity mask. In some embodiments, mask 712 is formed of another thin substrate mounted in a fixture similar to fixture 700.

[081] As shown in Figure 7C and 7D, fixture 700 and mask 712 can be positioned relative to mount 710. Mount 710, for example, can be a susceptor, mount, or an electrostatic chuck of a processing chamber such as that shown in Figures 1A and 1B. Fixture 700 and

mask 712 can have features that allow for ready alignment with respect to each other and with respect to mount 710. In some embodiments, mask 712 is resident in the processing chamber and aligned with fixture 700 during positioning of fixture 700 on mount 710, as shown in Figure 7D.

[082] Utilizing fixture 700 as shown in Figures 7A, 7B, 7C, and 7D allows processing of a thin film substrate in a processing chamber. In some embodiments, thin film substrates can be about 10 μm or more. Further, thin film substrate 601, once mounted within fixture 700, can be handled and moved from process chamber to process chamber. Therefore, a multiprocessor chamber system can be utilized to form stacks of layers, including one or more layers of LiCoO_2 deposited according to embodiments of the present invention.

[083] Figure 8 illustrates a cluster tool 800 for processing thin film substrates. Cluster tool 800 can, for example, include load lock 802 and load lock 803, through which mounted thin film substrate 601 is loaded and a resultant device is removed from cluster tool 800. Chambers 804, 805, 806, 807, and 808 are processing chambers for depositions of materials, heat treatments, etching, or other processes. One or more of chambers 804, 805, 806, 807, and 808 can be a pulsed-DC PVD chamber such as that discussed above with respect to Figures 1A and 1B and within which a LiCoO_2 film deposited according to embodiments of the present invention may be deposited.

[084] Processing chambers 804, 805, 806, 807, and 808 as well as load locks 802 and 803 are coupled by transfer chamber 801. Transfer chamber 801 includes substrate transfer robotics to shuttle individual wafers between processing chambers 804, 805, 806, 807, and 808 and load locks 802 and 803.

[085] In production of a conventional thin-film battery, ceramic substrates are loaded into load lock 803. A thin metallic layer can be deposited in chamber 804, followed

by a LiCoO_2 deposition performed in chamber 805. The substrate is then removed through load lock 803 for an in-air heat treatment external to cluster tool 800. The treated wafer is then reloaded into cluster tool 800 through load lock 802. A LiPON layer can be deposited in chamber 806. The wafer is then again removed from cluster tool 800 for deposition of the lithium anode layer, or sometimes chamber 807 can be adapted to deposition of the lithium anode layer. A second metallic layer is deposited in chamber 808 to form a charge collector and anode collector. The finished battery structure is then off-loaded from cluster tool 800 in load lock 802. Wafers are shuttled from chamber to chamber by robotics in transfer chamber 801.

[086] A battery structure produced according to the present invention could utilize thin film substrates loaded in a fixture such as fixture 700. Fixture 700 is then loaded into load lock 803. Chamber 804 may still include deposition of a conducting layer. Chamber 805 then includes deposition of a LiCoO_2 layer according to embodiments of the present invention. A LiPON layer can then be deposited in chamber 806. Chamber 807 may still be adapted to deposition of a lithium rich material such as lithium metal and chamber 808 can be utilized for deposition of the conducting layer of the current collector. In this process, no heat treatment is utilized to crystallize the LiCoO_2 layer.

[087] Another advantage of a thin film battery process is the ability to stack battery structures. In other words, substrates loaded into cluster tool 800 may traverse process chambers 804, 805, 806, 807, and 808 multiple times in order to produce multiply stacked battery structures. Figures 9A and 9B illustrate such battery structures.

[088] Figure 9A illustrates a parallel coupled stacking. As shown in Figure 9A, a substrate 601, which for example can be a plastic substrate, is loaded into load lock 803. A conducting layer 603, for example about 2 μm of aluminum, copper, iridium or other material, acts as a bottom current collector. Conducting layer 603, for example, can be

deposited in chamber 804. A LiCoO_2 layer 602 is then deposited on conducting layer 603. LiCoO_2 layer 602 can be about 3-10 μm and can be deposited in chamber 805 according to embodiments of the present invention. The wafer can then be moved to chamber 806 where a LiPON layer 901 of thickness of about .5 to about 2 μm can be deposited. In chamber 807, an anode layer 902, for example a lithium metal layer of up to about 10 μm , can then be deposited in chamber 807. A second conducting layer 903 can then be deposited over anode layer 902. A second battery stack can then be deposited over the first battery stack formed by metal layer 603, LiCoO_2 layer 602, LiPON layer 901, lithium layer 902, and current collection conduction layer 903. Over current collection conducting layer 903, another lithium layer 902 is formed. Another LiPON layer 901 is formed over lithium layer 902. Another LiCoO_2 layer 602 is formed over LiPON layer 901 and finally another metal layer 603 is formed over LiCoO_2 layer 602. In some embodiments, further stackings can be formed. In some embodiments, metal layers 603 and 903 differ in the mask utilized in deposition so that tabs are formed for electrical coupling of layers.

[089] As discussed above, any number of individual battery stacks can be formed such that parallel battery formations are formed. Such a parallel arrangement of battery stacking structure can be indicated as Current collector/ LiCoO_2 /LiPON/Anode/current collector/Anode/LiPON/ LiCoO_2 /current collector/ LiCoO_2 . . . /current collector. Figure 9B illustrates an alternative stacking corresponding to the battery structure current collector/ LiCoO_2 /LiPON/anode/current collector/ LiCoO_2 /LiPON/anode/current collector . . . /current collector. In this case, a series arrangement battery stacking structure is formed because the individual battery stacks share anodes.

[090] To form the structures shown in Figures 9A and 9B, substrates are rotated again through the chambers of cluster tool 800 in order to deposit the multiple sets of batteries. In general, a stack of any number of batteries can be deposited in this fashion.

[091] In some embodiments, stoichiometric LiCoO_2 can be deposited on iridium. Figures 10A through 10D illustrate an anneal procedure for Li-Co deposition over an iridium layer that has been deposited on a Si wafer. The LiCoO_2 deposition was accomplished as discussed above with a target power of 2 kW, no bias power, reverse time of 1.6 μs , a pulsing frequency of 300 kHz, with 60 sccm Ar flow and 20 sccm of O_2 flow, with no pre-heat for 7200 sec. As a result, a layer of LiCoO_2 of about 1.51 μm was deposited.

[092] Figures 10A through 10D show XRD analysis of both as-deposited and annealed layers of LiCoO_2 deposited as discussed above. The XRD analysis of the as-deposited layer demonstrates a shallow peak at $2\theta = 18.85^\circ$ denoting a $\langle 003 \rangle$ orientation of crystalline LiCoO_2 , a sharper peak at about $2\theta = 38.07^\circ$ corresponding with the desired $\langle 101 \rangle$ crystallographic direction, and a peak at $2\theta = 40.57^\circ$ corresponding to the $\langle 111 \rangle$ direction of iridium. However, the position of the $\langle 101 \rangle$ LiCoO_2 peak indicates that the $\langle 101 \rangle$ LiCoO_2 peak is nonstoichiometric LiCoO_2 . In order to be useful as a battery layer, stoichiometric LiCoO_2 provides for the best Li transport. One of ordinary skill in the art will notice that careful adjustment of deposition parameters can provide stoichiometric LiCoO_2 of desired orientation.

[093] Figure 10B shows an XRD analysis of the sample shown in figure 10A after a 300°C anneal in air for 2 hours. As shown in Figure 10B, the XRD peak corresponding to $\langle 003 \rangle$ LiCoO_2 grows, indicating crystallization of LiCoO_2 into the $\langle 003 \rangle$ direction. Further, the $\langle 101 \rangle$ peak of LiCoO_2 shifts slightly to $2\theta = 38.53^\circ$, indicating a more stoichiometric crystallization of the $\langle 101 \rangle$ LiCoO_2 . However, the crystalline LiCoO_2 is still not stoichiometric after this anneal. One of ordinary skill in the art will notice that longer anneals and/or further adjustment of the deposited stoichiometry may result in usefully oriented stoichiometric LiCoO_2 layers with anneal temperatures at 300 °C or less. Consequently, low temperature materials such as polymers, glass, or metal may be utilized as the substrate.

[094] Figure 10C illustrates an XRD analysis from the sample after a subsequent 500°C anneal in air for 2 hours. As shown in Figure 10C, more of the LiCoO₂ crystalizes into the <003> layer. Further, the <101> LiCoO₂ peak shifts again to $2\theta = 39.08^\circ$, indicating crystallization of a <012> layer of LiCoO₂. In this case, the <012> LiCoO₂ crystal is stoichiometric and therefore allows for efficient Li transport. One of ordinary skill in the art will notice that longer anneals and/or further adjustment of the deposited stoichiometry may result in usefully oriented stoichiometric LiCoO₂ layers with anneal temperatures at 500°C or less. Consequently, low temperature materials such as polymers, glass, or metal may be utilized as the substrate.

[095] Figure 10D illustrates an XRD analysis of the sample after a subsequent anneal of 700°C in air for 2 hours. As shown in Figure 10D, the <003> LiCoO₂ peak disappears, but the <012> LiCoO₂ peak remains relatively the same as that shown in the 500°C anneal illustrated in Figure 10C.

[096] Figures 10A through 10D demonstrate deposition of <101> LiCoO₂ at low temperature over an iridium layer. Subsequent anneals to 500°C may be desired to change the stoichiometry of the <101> LiCoO₂ layer, but anneals to 700 °C do not appear to be necessary. With anneal temperatures less than 500°C, depositions of a LiCoO₂ layer over a conducting iridium layer can be accomplished on glass, aluminum foil, plastic, or other low temperature substrate material. Anneal temperatures of less than 500°C but greater than 300°C or lengthening the time of lower temperature anneals may also result in desired orientations of stoichiometric crystalline LiCoO₂.

[097] Figures 11A through 11D illustrate formation of a single-layer battery according to some embodiments of the present invention. As shown in Figure 11A, a lift-off layer 1102 can be deposited on a substrate 1101. Further, an iridium layer 1103 can be

deposited over lift-off layer 1102. In some embodiments, substrate 1101 can be plastic, glass, Al foil, Si wafer, or any other material. Lift-off layer 1102 can be any lift off layer and can be a polymer layer such as polyimide, an inorganic layer such as CaF_2 or carbon, or an adhesive layer that loses its adhesion as a result of, for example, oxidation, heat, or light. Lift-off layers are well known. Iridium layer 1103 can be from about 500 Å or more.

[098] As shown in Figure 11B, a LiCoO_2 layer is deposited over iridium layer 1103 as was discussed above. In some embodiments, an anneal can be performed at this step. In some embodiments, further layers of the battery may be deposited before an anneal step is performed. In some embodiments, a stoichiometric LiCoO_2 layer of a useful crystalline orientation may result in the as-deposited LiCoO_2 with no further anneals necessary.

[099] Figure 11C illustrates deposition of a LiPON layer 1105 over the LiCoO_2 layer, deposition of a Li layer 1106 over LiPON layer 1105, and deposition of an electrode layer 1107 over Li layer 1106. In some embodiments, an anneal step of up to 500°C as discussed above may be performed here.

[0100] As shown in Figure 11D, the resulting single-layer battery formed from iridium layer 1103, LiCoO_2 layer 1104, LiPON layer 1105, Li layer 1106, and electrode layer 1107 can be "lifted off" from substrate 1101. Such a single-layer battery can be a free-standing battery of thickness about 5 μm or greater. Such a battery, without the requirement of a substrate 1101, is well known to have the potential of energy storage of greater than about 1 kW-hr/liter.

[0101] As an alternative to a lift-off process as described in Figures 11A through 11D, a substrate may be removed during anneal leaving a single-layer battery. Further, in some embodiments, substrate 1101 can be removed by a solvent, etching, or a photo process. Further, single-layer batteries may be combined or stacked in any fashion to provide a device of greater energy storage at a particular voltage.

[0102] Figures 12A through 12L illustrate the crystallinity of as-grown and post anneal LiCoO_2 layers according to samples 31 and 32 illustrated in Table I. Samples 31 and 32 were formed in the same deposition, utilizing a silicon substrate and an alumina substrate, respectively.

[0103] Figure 12A illustrates an XRD analysis of the as-deposited LiCoO_2 film on Al_2O_3 substrate (Example 32 in Table I). A broad $\langle 003 \rangle$ crystalline LiCoO_2 peak is observed. The remaining peaks in the analysis, which are not labeled in Figure 12A, result from the Al_2O_3 substrate. The $\langle 003 \rangle$ peak is characteristic of the layered structure in the as-deposited crystalline LiCoO_2 film according to embodiments of the present invention.

[0104] Figure 12B illustrates the crystallinity of the LiCoO_2 film shown in Figure 12A after a 2 hr 700 °C anneal. As shown in Figure 12B, the $\langle 003 \rangle$ peak becomes sharper and higher, indicating better crystallinity. As shown in Figures 12G through 12J, in comparison with figures 12C through 12F, the columnar structure ripens with the anneal and the grain size becomes larger with anneal. Figure 12B also shows $\langle 012 \rangle$ and $\langle 006 \rangle$ crystallinity peaks.

[0105] Figure 12C through 12F show SEM photos of the granularity of the as-deposited film corresponding to Example 32 in Figure I. Figures 12G through 12J show SEM photos of the granularity of the annealed film, as illustrated in Figure 12B. A comparison of Figures 12C through 12F with 12G through 12J illustrate the increased granularity resulting from the anneal process.

[0106] Figure 12K illustrates a fracture cross-section SEM that illustrates the morphology of the as-deposited crystalline film corresponding to Example 31 in Table I. Figure 12L illustrate a similar cross-section SEM corresponding to the film grown according to Example 32 in Table I.

[0107] Figures 13A through 13J illustrate rapid thermal anneal processes applied to a LiCoO_2 layer as in Example 49 of Table I. In that example, LiCoO_2 is deposited on alumina with a 2 kW pulsed DC power with no bias. Argon flow as set to 60 sccm and oxygen flow was set to 20 sccm. The deposition parameters are nearly identical with those of Example 32 in Table I, therefore XRD data for the as-deposited films are shown in Figure 12A. Figure 13A shows XRD data after a 15 minute 700 °C anneal in an argon atmosphere. Ramp-up time (room temperature to 700 °C) is 45 sec and ramp-down time (700 °C to about 300 °C) occurred over 10 min. At 300 °C, the sample is removed from the rapid-thermal-anneal (RTA) oven and cooled in air to room temperature. As shown in Figure 13A, substantial crystallinity is obtained. Figure 13B shows XRD data after a RTA as described with Figure 13A in an argon/oxygen atmosphere. The argon/oxygen ratio was 3:1.

[0108] As shown in a comparison of Figures 13A and 13B, more crystallinity is observed in an argon only RTA than with a RTA performed in the presence of oxygen. This is further illustrated in a comparison of Figures 13C and 13D with Figures 13E and 13F. Figures 13C and 13D show the granularity of the LiCoO_2 film after the RTA illustrated in Figure 13A. Figures 13E and 13F show the granularity of the LiCoO_2 film after the RTA illustrated in Figure 13B. As is observed, the granularity shown in Figures 13C and 13D (which differ in magnification) is better than that shown in Figures 13E and 13F (which also differ in magnification).

[0109] Figures 14A through 14D illustrate several anneal processes with the Example 37 of Table I. In that example, LiCoO_2 was deposited on alumina utilizing a pulsed-dc process with 2kW of power and 100 W of bias with an argon flow of 60 sccm and an oxygen flow of 20 sccm.

[0110] Figure 14A shows an SEM photo of an as-deposited LiCoO_2 film according to the process illustrated in Example 37 of Table I. Figure 14B shows an SEM photo of

LiCoO₂ film according to the process illustrated in Example 37 of Table I, annealed conventionally with a two-hour 700 °C anneal. Figures 14C and 14D show SEM photos of a LiCoO₂ film according to the process illustrated in Example 37 of Table I, annealed in an RTA process at 700 °C. The ramp-up and ramp-down times in the RTA process is illustrated above. Figure 14C shows an SEM photo of a LiCoO₂ film after an RTA process at 700 °C for five minutes whereas Figure 14D shows an SEM photo of a LiCoO₂ film after an RTA process at 700 °C for fifteen minutes. It is clear from a comparison of Figures 14C and 14D with Figure 14B, that much better granularity is achieved with the low thermal-budget RTA process rather than the conventional furnace anneal. A low thermal-budget RTA process allows for deposition of such films on low temperature substrates.

[0111] Figures 15A and 15B show SEM photos of a LiCoO₂ film that was annealed in an RTA process utilizing two different ramp-up times, illustrating the effects of the ramp time in the RTA process. A LiCoO₂ film was deposited on an alumina substrate according to the process described as Example 51 in Table I. The film shown in Figure 15A was annealed with a 45 sec ramp-up time (i.e., room temperature to 700 °C in 45 sec). The film shown in Figure 15B was annealed with a 240 sec ramp-up time. Both films were held at 700 °C for five minutes. As shown in a comparison between Figures 15A and 15B, it is clear that a short anneal ramp-up times yield better granularity than longer ramp-up times.

[0112] Figure 17 illustrates battery charge and discharge profiles of a battery structure formed utilizing LiCoO₂ films according to embodiments of the present invention. The LiCoO₂ film in the battery profiled in Figure 17 was deposited according to Example 54 in Table I. The LiCoO₂ film was deposited on an alumina substrate with a gold current collector. The LiCoO₂ film was annealed utilizing a fast-ramp (45 sec) RTA process as was described above. A 1.5 μm LiPON layer was then deposited with a standard RF deposition process without bias in a modified AKT reactor. A lithium anode and a nickel current

collector were then deposited. Data was taken at 0.33 mA, 1.65 mA, 3.3 mA, 16.5 mA, 33 mA, and 66 mA. As observed, the battery was capable of storing an exceptional 25 mA/cm² at voltages greater than 2.0 V.

[0113] One skilled in the art will recognize variations and modifications of the examples specifically discussed in this disclosure. These variations and modifications are intended to be within the scope and spirit of this disclosure. As such, the scope is limited only by the following claims.

TABLE I

Example #	Target Power (kW)	Bias Power (W)	Reverse Time (μ s)	Frequency (kHz)	Ar (sccm)	O ₂ (sccm)	Initial Substrate Temperature (temperature during deposit) (°C)	Deposition Time (sec)	Film Thickness (μ m)
1	2	0	1.6	250	80	0	30	10000	3.9
2	2	0		250	72	8	30	7200	1.7
3	2	100		250	72	8	30	7200	1.34
4	2	100		250	76	4	30	7200	1.57
5	2	100		250	76	4	200	7200	1.3
6	2	100		250	74	6	200	7200	1.3
7	2	0		300	72	8	30	7200	1.58
8	2	0		300	74	6	30	7200	
9	2	100		300	74	6	30	7200	
10	2	100		300	72	8	30	7200	
11	2	100		300	70	10	30	7200	
12	2	0		300	70	10	30	7200	
13	2	0		300	72	8	30	7200	1.58
14	2	0		300	74	6	30	7200	
15	2	0		300	60	20	30	7200	
16	2	0		300	50	30	30	7200	
17	2	200		300	60	20	30	7200	
18	2	50		300	60	20	30	7200	
19	2	0		300	70	10	30	7200	
20	2	0		300	65	15	30	7200	
21	3	0		300	65	15	30	7200	
22	2	0	1.6	250	60	20	30	7200	
23	3	0	1.6	250	60	20	30	7200	
24	2	0	1.6	250	60	20	30 (NPH)	7200	

25		2	0	1.6	250	60	20	10min heat 30min coc	7200	
26		2	0	1.6	250	60	20	no preheat	9000	
27		2	0		300	60	20	no preheat	7200	
28		2	0		300	60	20	15min heat, 10min	7200	
29		2	0		250	60	20	no preheat		
30		2	0		250	60	20	10min, 10min		
31		2	0	1.3	300	60	20	30 (220)	7200	4.81
32		2	0	1.3	300	60	20	30 (220)	7200	4.74
33		2	0	1.3	300	22.5	7.5	30 (220)	7200	3.99
34		2	0	1.3	300	22.5	7.5	30 (220)	7200	3.93
35		2	0	1.3	300	37.5	12.5	30 (220)	7200	3.64
36		2	0	1.3	300	37.5	12.5	30 (220)	7200	3.54
37		2	100	1.3	300	60	20	30 (220)	7200	4.54
38		2	200	1.3	300	60	20	30 (220)	7200	4.84
39		2	100	1.3	300	37.5	12.5	30 (220)	7200	4.30
40		2	100	1.3	300	22.5	7.5	30 (220)	7200	3.77
41		2	200	1.3	300	37.5	12.5	30 (220)	7200	3.92
42		2	200	1.3	300	60	20	400	7200	3.77
43		2	0	1.3	300	22.5	7.5	30(220)	7200	3.24
44		2	0	1.3	300	60	20	30(220)	7200	3.88
45		2	0	1.3	300	60	20	30(220)	3600	1.78
46		2	200	1.3	300	60	20	30(220)	3600	1.87
47		2	200	1.3	300	22.5	7.5	30(220)	3600	1.52
48		2	0	1.3	300	60	20	30(220)	6000	1.12
49		2	0	1.3	300	60	20	30(220)	10800	1.89
50		2	0	1.3	300	60	20	30(220)	14400	2.52
51		2	100	1.3	300	60	20	30(220)	10000	1.57
52		2	100	1.3	300	60	20	30(220)	10000	2.11
53		2	100	1.3	300	60	20	30(220)	6000	2.70

54	2	100	1.3	300	60	20	30(220)	6000	2.70
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TABLE II

Example #	Phase	Lattice	Texture	d_{101} [Å]	2θ [°]	crystallite size [Å]
15	LiCoO ₂	rhombohedral	strong [101]	2.376(1)	37.83	~1300
16	LiCoO ₂	Rhombohedral	strong [101]	2.375(1)	37.85	~750
17	Co	cubic	random	--	--	<50
18	Co	cubic	random	--	--	<50
19	LiCoO ₂	rhombohedral	strong [101]	2.370(1)	37.93	~1400
20	LiCoO ₂	rhombohedral	strong [101]	2.372(1)	37.90	~1500
21	LiCoO ₂	rhombohedral	strong [101]	2.370(1)	37.92	~1700
PDF	LiCoO ₂	Rhombohedral	random	2.408(1)	37.31	--

WHAT IS CLAIMED IS:

1. A method of depositing a LiCoO₂ layer, comprising:
placing a substrate in a reactor;
flowing a gaseous mixture including argon and oxygen through the reactor; and
applying pulsed DC power to a target formed of LiCoO₂ positioned opposite the
substrate,
wherein a crystalline layer of LiCoO₂ is deposited over the substrate.
2. The method of claim 1, further including applying an RF bias to the substrate.
3. The method of claim 1, wherein the crystalline layer is <101> oriented.
4. The method of claim 1, wherein the crystalline layer is <003> oriented.
5. The method of claim 1, wherein a grain size of the crystalline layer is between
about 750 Å and about 1700 Å.
6. The method of claim 1 wherein the substrate is a material chosen from a set
comprised of silicon, polymers, glasses, ceramics, and metals.
7. The method of claim 1, further including preheating the substrate to a temperature
of about 200 °C.
8. The method of claim 1, wherein the substrate is a low temperature substrate.
9. The method of claim 8, wherein the low temperature substrate is one of a set of
substrates including glass, plastic, and metal foil.
10. The method of claim 1, further including depositing an oxide layer on the
substrate.
11. The method of claim 10, wherein the oxide layer is a silicon dioxide layer.
12. The method of claim 3, wherein the crystalline layer is deposited at a rate of
greater than 1 µm per hour.

13. The method of claim 1 wherein the target is a ceramic LiCoO_2 sputter target with a resistance measured across about 4 cm of surface of less than about 500 k Ω .
14. The method of claim 1, further including depositing a metal layer on the substrate.
15. The method of claim 14, wherein the metal layer is iridium.
16. The method of claim 14, wherein the metal layer is platinum.
17. The method of claim 1, further including annealing the crystalline layer with a low thermal budget.
18. The method of claim 17, wherein annealing the crystalline layer includes annealing to 700°C in a rapid thermal anneal process for a period of time less than about 10 minutes.
19. The method of claim 14, further including annealing the LiCoO_2 layer at a temperature of less than or equal to about 500°C.
20. The method of claim 14, further including annealing the LiCoO_2 layer at a temperature of less than or equal to about 400°C.
21. A battery structure, comprising:
 - a crystalline LiCoO_2 layer deposited over a low-temperature substrate.
22. The structure of claim 21, further including a conducting layer deposited between the crystalline LiCoO_2 layer and the low-temperature substrate.
23. The structure of claim 22, wherein the conducting layer is an iridium layer.
24. The structure of claim 22, wherein the conducting layer is a platinum layer.
25. The structure of claim 21, further including a LiPON layer deposited over the LiCoO_2 layer.
26. The structure of claim 21, further including a second conducting layer deposited over the LiCoO_2 layer.

27. A stacked battery structure, comprising:

one or more battery stacks deposited on a thin substrate, wherein each battery stack comprises:

- a conducting layer,
- a LiCoO_2 layer deposited as a crystalline layer over the conducting layer,
- a LiPON layer deposited over the crystalline LiCoO_2 layer,
- an anode layer deposited over the LiPON layer; and
- a top conducting layer deposited over the one or more battery stacks.

28. The stacked battery structure of claim 27, wherein the battery stacks form a parallel stacked battery structure.

29. The stacked battery structure of claim 27, wherein the battery stacks form a series stacked battery structure.

30. The stacked battery structure of claim 27, wherein the conducting layer is a metal layer deposited on a substrate.

31. The stacked battery structure of claim 30, wherein the metal layer is an iridium layer.

32. The stacked battery structure of claim 30, wherein the metal layer is a platinum layer.

33. The stacked battery structure of claim 30, wherein the substrate is a low temperature substrate.

34. The stacked battery structure of claim 27, wherein the conducting layer is a metallic foil.

35. The stacked battery structure of claim 34, wherein the metallic foil is formed of a metal from a group consisting of copper, gold, platinum, aluminum, stainless steel and other nickel or cobalt based super alloy.

36. A method of producing a battery, comprising:
- loading a substrate into a cluster tool;
 - depositing a crystalline LiCoO_2 layer over a conducting layer in a chamber of the cluster tool with a pulsed-dc PVD process.
37. The method of claim 36, wherein depositing a crystalline LiCoO_2 layer includes depositing crystalline LiCoO_2 through a mask.
38. The method of claim 36, further including
- depositing a conducting layer on the substrate.
39. The method of claim 36, further including depositing a LiPON layer over the LiCoO_2 layer.
40. The method of claim 39, further including deposition an anode over the LiPON layer.
41. The method of claim 40, further including depositing a conducting layer over the anode.
42. The method of claim 36, wherein the conducting layer is an iridium layer.
43. A fixture for holding a thin substrate, comprising:
- a top portion; and
 - a bottom portion, wherein
- the thin substrate is held when the top portion is attached to the bottom portion.

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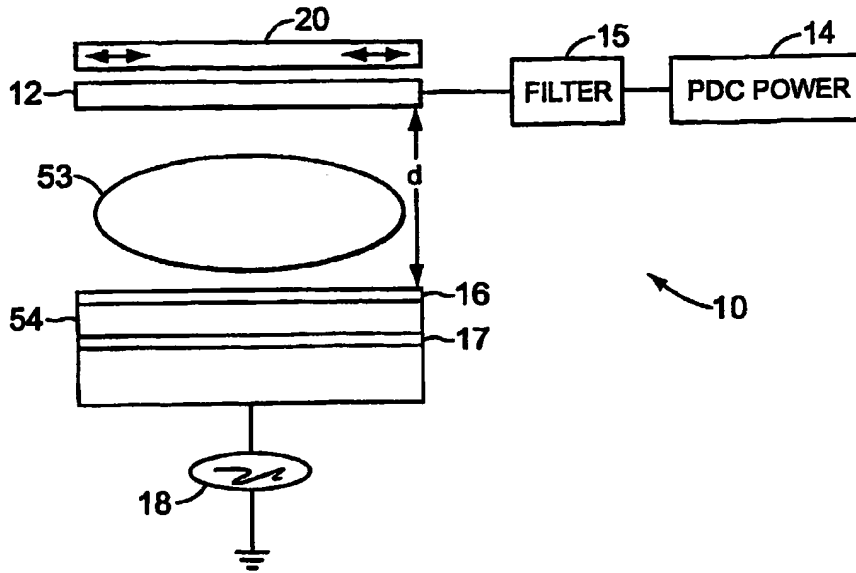


FIG. 1A

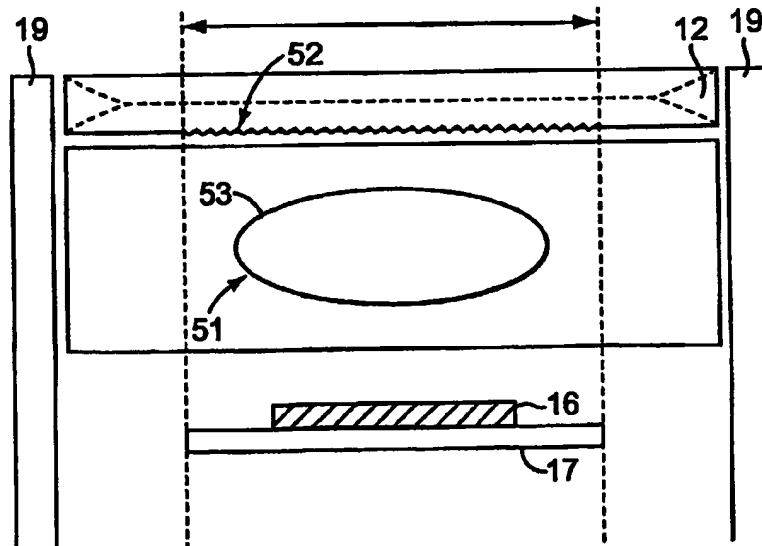


FIG. 1B

SUBSTITUTE SHEET (RULE 26)

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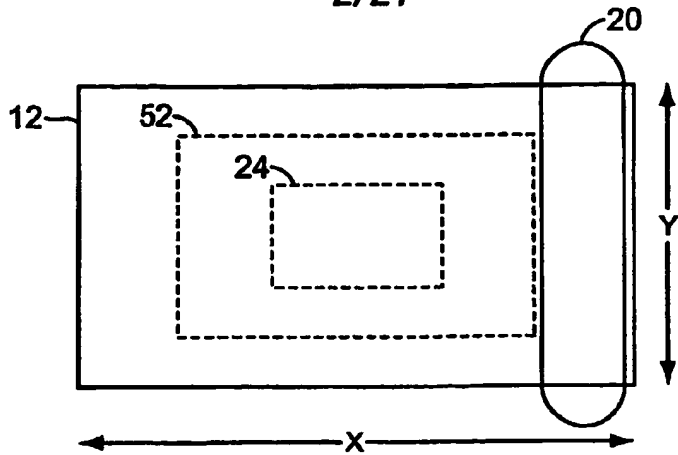


FIG. 2

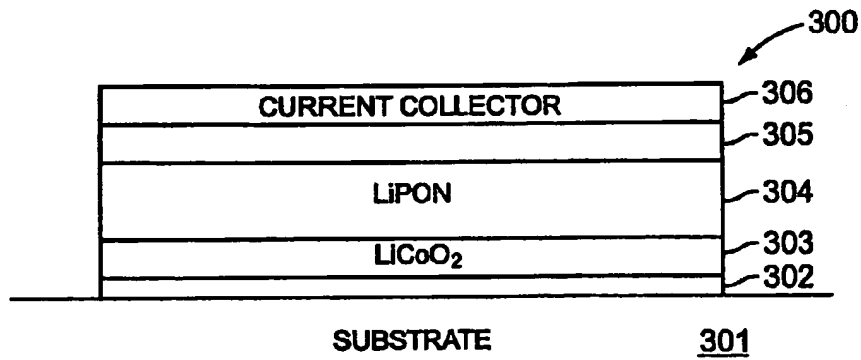


FIG. 3

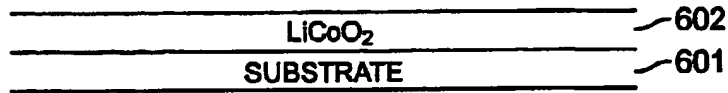


FIG. 6A

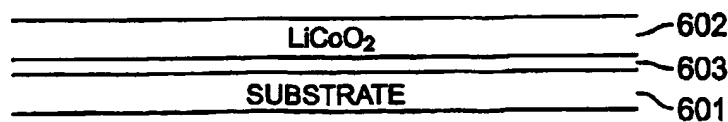


FIG. 6B

SUBSTITUTE SHEET (RULE 26)

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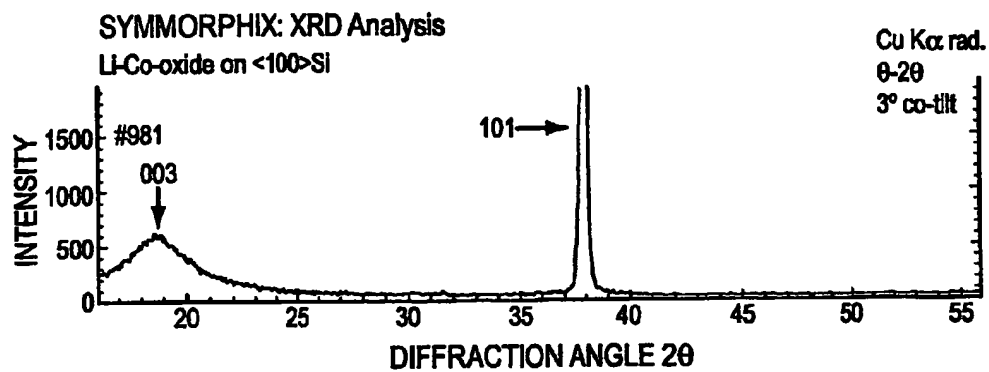


FIG. 4A



FIG. 4B

SUBSTITUTE SHEET (RULE 26)

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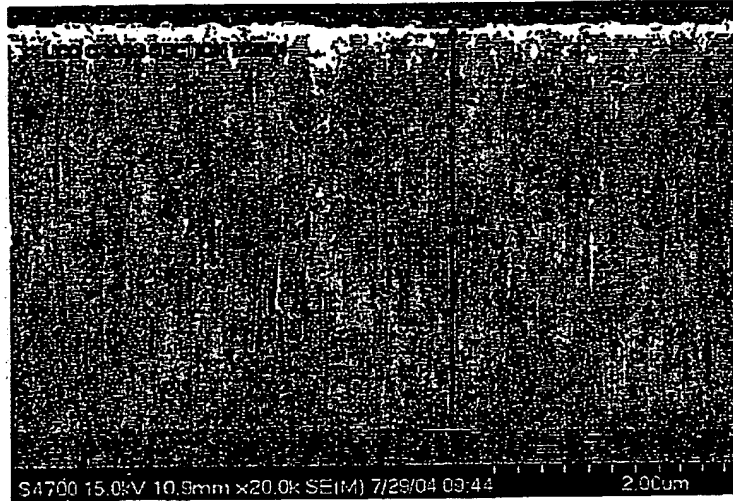


FIG. 5A

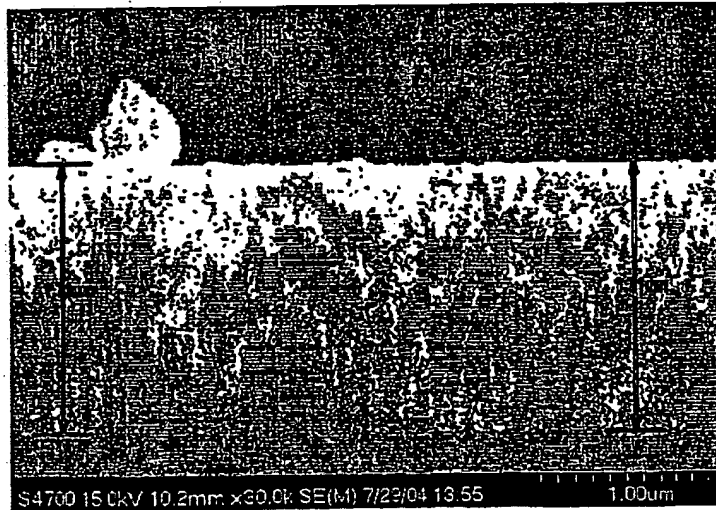


FIG. 5B

SUBSTITUTE SHEET (RULE 26)

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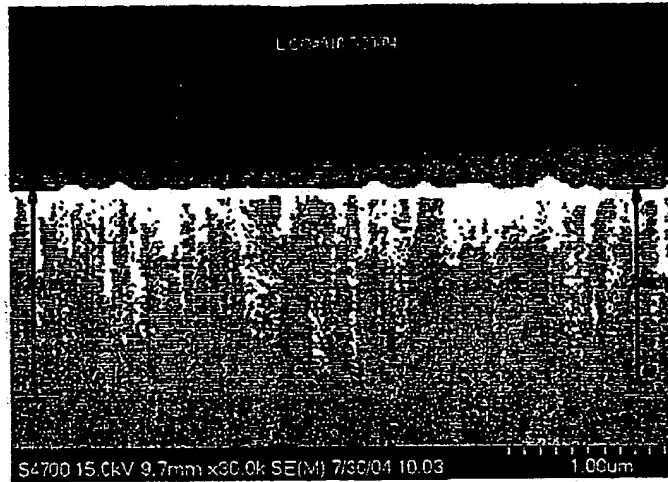


FIG. 5C



FIG. 5D

SUBSTITUTE SHEET (RULE 26)

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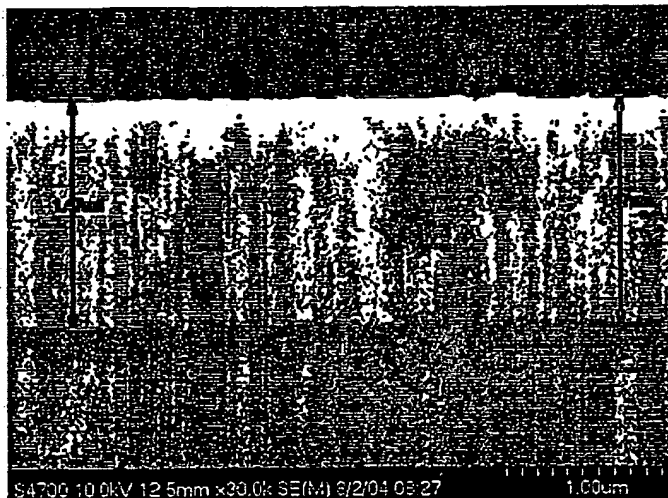


FIG. 5E



FIG. 5F

SUBSTITUTE SHEET (RULE 26)

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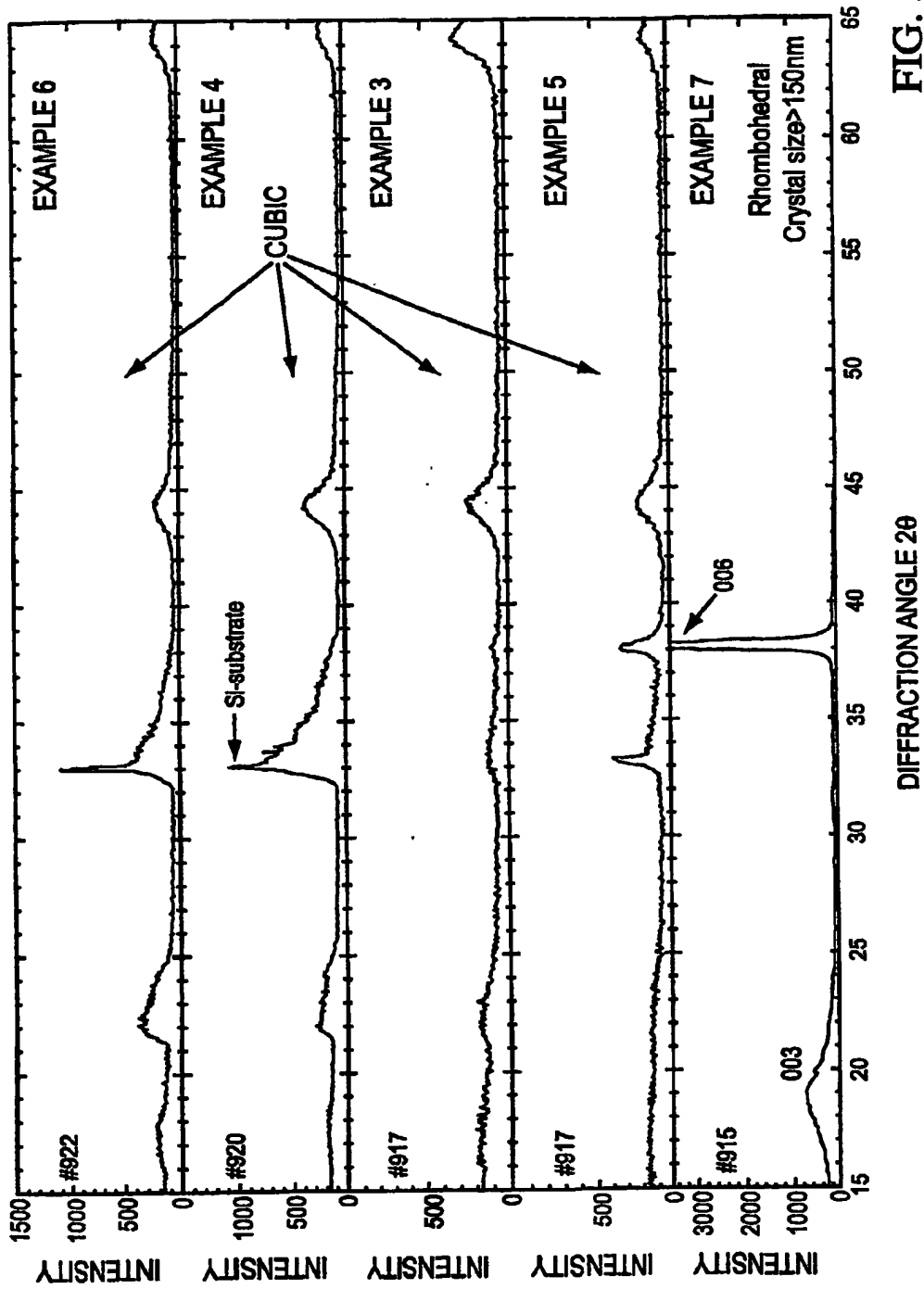


FIG. 5G

SUBSTITUTE SHEET (RULE 26)

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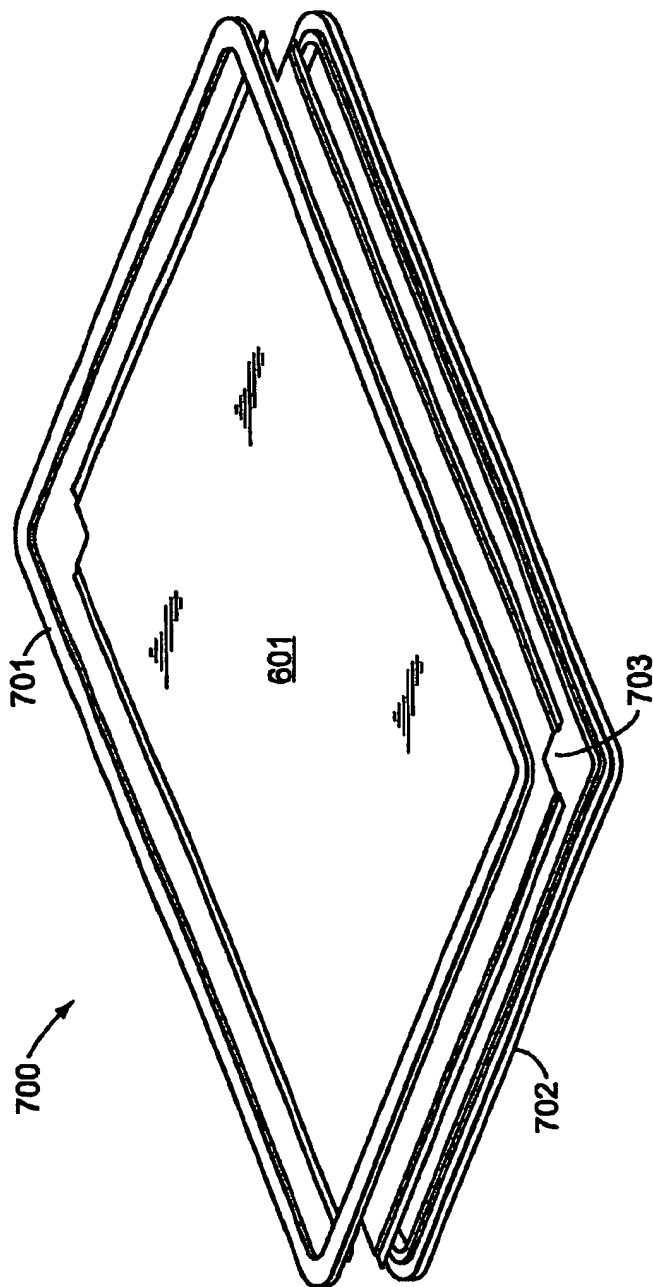


FIG. 7A

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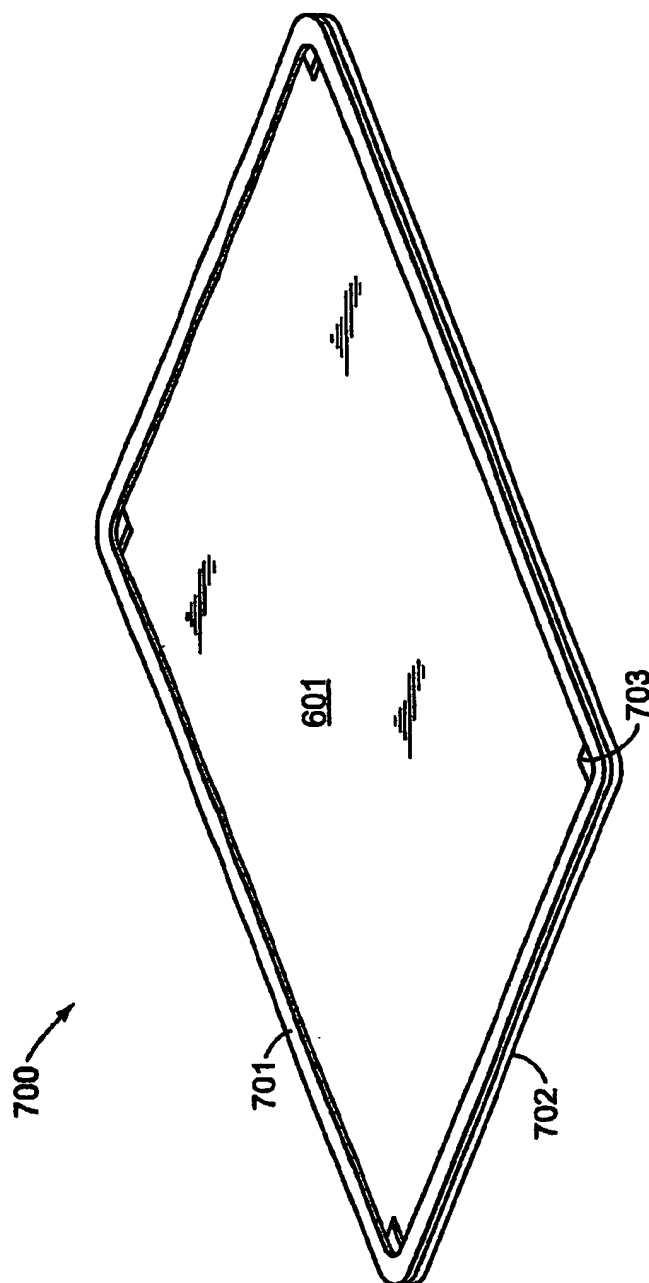


FIG. 7B

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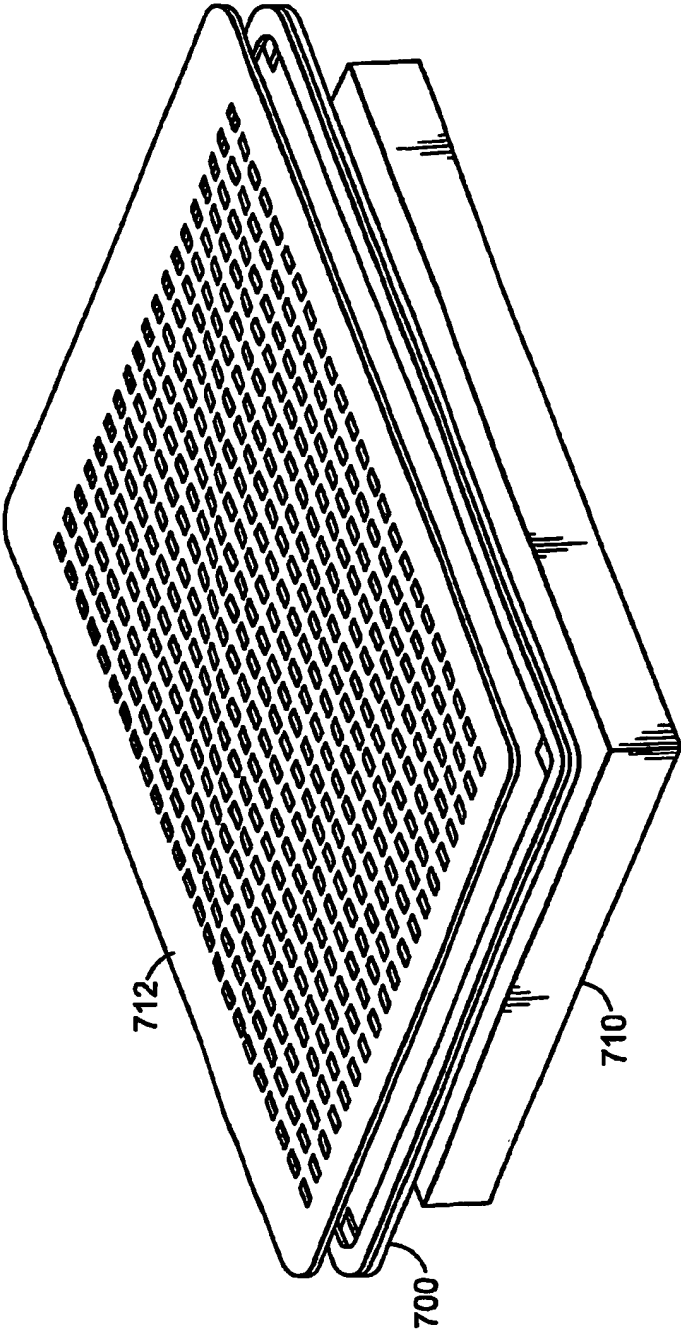


FIG. 7C

SUBSTITUTE SHEET (RULE 26)

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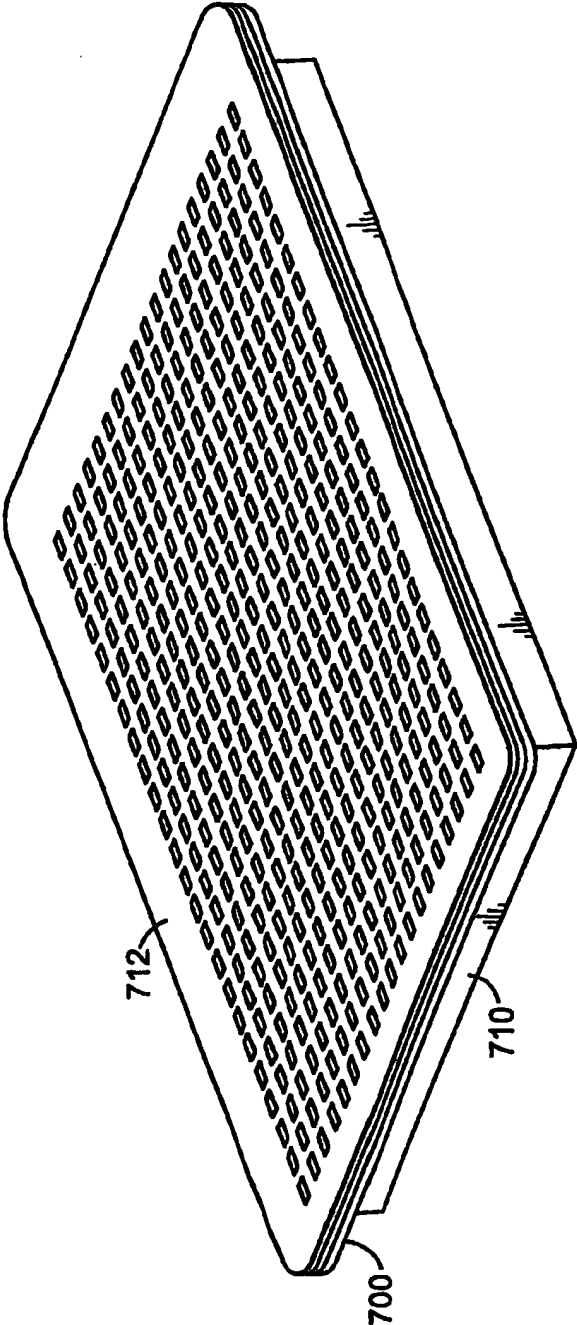


FIG. 7D

SUBSTITUTE SHEET (RULE 26)

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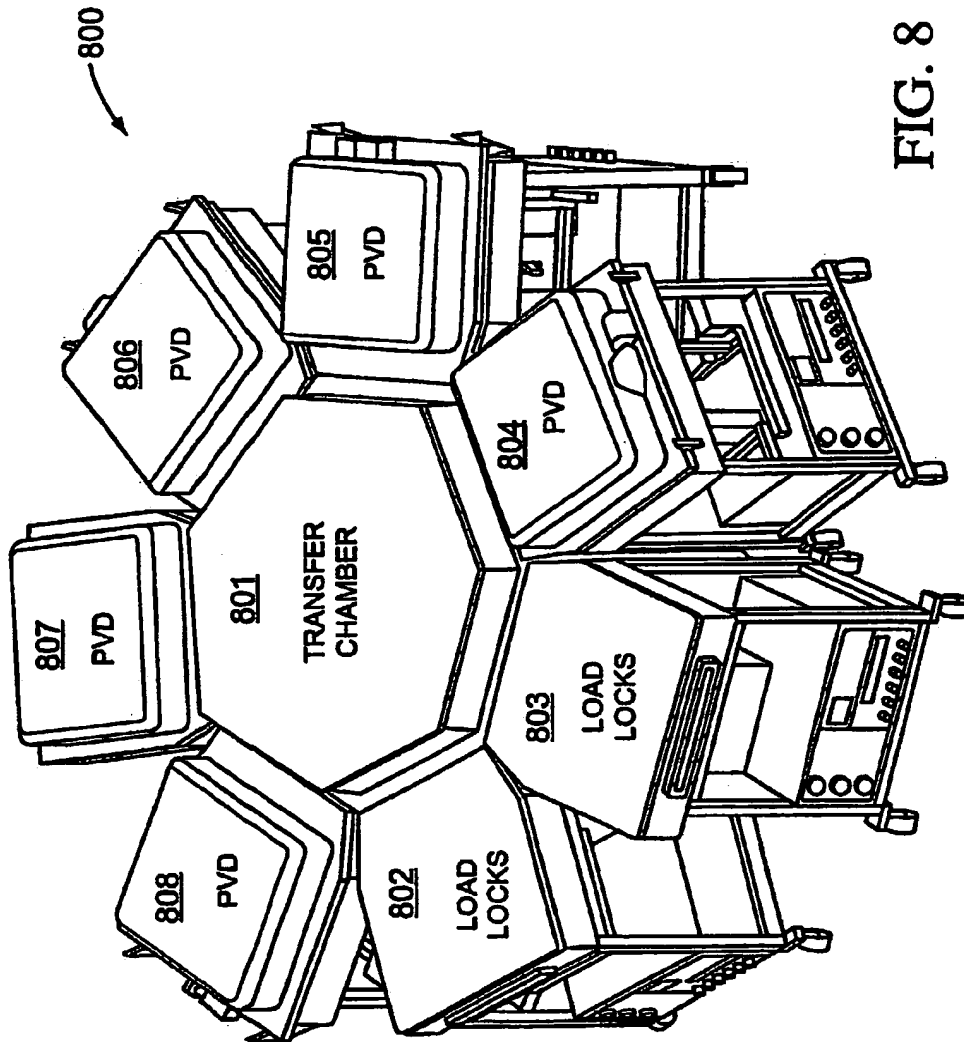


FIG. 8

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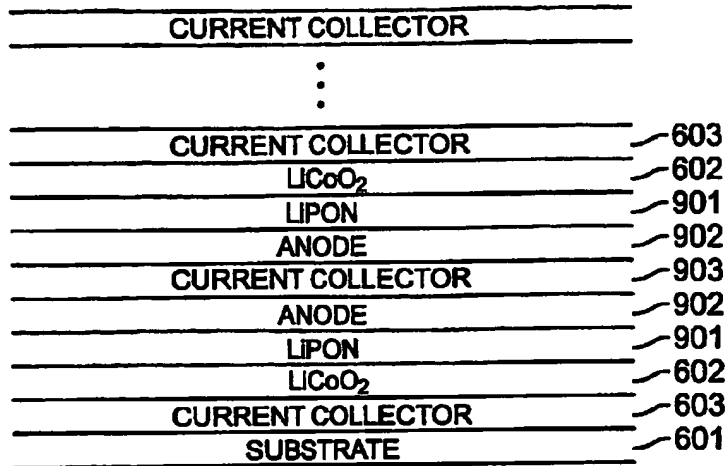


FIG. 9A

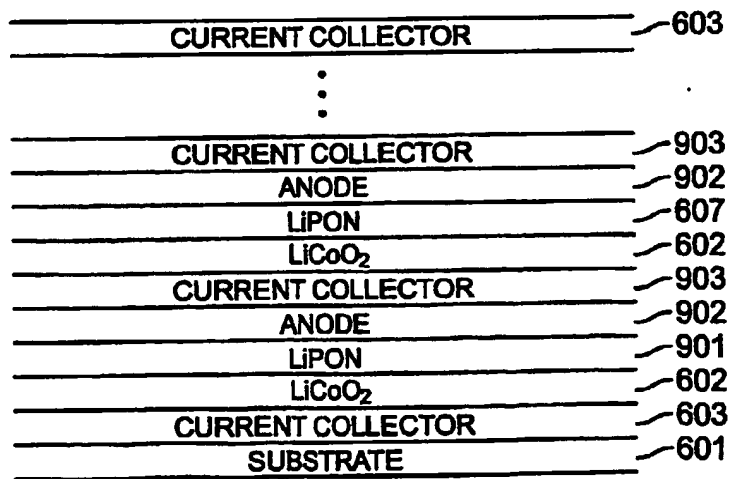
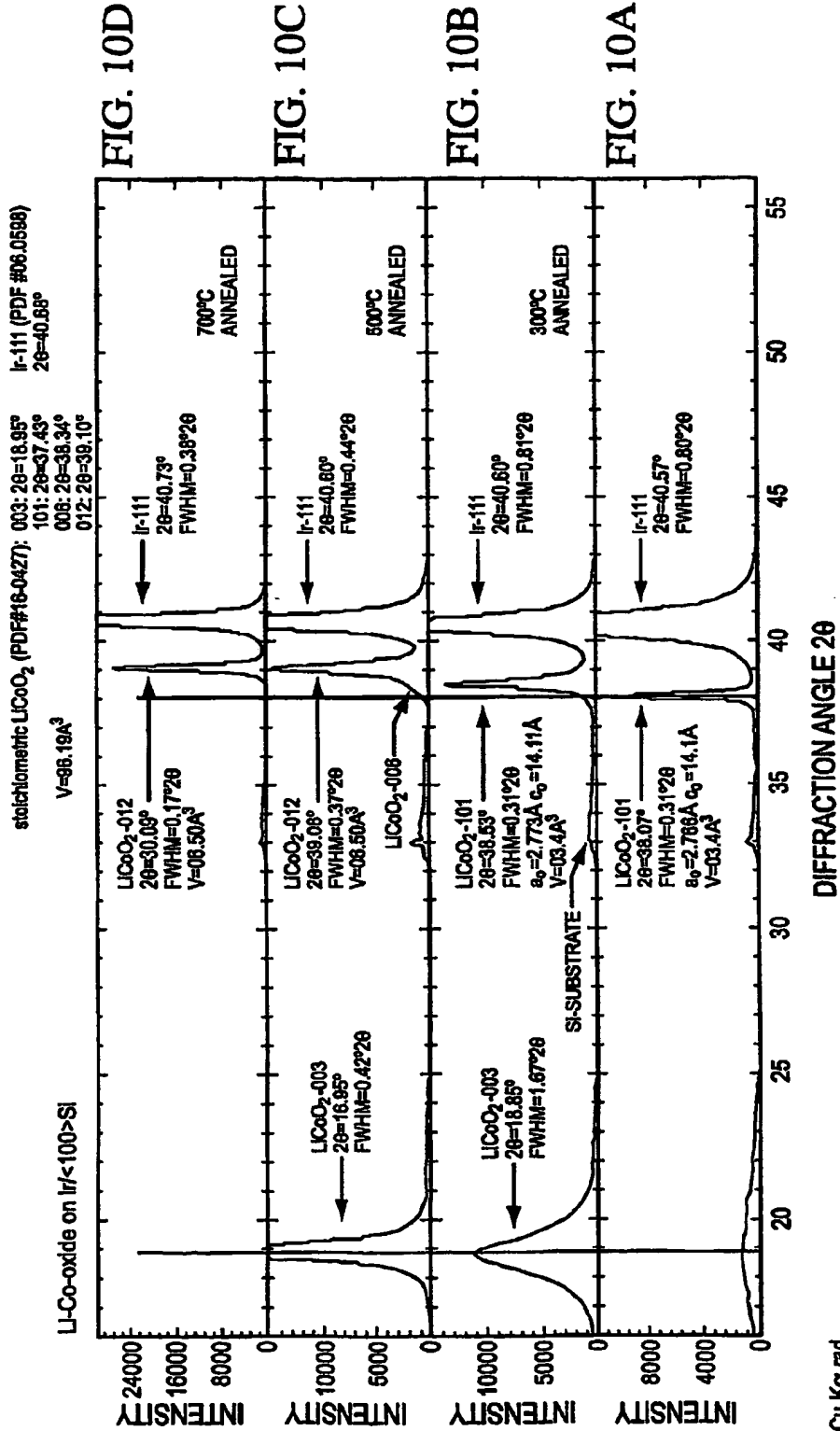


FIG. 9B

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Cu Kα rad.
θ=2θ
° co-tilt

SUBSTITUTE SHEET (RULE 26)

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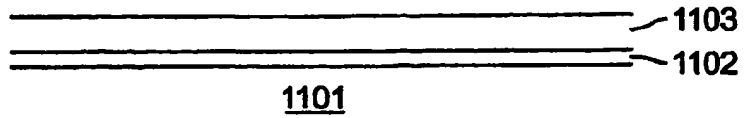


FIG. 11A

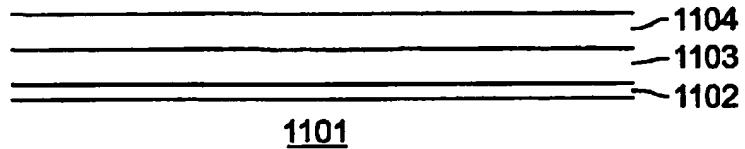


FIG. 11B

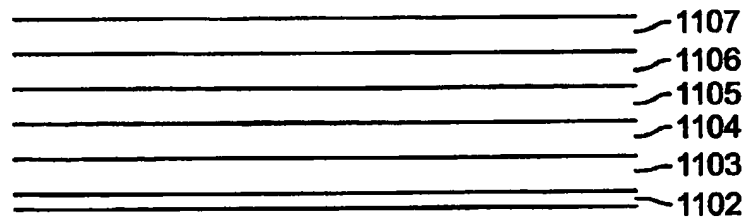


FIG. 11C

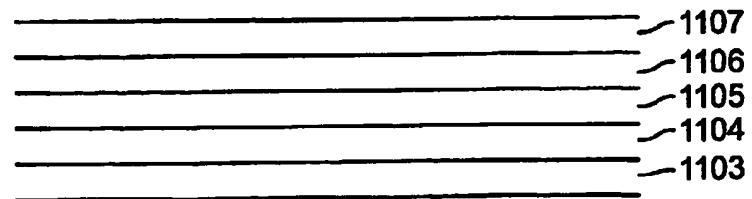


FIG. 11D

SUBSTITUTE SHEET (RULE 26)

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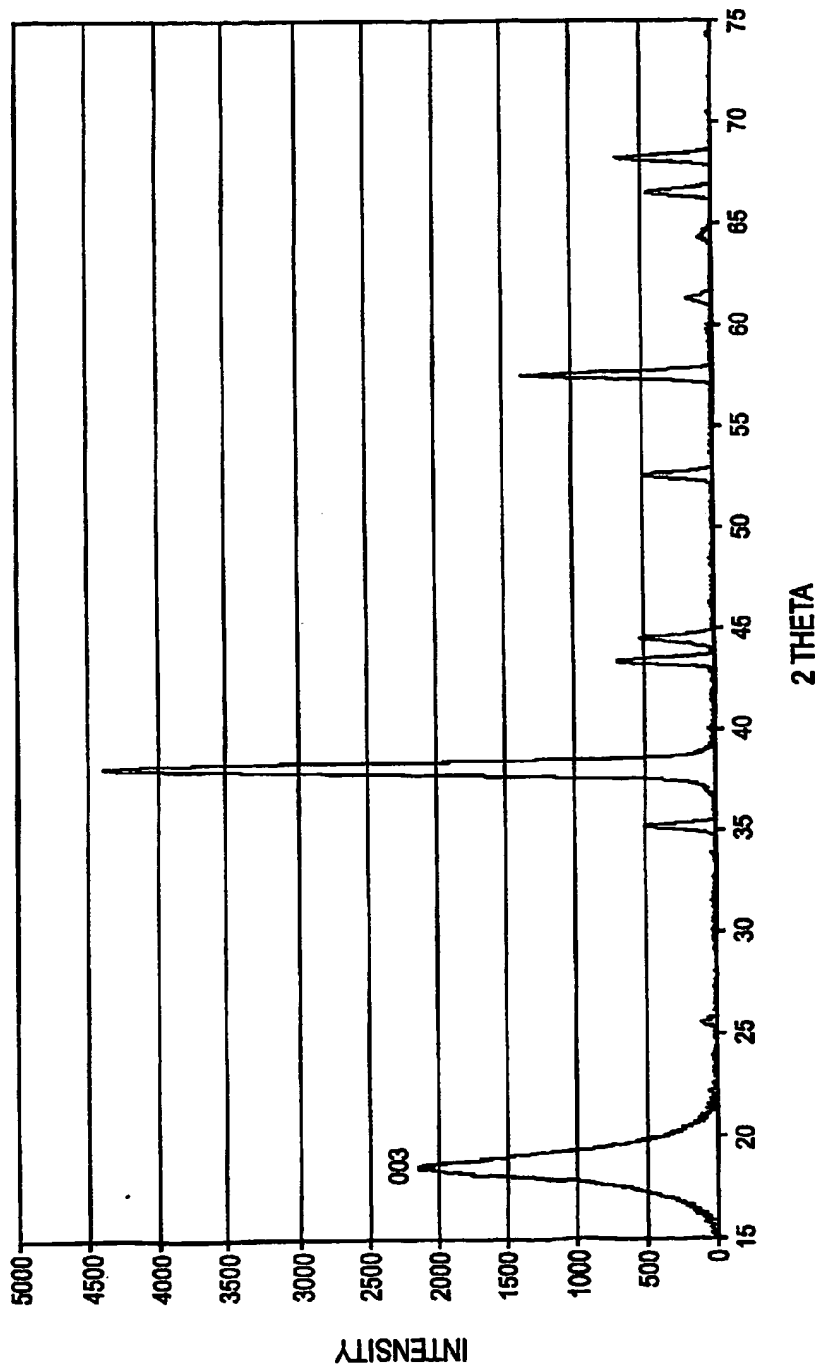
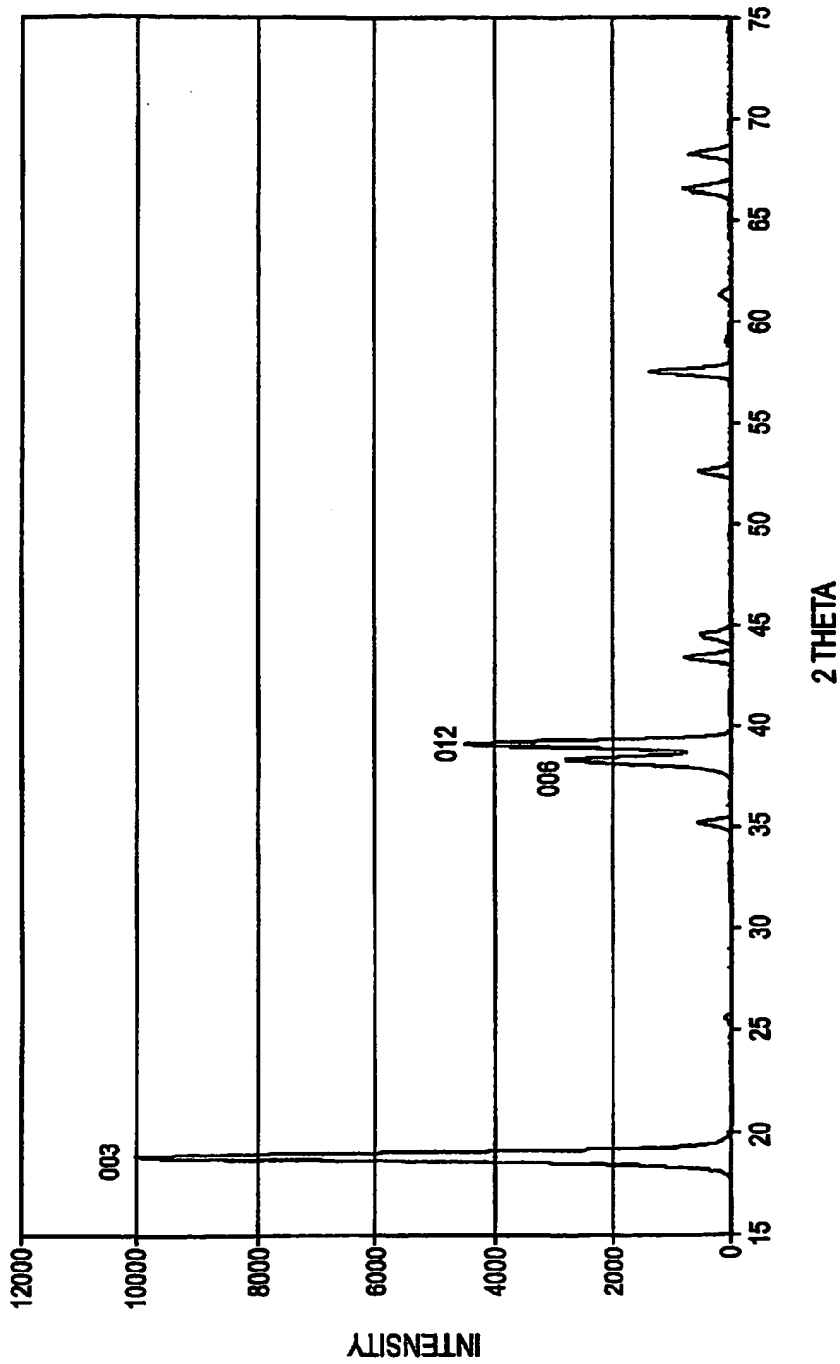


FIG. 12A

SUBSTITUTE SHEET (RULE 26)

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2 THETA

FIG. 12B

SUBSTITUTE SHEET (RULE 26)

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FIG. 12D

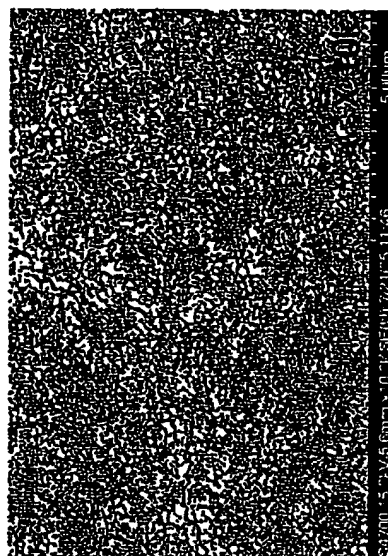


FIG. 12F



FIG. 12C



FIG. 12E

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FIG. 12H

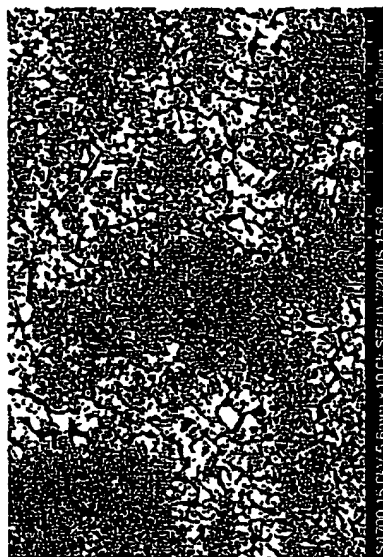


FIG. 12J

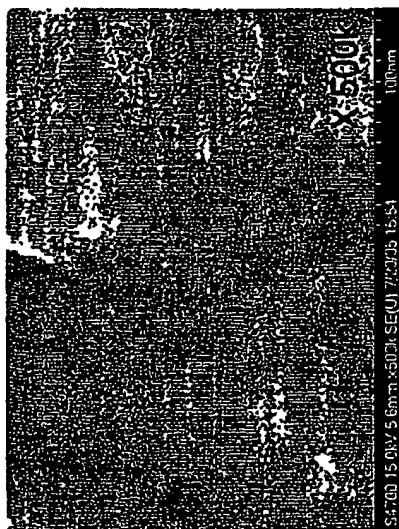
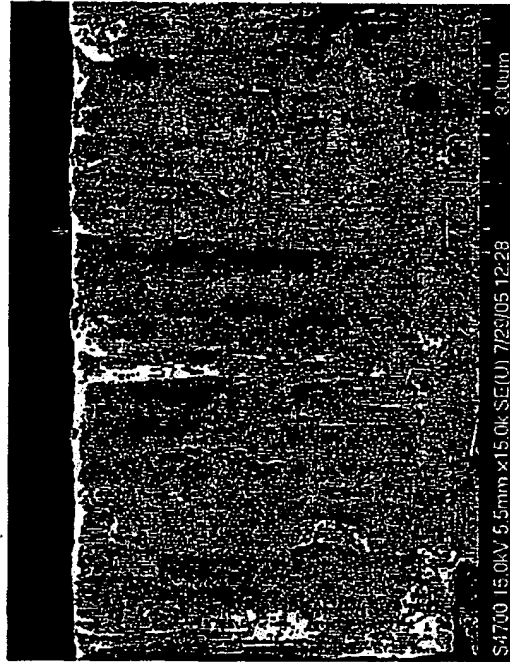


FIG. 12G



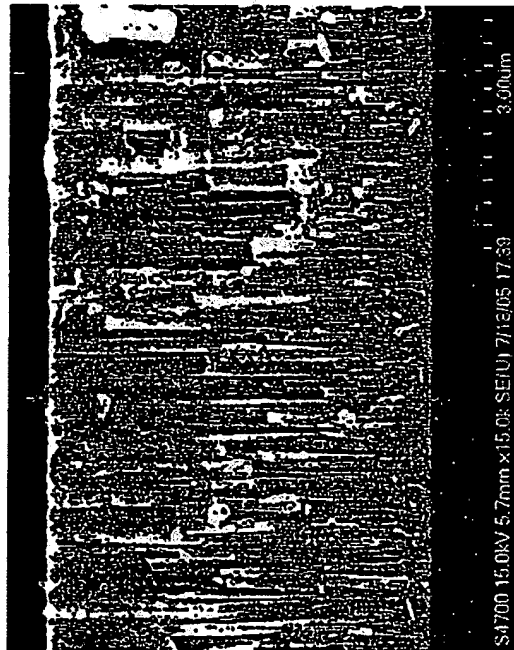
FIG. 12I

FIG. 12L



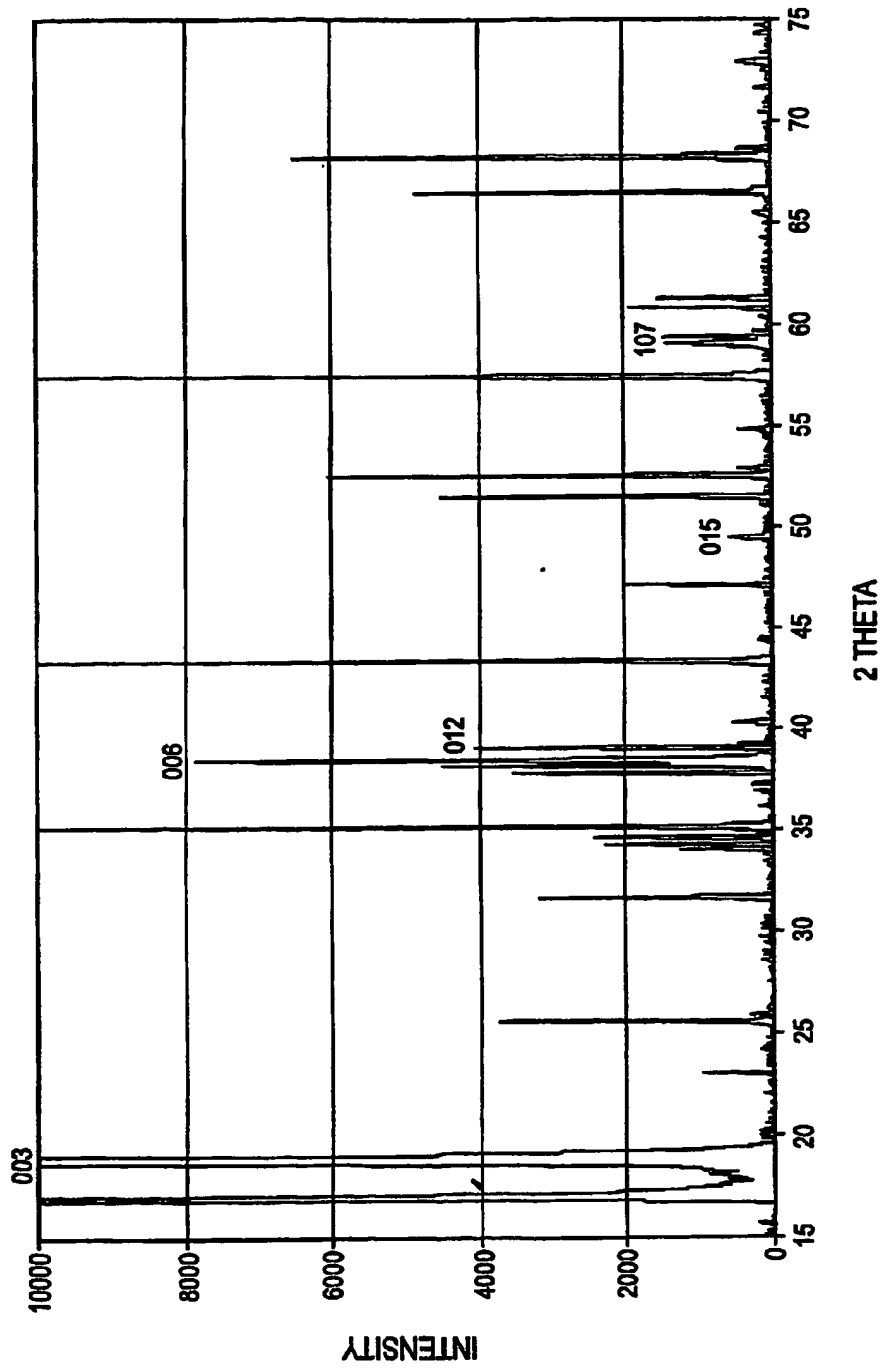
On alumina

FIG. 12K



On Si

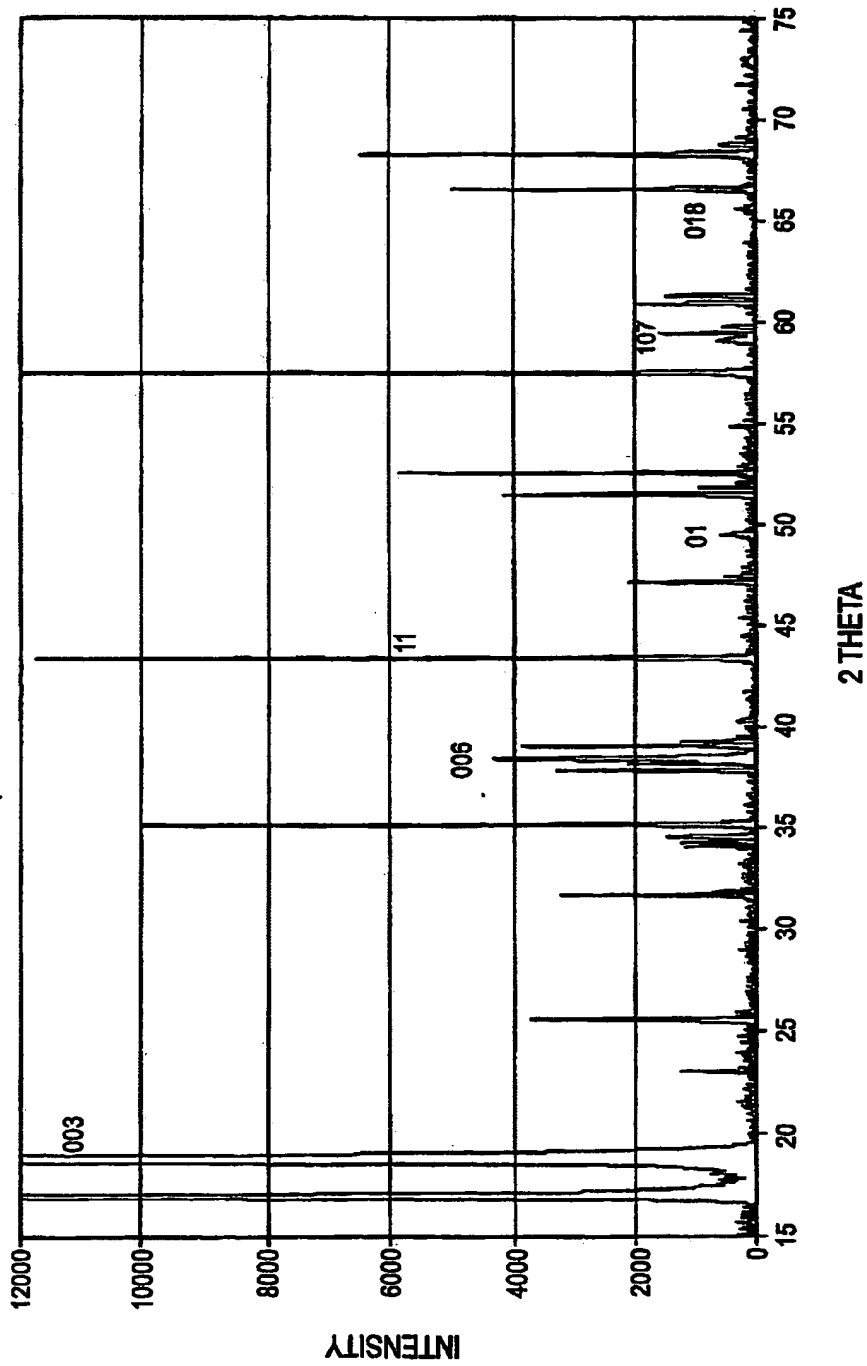
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2 THETA
FIG. 13A

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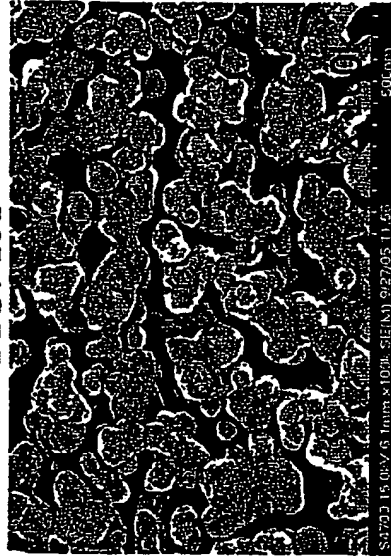
2 THETA
FIG. 13B

SUBSTITUTE SHEET (RULE 26)

FIG. 13E



FIG. 13F



In Ar/O₂ (3/1)

FIG. 13C



FIG. 13D



In Ar

FIG. 14B



700CX2h

FIG. 14D



RTA-700C15m

FIG. 14A



As deposited

FIG. 14C



RTA-5m

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FIG. 15A



FIG. 15B

SUBSTITUTE SHEET (RULE 26)

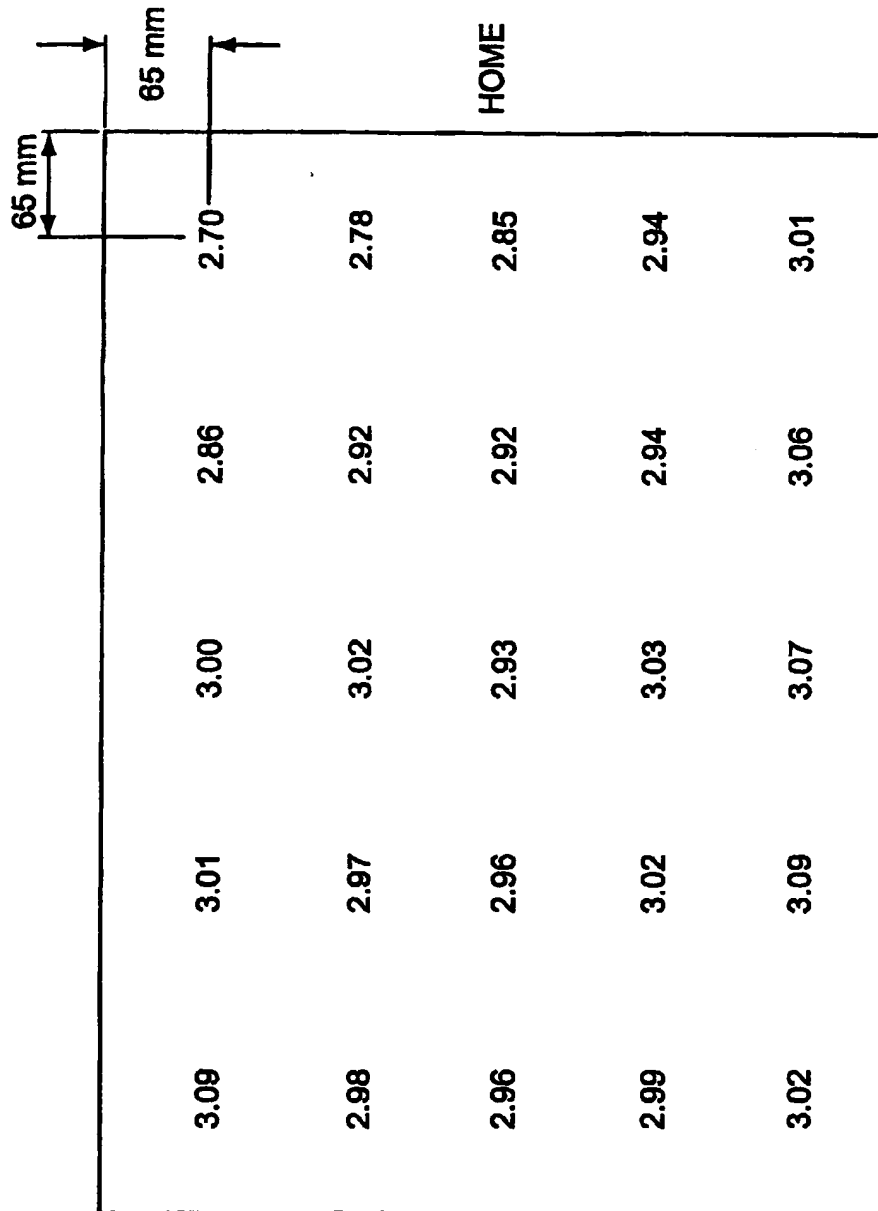


FIG. 16

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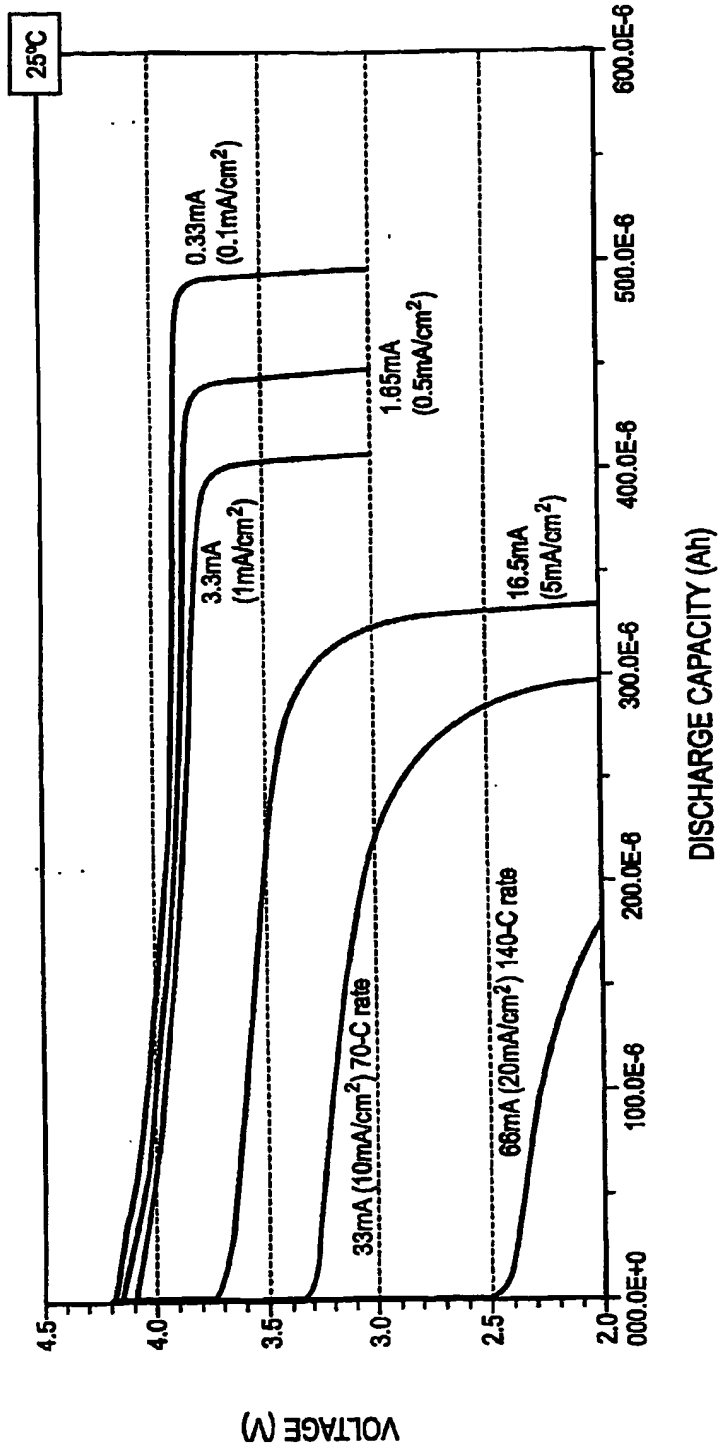


FIG. 17

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PATENT APPLICATION FEE DETERMINATION RECORD
Effective December 8, 2004

10/954182

CLAIMS AS FILED - PART I

	(Column 1)	(Column 2)
TOTAL CLAIMS		
FOR	NUMBER FILED	NUMBER EXTRA
TOTAL CHARGEABLE CLAIMS	minus 20 =	
INDEPENDENT CLAIMS	minus 3 =	
MULTIPLE DEPENDENT CLAIM PRESENT <input type="checkbox"/>		

* If the difference in column 1 is less than zero, enter "0" in column 2

CLAIMS AS AMENDED - PART II

	(Column 1)	(Column 2)	(Column 3)
AMENDMENT A	12-21-05		
	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
Total	31	61	/
Independent	4	9	/
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

	(Column 1)	(Column 2)	(Column 3)
AMENDMENT B	8/9/06		
	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
Total	57	61	/
Independent	5	9	/
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

	(Column 1)	(Column 2)	(Column 3)
AMENDMENT C			
	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
Total			=
Independent			=
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

SMALL ENTITY TYPE

OR OTHER THAN SMALL ENTITY

RATE	FEE		RATE	FEE
BASIC FEE	150.00	OR	BASIC FEE	300.00
X\$ 25=		OR	X\$50=	
X100=		OR	X200=	
+180=		OR	+360=	
TOTAL		OR	TOTAL	

SMALL ENTITY

OR OTHER THAN SMALL ENTITY

RATE	ADDITIONAL FEE		RATE	ADDITIONAL FEE
X\$ 25=	/	OR	X\$50=	/
X100=	/	OR	X200=	/
+180=	/	OR	+360=	/
TOTAL ADDIT. FEE		OR	TOTAL ADDIT. FEE	

RATE	ADDITIONAL FEE
X\$ 25=	/
X100=	/
+180=	/
TOTAL ADDIT. FEE	

RATE	ADDITIONAL FEE
X\$50=	/
X200=	/
+360=	/
TOTAL ADDIT. FEE	

RATE	ADDITIONAL FEE
X\$ 25=	
X100=	
+180=	

RATE	ADDITIONAL FEE
X\$50=	
X200=	
+360=	

10/954/182

APPLICATION NUMBER

?	Rejected	-	(Through Numerals)	Cancelled	N	Non-Elected	A	Appeal
=	Allowed	+	Restricted	I	Interference	O	Objected	

Claim	Date
Final	Original
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49	4
50	4

Claim	Date
Final	Original
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Claim	Date
Final	Original
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If more than 150 claims or 10 actions
staple additional sheet here



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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/954,182	10/01/2004	Hongmei Zhang	09140-0016-01000	9873

22852 7590 10/31/2006

FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER
LLP
901 NEW YORK AVENUE, NW
WASHINGTON, DC 20001-4413

EXAMINER

ESTRADA, MICHELLE

ART UNIT PAPER NUMBER

2823

DATE MAILED: 10/31/2006

Please find below and/or attached an Office communication concerning this application or proceeding.

Office Action Summary

Application No. 10/954,182	Applicant(s) ZHANG ET AL.	
Examiner Michelle Estrada	Art Unit 2823	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

- 1) Responsive to communication(s) filed on 09 August 2006.
- 2a) This action is FINAL.
- 2b) This action is non-final.
- 3) Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims

- 4) Claim(s) 41-43, 45-62, 85 and 87-89 is/are pending in the application.
4a) Of the above claim(s) _____ is/are withdrawn from consideration.
- 5) Claim(s) 61, 62 and 85 is/are allowed.
- 6) Claim(s) 41-43, 45-60 and 87-89 is/are rejected.
- 7) Claim(s) _____ is/are objected to.
- 8) Claim(s) _____ are subject to restriction and/or election requirement.

Application Papers

- 9) The specification is objected to by the Examiner.
- 10) The drawing(s) filed on _____ is/are: a) accepted or b) objected to by the Examiner.
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

Priority under 35 U.S.C. § 119

- 12) Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
a) All b) Some * c) None of:
1. Certified copies of the priority documents have been received.
2. Certified copies of the priority documents have been received in Application No. _____.
3. Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

* See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

- 1) Notice of References Cited (PTO-892)
- 2) Notice of Draftsperson's Patent Drawing Review (PTO-948)
- 3) Information Disclosure Statement(s) (PTO/SB/08)
Paper No(s)/Mail Date 8/9/06.
- 4) Interview Summary (PTO-413)
Paper No(s)/Mail Date. _____
- 5) Notice of Informal Patent Application
- 6) Other: _____

DETAILED ACTION

Claim Rejections - 35 USC § 103

The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

Claims 45, 47, 49, 51, 52, 59 and 60 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff et al. (6,117,279) in view of D'Couto et al. (6,673,716).

Re claims 59 and 60, Smolanoff et al. disclose providing a process gas between the target (16) and a substrate (15); providing pulsed DC power to the target (Col. 5, lines 50-55); providing a magnetic field to the target (Col. 6, lines 1-7); and wherein a material is deposited on the substrate (Col. 5, lines 22-26); and an oxide film is formed by reactive sputtering (Col. 6, lines 15+). Using an specific type of filter (narrow band-rejection) is a matter of design choice depending on the quality of product needed, and it is obvious that the filter is going to work at certain frequencies.

Smolanoff et al. do not disclose wherein the material sputtered is in the metallic mode or in the poison mode.

D'Couto et al. disclose that a material can be sputter neither in the poison mode or metallic mode (Col. 1, line 65-Col. 2, line 20).

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff et al. and D'Couto et al. to enable the sputtering mode step of Smolanoff et al. to be performed according to the teachings of D'Couto et al. because one of ordinary skill in the art would have been motivated to look to alternative suitable methods of performing the disclosed sputtering step of Smolanoff et al. and art recognized suitability for an intended purpose has been recognized to be motivation to combine. See MPEP 2144.07.

Re claim 45, Smolanoff et al. disclose wherein the magnetic field is provided by a moving magnetron (Col. 5, lines 39-49).

Re claim 47, Smolanoff et al. disclose wherein the process gas includes a mixture of oxygen and argon (Col. 7, lines 22-27).

Re claim 49, Smolanoff et al. disclose wherein the process gas further includes nitrogen (Col. 7, lines 25-26).

Re claim 51, Smolanoff et al. disclose further including uniformly sweeping the target with a magnetic field (Col. 6, lines 1-6).

Re claim 52, Smolanoff et al. disclose wherein sweeping the target with a magnetic field includes sweeping a magnet in one direction across the target where the magnet extends beyond the target in the opposite direction (Col. 6, lines 1-6).

Art Unit: 2823

Claims 41, 42, 46, 48 and 50 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff in view of D'Couto et al. as applied to claims 45, 47, 49, 51, 52, 59 and 60 above, and further in view of Chen et al. (2004/0077161).

Re claim 41, Smolanoff et al. disclose wherein the process gas includes oxygen.

Re claim 42, Smolanoff et al. disclose wherein the process gas includes N₂ or NH₃.

The combination of Smolanoff et al. and D'Couto et al. does not disclose wherein the target is a metallic target.

Re claims 41 and 42, Chen et al. disclose forming a coating layer in a substrate; applying a magnetic field to a target; wherein the target is a metallic target.

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff et al., D'Couto et al. and Chen et al. to enable the target material of Smolanoff et al. to be the same according to the teachings of Chen et al. because one of ordinary skill in the art would have been motivated to look to alternative suitable target materials of the disclosed target of Smolanoff et al. and art recognized suitability for an intended purpose has been recognized to be motivation to combine. See MPEP 2144.07.

Re claim 46, Chen et al. disclose further including holding the temperature of the substrate substantially constant (Page 3, Paragraph [0046]).

Re claim 48, Chen et al. disclose wherein the oxygen flow is adjusted by the mass flow controllers; thereby it will adjust the index refraction of the film.

Re claim 50, Chen et al. disclose wherein providing pulsed DC power to a target includes providing pulsed DC power to a target, which has an area larger than that of the substrate (See fig. 3).

Claims 43, 53-58 and 87-89 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff et al. in view of D'Couto et al. as applied to claims 45, 47, 49, 51, 52, 59 and 60 above, and further in view of Milonopoulou et al. (2003/0175142).

Re claim 43, The combination of Smolanoff et al. and D'Couto et al. does not disclose wherein the target is a ceramic target.

Milonopoulou et al. disclose forming a coating layer on a substrate; providing a target (12), which is ceramic (Abstract).

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff et al., D'Couto et al. and Chen et al. to enable the target material of Smolanoff et al. to be the same according to the teachings of Milonopoulou et al. because one of ordinary skill in the art would have been motivated to look to alternative suitable target materials of the disclosed target of Smolanoff et al. and art recognized suitability for an intended purpose has been recognized to be motivation to combine. See MPEP 2144.07.

Using an specific type of filter (narrow band-rejection) is a matter of design choice depending on the quality of product needed, and it is obvious that the filter is going to work at certain frequencies.

Re claim 53, Milonopoulou et al. disclose wherein the target is an alloyed target (Abstract).

Re claim 54, Milonopoulou et al. disclose wherein the alloyed target includes one or more rare earth ions.

Re claim 55, Milonopoulou et al. disclose wherein the alloyed target includes Si and Al.

Re claim 56, Milonopoulou et al. disclose wherein the alloyed target includes one or more elements taken from a set consisting of Si, Al, Er and Yb.

Re claim 57, Milonopoulou et al. disclose wherein the alloyed target is a tiled target.

Re claim 58, Milonopoulou et al. disclose wherein each tiled target is formed by prealloy atomization and hot isostatic pressing of a powder (Page 2, Paragraph [0020]).

Re claim 87-89, using an specific type of filter is a matter of design choice depending on the quality of product needed, and it is obvious that the filter is going to work at certain frequencies. Furthermore, the limitation "the filter is a narrow-band rejection filter rejects power at an RF frequency including an RF bias to the substrate at the RF frequency" is a structural limitation in a method claim, so no matter what filter is used, as long as the same result is achieved, as explained above.

Re claims 88 and 89, one of ordinary skill in the art would have been led to the recited bandwidth and frequency through routine experimentation to achieve a desired band rejection in the process. In addition, the selection of bandwidth and frequency, its obvious because it is a matter of determining optimum process conditions by routine

Art Unit: 2823

experimentation with a limited number of species of result effective variables. These claims are prima facie obvious without showing that the claimed ranges achieve unexpected results relative to the prior art range. In re Woodruff, 16 USPQ2d 1935, 1937 (Fed. Cir. 1990). See also In re Huang, 40 USPQ2d 1685, 1688 (Fed. Cir. 1996)(claimed ranges or a result effective variable, which do not overlap the prior art ranges, are unpatentable unless they produce a new and unexpected result which is different in kind and not merely in degree from the results of the prior art). See also In re Boesch, 205 USPQ 215 (CCPA) (discovery of optimum value of result effective variable in known process is ordinarily within skill or art) and In re Aller, 105 USPQ 233 (CCPA 1995) (selection of optimum ranges within prior art general conditions is obvious).

Note that the specification contains no disclosure of either the critical nature of the claimed bandwidth and frequency or any unexpected results arising therefrom. Where patentability is said to be based upon particular chosen bandwidth and frequency or upon another variable recited in a claim, the Applicant must show that the chosen bandwidth and frequency are critical. *In re Woodruff*, 919 F.2d 1575, 1578, 16 USPQ2d 1934, 1936 (Fed. Cir. 1990).

Allowable Subject Matter

Claims 61, 62 and 85 are allowed.

Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Michelle Estrada whose telephone number is 571-272-1858. The examiner can normally be reached on Monday through Friday.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Matthew Smith can be reached on 571-272-1907. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

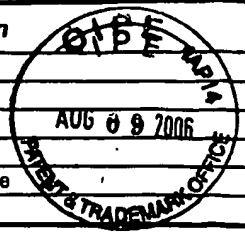
Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 571-272-2800.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).


Michelle Estrada
Primary Examiner
Art Unit 2823

ME
October 30, 2006

IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known		
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>			Application Number	10/854,182	
			Filing Date	October 1, 2004	
			First Named Inventor	ZHANG, Hongmei	
			Art Unit	2823	
			Examiner Name	ESTRADA, Michelle	
Sheet	1	of	3	Attorney Docket Number	9140.0016-01



U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS						
Examiner Initials	Cite No. ¹	Document Number		Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)				
<i>ME</i>		US 4,082,569		04/04/1978	Evans, Jr.	
		US 4,710,940		12/01/1987	Sipes, Jr.	
		US 4,785,459		11/15/1988	Baer	
		US 5,435,826		07/25/1995	Sakakibara et al.	
		US 5,472,795		12/05/1995	Atita	
		US 5,512,147		04/30/1996	Bates et al.	
		US 5,569,520		10/29/1996	Bates	
		US 5,597,660		01/28/1997	Bates et al.	
		US 5,612,152		03/18/1997	Bates	
		US 5,645,626		07/08/1997	Edlund et al.	
		US 5,702,829		12/30/1997	Paidassi et al.	
		US 6,045,626		04/04/2000	Yano et al.	
		US 6,168,884 B1		01/02/2001	Neudecker et al.	
		US 6,236,793 B1		05/22/2001	Lawrence et al.	
		US 6,242,132 B1		06/05/2001	Neudecker et al.	
		US 6,306,265 B1		10/23/2001	Fu et al.	
		US 6,365,300 B1		04/02/2002	Ota et al.	
		US 6,760,520 B1		07/06/2004	Medin et al.	
		US 6,818,356 B1		11/16/2004	Bates	
		US 6,884,327 B2		04/26/2005	Pan et al.	
		US 2001/0031122 A1		10/18/2001	Lackritz et al.	
		US 2002/0014406 A1		02/07/2002	Takashima	
		US 2002/0115252 A1		08/22/2002	Haukka et al.	
		US 2003/0035906 A1		02/20/2003	Memarian et al.	
		US 2003/0178637 A1		09/25/2003	Chen et al.	
		US 2003/0185266 A1		10/02/2003	Henrichs	
<i>ME</i>		US 2004/0043557 A1		03/04/2004	Haukka et al.	

Examiner Signature	<i>Michelle Estrada</i>	Date Considered	10/30/06
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

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IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known		
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <small>(Use as many sheets as necessary)</small>			Application Number	10/954,182	
			Filing Date	October 1, 2004	
			First Named Inventor	ZHANG, Hongmei	
			Art Unit	2823	
			Examiner Name	ESTRADA, Michelle	
Sheet	2	of	3	Attorney Docket Number	9140.0016-01

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS				
<i>me</i>	US 2005/0183946 A1	08/25/2005	Pan et al.	
	US 2006/0054496 A1	03/16/2006	Zhang et al.	
	US 2006/0057283 A1	03/16/2006	Zhang et al.	
	US 2006/0057304 A1	03/16/2006	Zhang et al.	
<i>me</i>	US 2006/0071592 A1	04/06/2006	Narasimhan et al.	
	US 2006/0134522 A1	06/22/2006	Zhang et al.	

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

FOREIGN PATENT DOCUMENTS						
Examiner Initials ⁵	Cite No. ¹	Foreign Patent Document	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁶
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)				
<i>me</i>		DE 37 38 738 C1	01/26/1989	Degussa AG		
		EP 1 092 689 A1	04/18/2001	BPS Alzenau GmbH		Abstract
		JP 5-230642 A	09/07/1993	Nissin High Voltage Co., Ltd.		
		JP 7-224379 A	08/22/1995	Ulvac Japan Ltd		Abstract
<i>me</i>		WO 99/61674 A1	12/02/1999	Universiteit Gent		
		WO 2006/063308 A2	06/15/2006	Symmorphix, Inc.		

NON PATENT LITERATURE DOCUMENTS			
Examiner Initials ⁵	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
<i>me</i>		ASM Handbook, Formerly Ninth Edition, Metals Handbook, Volume 15, Casting, Davis et al. (Eds.), ASM International, pp. 372-373, 376-383, and 410-411 (1988).	
		COCORULLO, G. et al., "Amorphous silicon waveguides and light modulators for integrated photonics realized by low-temperature plasma-enhanced chemical-vapor deposition," <i>Optics Lett.</i> 21(24):2002-2004 (1996).	
<i>me</i>		KELLY, P.J. et al., "A novel technique for the deposition of aluminum-doped zinc oxide films," <i>Thin Solid Films</i> 426(1-2):111-116 (2003).	
		TOMASZEWSKI, H. et al., "Ytria-stabilized zirconia thin films grown by reactive r.f. magnetron sputtering," <i>Thin Solid Films</i> 287:104-109 (1996).	

Examiner Signature	<i>Michelle Estrada</i>	Date Considered	10/30/04
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

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IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known		
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>			<i>Application Number</i>	10/954,182	
			<i>Filing Date</i>	October 1, 2004	
			<i>First Named Inventor</i>	ZHANG, Hongmei	
			<i>Art Unit</i>	2823	
			<i>Examiner Name</i>	ESTRADA, Michelle	
<i>Sheet</i>	3	<i>of</i>	3	<i>Attorney Docket Number</i>	8140.0016-01

NON PATENT LITERATURE DOCUMENTS		
[initials]		Response to Final Office Action filed April 14, 2006, in U.S. Appl. No. 10/291,179 (Atty. Docket No. 9140.0001-00).
		Office Action mailed April 27, 2006, in U.S. Appl. No. 10/291,179 (Atty. Docket No. 9140.0001-00).
		Response to Office Action filed July 27, 2006, in U.S. Appl. No. 10/291,179 (Atty. Docket No. 9140.0001-00).
		Notice of Allowance mailed August 6, 2002, for US Patent No. 6,506,289 (Atty. Docket No. 09140.0002-01).
		Response to Office Action filed February 28, 2006 in U.S. Application No. 09/903,081 (Atty. Docket No. 09140-0014-00).
		Final Office Action mailed May 8, 2006 in U.S. Application No. 09/903,081 (Atty. Docket No. 09140-0014-00).
		Final Office Action mailed June 9, 2006 in U.S. Appl. No. 11/100,856 (Atty. Docket No. 09140.0015-01).
		Office Action mailed March 22, 2006, in U.S. Appl. No. 10/101,863 (Atty. Docket No. 09140.0016-00).
		Response to Office Action filed June 12, 2006, in U.S. Appl. No. 10/101,863 (Atty. Docket No. 09140.0016-00).
		Response to Office Action filed on May 15, 2006, in U.S. Application No. 10/101,341 (Atty. Docket No. 09140-0017-00).
		Office Action issued on August 2, 2006, in U.S. Application No. 10/101,341 (Atty. Docket No. 09140-0017-00).
		Office Action issued on March 23, 2006, in U.S. Application No. 10/650,461 (Atty. Docket No. 09140-0025-00).
		Response to Office Action filed on July 24, 2006, in U.S. Application No. 10/650,461 (Atty. Docket No. 09140-0025-00).
		Response to Office Action filed March 2, 2006 in U.S. Application No. 10/789,953 (Atty. Docket No. 09140.0030-00).
		Final Office Action issued on May 19, 2006 in U.S. Application No. 10/789,953 (Atty. Docket No. 09140.0030-00).
		Office Action from Singapore Patent Office in Appl. No. 200505388-9, dated March 20, 2006 (Atty. Docket No. 9140.0030-00256).
		Office Action mailed April 19, 2006 in U.S. Application No. 10/851,542 (Atty. Docket No. 09140.0033-00).
		Response to Office Action filed July 26, 2006 in U.S. Application No. 10/851,542 (Atty. Docket No. 09140.0033-00).
[initials]		Specification as filed September 2, 2005, for U.S. Appl. No. 11/218,652 (Atty. Docket No. 09140.0052-00).

<i>Examiner Signature</i> Michelle Estrada	<i>Date Considered</i> 10/30/06
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

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12-08-06

16W



PATENT
Customer No. 22,852
Attorney Docket No. 9140.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
)	
ZHANG, Hongmei et al.)	Group Art Unit: 2823
)	
Application No.: 10/954,182)	Examiner: ESTRADA, Michelle
)	
Filed: October 1, 2004)	
)	Confirmation No.: 9873
For: BIASED PULSE DC REACTIVE)	
SPUTTERING OF OXIDE FILMS)	

MAIL STOP AMENDMENT
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

AMENDMENT AND RESPONSE TO OFFICE ACTION

In reply to the Office Action mailed October 31, 2006, please amend the above-identified application as follows:

Amendments to the Claims are reflected in the listing of claims that begins on page 2 of this paper.

Remarks/Arguments follow the amendment sections on page 6 of this paper.

AMENDMENTS TO THE CLAIMS:

This listing of claims will replace all prior versions and listings of claims in the application:

Claims 1-40 (Canceled).

Claim 41 (Currently amended): The method of claims ~~59, 60, 61~~ or 85, wherein the target is a metallic target and the process gas includes oxygen.

Claim 42 (Currently amended): The method of claims ~~43, 59, 60, 61~~ or 85, wherein the target is a metallic target and the process gas includes one or more of a set consisting of N₂, NH₃, CO, NO, CO₂, halide containing gasses.

Claim 43 (Currently amended): ~~A method of depositing a film on a substrate, comprising:~~
~~providing a process gas between a target and a substrate;~~
~~providing pulsed DC power to the target through a narrow band rejection filter;~~
~~providing a magnetic field to the target; and~~
~~wherein a material is deposited on the substrate, The method of claims 61 or 85~~
wherein the target is a ceramic target.

Claim 45 (Currently amended): The method of claims ~~43, 59, 60, 61~~ or 85, wherein the magnetic field is provided by a moving magnetron.

Claim 46 (Currently amended) The method of claims ~~43, 59, 60, 61~~ or 85, further including holding the temperature of the substrate substantially constant.

Claim 47 (Currently amended): The method of claims ~~43, 59, 60, 61~~ or 85, wherein the process gas includes a mixture of Oxygen and Argon.

Claim 48 (Currently amended): The method of claims ~~43, 59, 60, 61~~ or 85, wherein the

Oxygen flow is adjusted to adjust the index of refraction of the film.

Claim 49 (Currently amended): The method of claims ~~43, 59, 60, 61~~ or 85, wherein the process gas further includes nitrogen.

Claim 50 (Currently amended): The method of claims ~~43, 59, 60, 61~~ or 85, wherein providing pulsed DC power to a target includes providing pulsed DC power to a target which has an area larger than that of the substrate.

Claim 51 (Currently amended): The method of claims ~~43, 59, 60, 61~~ or 85, further including uniformly sweeping the target with a magnetic field.

Claim 52 (Previously presented): The method of claim 51 wherein uniformly sweeping the target with a magnetic field includes sweeping a magnet in one direction across the target where the magnet extends beyond the target in the opposite direction.

Claim 53 (Currently amended): The method of claims ~~43, 59, 60, 61~~ or 85, wherein the target is an alloyed target.

Claim 54 (Previously presented): The method of claim 53 wherein the alloyed target includes one or more rare-earth ions.

Claim 55 (Previously presented): The method of claim 53 wherein the alloyed target includes Si and Al.

Claim 56 (Previously presented): The method of claim 53 wherein the alloyed target includes one or more elements taken from a set consisting of Si, Al, Er, Yb, Zn, Ga, Ge, P, As, Sn, Sb, Pb, Ag, Au, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Tm, and Lu.

Claim 57 (Previously presented): The method of claim 53 wherein the alloyed target is a tiled target.

Claim 58 (Previously presented): The method of claim 57 wherein each tile of the tiled target is formed by prealloy atomization and hot isostatic pressing of a powder.

Claims 59-60 (Canceled).

Claim 61 (Currently amended): A method of depositing a film on a substrate, comprising:

providing a process gas between a ~~metallie~~ conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning a ~~metallie~~ the target;
wherein a material is deposited on the substrate.

Claim 62 (Currently amended): The method of claim 61, wherein reconditioning the ~~metallie~~ target includes:
reactive sputtering in the metallic mode and then reactive sputtering in the poison mode.

Claims 63-84 (Canceled).

Claim 85 (Previously presented): A method of depositing a film on a substrate, comprising:
providing a process gas between a target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
wherein a material is deposited on the substrate, and an oxide film is formed by reactive sputtering in a mode between a metallic mode and a poison mode.

Claim 86 (Canceled).

Claim 87 (Currently amended): The method according to claims ~~43, 59, 60, 61, 62,~~ or 85, ~~wherein the~~ further including a narrow band-rejection filter that rejects power at an RF frequency, and further including
providing an RF bias to the substrate at the RF frequency.

Claim 88 (Previously presented): The method according to claim 87, wherein the narrow band-rejection filter has a bandwidth of about 100 kHz.

Claim 89 (Previously presented): The method according to claim 87, wherein the RF frequency is about 2 MHz.

Claims 90-92 (Canceled).

REMARKS

Claims 41-43, 45-62, 85, and 87-89 are pending in present application. The Examiner has rejected claims 41-43 and 45-60, objected to claims 87-89, and allowed claims 61, 62, and 85. In this Amendment, claims 41-43, 45-51, 53, 61, and 87 have been amended and claims 59-60 have been canceled. Claims 41-43, 45-51, and 87 have been amended to depend from claims 61 or 85.

The Examiner has allowed independent claims 61 and 85. Claim 61 has been amended to replace “metallic” with “conductive” and therefore is still allowable. Claims 41-43, 45-51, 53, and 87 have been amended to depend from allowed claims 61 or 85 and are therefore allowable for at least the same reasons as is claim 61 and 85. Applicants reserve the right to pursue claims 59-60 and claims depending from them in a separate application.¹

Claim Rejections under 35 U.S.C. § 103

Claims 45, 47, 49, 51, 52, 59, and 60

The Examiner rejected claims 45, 47, 49, 51, 52, 59, and 60 under 35 U.S.C. § 103(a) as being unpatentable over U.S. Patent No. 6,117,279 (“Smolonoff et al.”) in view of U.S. Patent No. 6,673,716 (“D’Couto et al.”).

Claims 59 and 60 have been canceled. Claims 45, 47, 49, 51, and 52 have been amended to depend from allowed claims 61 and 85. Therefore, claims 45, 47, 49, 51, and 52 are allowable for at least the same reasons as is claims 61 and 85.

¹ The Examiner has made many assertions characterizing both the claims and the prior art. Applicants do not necessarily agree with these assertions and nothing in this response should be interpreted such that Applicants acquiesce in any way in the Examiner’s characterizations.

Claims 41, 42, 46, 48, and 50

The Examiner rejected claims 41, 42, 46, 68, and 50 under 35 U.S.C. § 103(a) as being unpatentable over Smolonoff et al. in view of D'Couto et al. as applied to claims 45, 47, 49, 51, 52, 59, and 60, and further in view of U.S. Published Application No. 2004/0077161 ("Chen et al.").

Claims 41, 42, 46, 68, and 50 have been amended to depend from allowed claims 61 and 85 and are therefore allowable for at least the same reasons as is claims 61 and 85.

Claims 43, 53-58, and 87-89

The Examiner has rejected claims 43, 53-58, and 87-89 under 35 U.S.C. § 103(a) as being unpatentable over Smolonoff et al. in view of D'Couto et al. as applied to claims 45, 47, 49, 51, 52, 59, and 60 above, and further in view of U.S. Published Application No. 2003/0175142 ("Milonopoulou et al.").

The Examiner is reminded that Milonopoulou is not prior art to this application. Milonopoulou was filed on the same day by many of the same inventors as the present application and is assigned to the same entity as the present application.

Additionally, claims 45, 47, 49, 51, and 52 have been amended to depend from allowed claims 61 and 85 and are allowable for at least the same reasons as is claims 61 and 85.

Allowable Subject Matter

The Examiner has indicated that claims 61, 62, and 85 are allowed.


Conclusion

In view of the foregoing amendments and remarks, Applicants respectfully request reconsideration and reexamination of this application and the timely allowance of the pending claims.

Please grant any extensions of time required to enter this response and charge any additional required fees to Deposit Account No. 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

By:  *Reg. No. 56,170*

Gary J. Edwards
Reg. No. 41,008

Date: December 6, 2006

**EXPRESS MAIL LABEL NO.
EV 746096154 US**



PATENT
Customer No. 22,852
Attorney Docket No. 9140.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
)	
ZHANG, Hongmei et al.)	Group Art Unit: 2823
)	
Application No.: 10/954,182)	Examiner: ESTRADA, Michelle
)	
Filed: October 1, 2004)	
)	Confirmation No.: 9873
For: BIASED PULSE DC REACTIVE)	
SPUTTERING OF OXIDE FILMS)	

MAIL STOP AMENDMENT

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

SIXTH SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT
UNDER 37 C.F.R. § 1.97(c)

Pursuant to 37 C.F.R. §§ 1.56 and 1.97(c), Applicants bring to the attention of the Examiner the documents on the attached listing. This Information Disclosure Statement is being filed after the events recited in Section 1.97(b) but, to the undersigned's knowledge, before the mailing date of either a Final action, Quayle action, or a Notice of Allowance. Under the provisions of 37 C.F.R. § 1.97(c), the Commissioner is hereby authorized to charge the fee of \$180.00 to Deposit Account No. 06-0916 for this Information Disclosure Statement.

Copies of the listed foreign and non-patent literature documents are attached. Copies of the U.S. patents and patent publications are not enclosed.

Applicants respectfully request that the Examiner consider the listed documents and indicate that they were considered by making appropriate notations on the attached form.


This submission does not represent that a search has been made or that no better art exists and does not constitute an admission that each or all of the listed documents are material or constitute "prior art." If the Examiner applies any of the documents as prior art against any claims in the application and Applicants determine that the cited documents do not constitute "prior art" under United States law, Applicants reserve the right to present to the office the relevant facts and law regarding the appropriate status of such documents.

Applicants further reserve the right to take appropriate action to establish the patentability of the disclosed invention over the listed documents, should one or more of the documents be applied against the claims of the present application.

If there is any fee due in connection with the filing of this Statement, please charge the fee to our Deposit Account No. 06-0916.

Respectfully submitted,

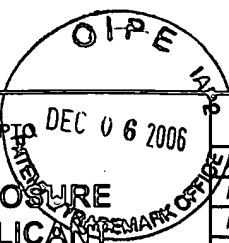
FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

By:  *Reg. No. 56,170*

Gary J. Edwards
Reg. No. 41,008

Date: December 6, 2006

**EXPRESS MAIL LABEL NO.
EV 746096154 US**



IDS Form PTO/SB/08: Substitute for form 1449A/PTO

Complete if Known

INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>		Application Number	10/954,182		
		Filing Date	October 1, 2004		
		First Named Inventor	ZHANG, Hongmei		
		Art Unit	2823		
		Examiner Name	ESTRADA, Michelle		
Sheet	1	of	3	Attorney Docket Number	9140.0016-01

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
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		Number-Kind Code ² (if known)	MM-DD-YYYY		
		US-6,088,492	07-11-2000	Kaneko et al.	
		US-6,288,835 B1	09-11-2001	Nilsson et al.	
		US-6,452,717 B1	09-17-2002	Endo	
		US-2002/0191916 A1	12-19-2002	Frish et al.	
		US-2003/0044118 A1	03-06-2003	Zhou et al.	
		US-2003/0143853 A1	07-31-2003	Celii et al.	

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

FOREIGN PATENT DOCUMENTS						
Examiner Initials	Cite No. ¹	Foreign Patent Document	Publication Date	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁹
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)	MM-DD-YYYY			
		KR 2002-26187	04-06-2002	Hyundai Motor Co Ltd		Abstract
		WO 01/82297 A1	11-01-2001	Koninklijke Philips Electronics N.V.		

NON PATENT LITERATURE DOCUMENTS			
Examiner Initials	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
		AGRAWAL, G.P., in: Fiber-Optic Communication Systems, 2nd Edition, John Wiley & Sons, New York, pp. 362-399 and 415 (1997).	
		MASUDA, H. & KAWAI, S., "Wide-band and gain-flattened hybrid fiber amplifier consisting of an EDFA and a multiwavelength pumped raman amplifier," IEEE Photonics Technology Lett. 11(6):647-649 (1999).	
		SNOEKS, E. et al., "Cooperative upconversion in erbium-implanted soda-lime silicate glass optical waveguides," J. Opt. Soc. Am. B 12(8):1468-1474 (1995).	
		Final Office Action dated October 12, 2006, in U.S. Application No. 10,291,179 (Attorney Docket No. 9140.0001-00).	
		Response to Final Office Action mailed November 3, 2006, in U.S. Application	

Examiner Signature	Date Considered	
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**EXPRESS MAIL LABEL NO.
EV 746096154 US**

IDS Form PTO/SB/08: Substitute for form 1449A/PTO				Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>				<i>Application Number</i>	10/954,182
				<i>Filing Date</i>	October 1, 2004
				<i>First Named Inventor</i>	ZHANG, Hongmei
				<i>Art Unit</i>	2823
				<i>Examiner Name</i>	ESTRADA, Michelle
Sheet	2	of	3	<i>Attorney Docket Number</i>	9140.0016-01

NON PATENT LITERATURE DOCUMENTS		
		No. 10,291,179 (Attorney Docket No. 9140.0001-00).
		Office Action dated December 1, 2006, in U.S. Application No. 10,291,179 (Attorney Docket No. 9140.0001-00).
		Notice of Allowance mailed March 25, 2004 for US Patent No. 6,827,826 (Atty. Docket No. 09140.0002-02).
		Notice of Allowance issued on October 8, 2002, in U.S. Patent No. 6,533,907 (Atty. Docket No. 09140-0004-00).
		Amendment dated October 19, 2006, in U.S. Application No. 09/903,081 (Atty. Docket No. 09140.0014-00).
		Notice of Allowance issued on October 21, 2004, in U.S. Application No. 10/101,492 (Atty. Docket No. 09140-0015-00).
		Response to Office Action filed September 11, 2006 in U.S. Application No. 11/100,856 (Atty. Docket No. 09140.0015-01.)
		Office Action mailed December 1, 2006, in U.S. Application No. 11/100,856 (Attorney Docket No. 09140.0015-01).
		Office Action mailed September 6, 2006, in U.S. Appl. No. 10/101,863 (Atty. Docket No. 09140.0016-00).
		Final Office Action mailed October 19, 2006, in U.S. Application No. 10/650,461 (Attorney Docket No. 09140.0025-00).
		Voluntary Amendment filed July 26, 2006 in TW Appl. No. 92123625 (Atty. Docket No. 09140.0025-00270).
		Response to Final Office Action filed August 3, 2006, in U.S. Application No. 10/789,953 (Atty. Docket No. 09140.0030-00).
		Notice of Allowance mailed October 23, 2006, in U.S. Application No. 10/789,953 (Atty. Docket No. 09140.0030-00).
		Office Action dated October 12, 2006, for U.S. Application No. 11/228,805 (Attorney Docket No. 09140.0030-01000).
		Office Action dated September 22, 2006 from Korean Patent Office in Appl. No. 10-2005-7016055 (Atty. Docket No. 09140.0030-00202)
		Response to Office Action mailed November 8, 2006, to the Korean Patent Office in Application No. 10-2005-7016055 (Attorney Docket No. 09140.0030-00202).
		Response to Office Action from Singapore Patent Office in Appl. No. 200505388-9, dated August 11, 2006 (Atty. Docket No. 9140.0030-00256).

Examiner Signature		Date Considered	
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				<i>Art Unit</i>	2823
				<i>Examiner Name</i>	ESTRADA, Michelle
Sheet	3	of	3	<i>Attorney Docket Number</i>	9140.0016-01

NON PATENT LITERATURE DOCUMENTS			
		Final Office Action dated October 26, 2006, in U.S. Application No. 10/851,542 (Atty. Docket No. 09140.0033-00).	
		Preliminary Amendment filed July 21, 2006, in U.S. Application No. 11/297,057 (Attorney Docket No. 9140.0042-00).	
		Supplemental Preliminary Amendment, Substitute Specification, and replacement drawing filed December 6, 2006, in U.S. Application No. 11/297,057 (Attorney Docket No. 9140.0042-00).	
		Voluntary Amendment filed August 15, 2006 in TW Appl. No. 94143175 (Atty. Docket No. 09140.0042-00270).	
		PCT International Search Report and Written Opinion for Application No. PCT/US05/44781 dated October 3, 2006 (Attorney Docket No. 9140.0042-00304).	

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Derwent WPI

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0014040325

WPI Acc no: 2004-222504/200421

Flywheel for radiating heat and increasing rigidity

Patent Assignee: HYUNDAI MOTOR CO LTD (HYUN-N)

Inventor: SONG J H

Patent Family: 2 patents, 1 countries

Patent Number	Kind	Date	Application Number	Kind	Date	Update	Type
KR 2003088236	A	20031119	KR 200226187	A	20020513	200421	B
KR 507142	B	20050809	KR 200226187	A	20020513	200662	E

Priority Applications (no., kind, date): KR 200226187 A 20020513

Patent Details

Patent Number	Kind	Lan	Pgs	Draw	Filing Notes	
KR 2003088236	A	KO	1	10		
KR 507142	B	KO			Previously issued patent	KR 2003088236

Alerting Abstract KR A

NOVELTY - A flywheel for radiating and reinforcing is provided to prevent crack by absorbing burst force with elastic force and radiating friction heat in operating the clutch because the flywheel is divided into plural portions and manufactured with different materials.

DESCRIPTION - A flywheel(10) is composed of a support plate(10a) combined with a crankshaft with pressing and fitting a ring gear(11); a cover plate(10b) fixed to the support plate and bent to form the storage space; an insert plate(10c) fastened inside the cover plate; and a friction plate(10d) contacted to the insert plate and inserted to the cover plate to receive clamping load from a clutch plate in operating the clutch. The durability is improved by preventing crack from burst force in the flywheel and radiating friction heat from clamping load.

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(19)대한민국특허청(KR)
(12) 등록특허공보(B1)

(51) . Int. Cl.⁷
F16F 15/30

(45) 공고일자 2005년08월09일
(11) 등록번호 10-0507142
(24) 등록일자 2005년08월01일

(21) 출원번호 10-2002-0026187 (65) 공개번호 10-2003-0088236
(22) 출원일자 2002년05월13일 (43) 공개일자 2003년11월19일

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심사관 : 방승훈

(54) 방열과 강성 강화용 플라이 휠

요약

본 발명은 방열과 강성 강화용 플라이 휠에 관한 것으로, 플라이휠을 서로 결합되는 여러 부분으로 분리해 제작하면서 그 재질을 달리해 버스트포스를 자체적인 탄성을 통해 흡수하면서 클러치의 조작에 따라 발생하는 마찰열의 방열성도 향상시켜 균열을 방지함에 그 목적이 있다.

상기와 같은 목적을 달성하기 위한 본 발명은, 플라이휠(10)이 링기어(11)가 외주 측에 압입되면서 크랭크축(5)에 결합되는 지지플레이트(10a)와, 이 지지플레이트(10a)에 일단이 고정되면서 절곡된 내부로 수용공간을 형성하는 커버플레이트(10b), 이 커버플레이트(10b)의 안쪽에서 고정되는 인서트플레이트(10c) 및 이 인서트플레이트(10c)에 접촉됨과 더불어 커버플레이트(10b)내에 삽입·고정되어 클러치 조작에 따라 클러치플레이트(22)로부터 클램핑 로드(F)를 받는 마찰플레이트(10d)로 이루어진 것을 특징으로 한다.

대표도

도 4

명세서

도면의 간단한 설명

- 도 1은 일반적인 엔진의 구성도
- 도 2는 종래에 따른 플라이 휠부위의 구성도
- 도 3은 종래에 따른 플라이휠의 구성 단면도

도 4는 본 발명에 따른 플라이휠의 구성 단면도

<도면의 주요부분에 대한 부호의 설명>

- 1 : 실린더 2 : 피스톤
- 3 : 커넥팅 로드 5 : 크랭크축
- 6 : 워터펌프 7 : 밸브기구
- 8 : 크랭크풀리 9 : 타이밍 기어
- 10 : 플라이휠 10a : 지지플레이트
- 10b : 커버플레이트 10c : 인서트플레이트
- 10d : 마찰플레이트 10d' : 슬랏
- 11 : 링기어 20 : 클러치어셈블리
- 21 : 클러치커버 22 : 클러치플레이트
- H : 휠하우징
- F : 클랭핑로드 K : 잭

발명의 상세한 설명

발명의 목적

발명이 속하는 기술 및 그 분야의 종래기술

본 발명은 방열과 강성 강화용 플라이 휠에 관한 것으로, 보다 상세하게는 엔진의 회전력에 의한 모서리부위의 균열을 방지하도록 강성을 강화함과 더불어 클러치와의 마찰에 의한 마찰열의 방열성도 향상할 수 있도록 된 방열과 강성 강화용 플라이휠에 관한 것이다.

일반적으로 엔진은 도 1에 도시된 바와 같이 실린더(1) 내에서 혼합 가스가 폭발하여 피스톤(2)이 왕복 운동되고, 상기 피스톤(2)과 커넥팅 로드(3)로 연결된 크랭크축(5)이 회전 운동되면서 회전 동력이 발생하게 된다.

또한, 상기 크랭크축(5)에는 그 선단측에 워터펌프(6) 및 밸브기구(7)를 작동하기 위한 크랭크풀리(8) 및 타이밍기어(9)가 설치되어 있고, 반대쪽에는 하우징(H)내로 수용되어 엔진의 초기 시동시에 스타트 모터의 피니언 기어와 맞물려 회전력을 전달받는 링기어(11)가 외주 측에 열 박음으로 압입되면서 클러치와 변속기에 회전 동력을 전달하는 플라이휠(10)이 설치되어 있다.

여기서, 상기 플라이휠(10)은 도 2에 도시된 바와 같이, 링기어(11)가 압입된 반대측으로 클러치페달에 의해 플라이휠(10)과 마찰되는 클러치플레이트(22)를 감싸는 클러치커버(21)로 이루어져 엔진과 변속기사이의 회전 전달을 분리·연결하는 클러치어셈블리(20)가 구비되어진다.

이러한 플라이휠(10)은 엔진의 4행정 중에서 폭발행정에서 얻어진 토크(Torque)를 흡입·압축·배기 행정에 고르게 분배하여 승차감 및 차량 출발성에 맞춘 형상으로 설계됨은 물론 엔진과 변속기를 연결하는 역할로 클러치의 용량에 맞게 설계됨은 물론이다.

그러나, 이와 같은 플라이휠(10)은 도 3에 도시된 바와 같이, 크랭크축(5)에 보울트등을 매개로 고정된 상태에서 엔진의 회전력을 변속기로 전달하기 위해 고속으로 회전하고, 또한 클러치의 조작에 따라 클러치플레이트(22)와 서로 강하게 마찰되어 엔진의 회전력을 단속하는데 이로 인해, 포트(Port)형 클러치인 경우에는 클러치플레이트(22)의 취부면과 플라이휠(10)좌면 사이의 노치(C : Notch)부위가 회전에 따른 버스트 포스(Burst Force)에 의해 균열(Crack)이 발생할 소지가 있는 문제가 있게 된다.

또한, 트럭과 버스와 같이 대형 디젤엔진을 사용하는 경우에는 플라이휠(10)의 형상도 이너시아(Inertia)값이 매우 큰 형상을 갖고 주철로 제조되는데 특히, 이와 같은 경우에는 빈번하면서 장시간동안 클러치를 조작하는 경우에는, 클러치에서 발생되는 약 250 - 300℃ 정도의 고온에 의해 플라이휠(10)의 열변형을 가져오고 이에 따른 강도 저하는 물론 균열을 발생시킬 수 있는 문제가 있게 된다.

발명이 이루고자 하는 기술적 과제

이에 본 발명은 상기와 같은 점을 감안하여 발명된 것으로, 플라이휠을 서로 결합되는 여러 부분으로 분리해 제작하면서 그 재질을 달리해 버스트포스를 자체적인 탄성을 통해 흡수하면서 클러치의 조작에 의한 마찰열의 방열성도 향상시켜 균열을 방지함에 그 목적이 있다.

상기와 같은 목적을 달성하기 위한 본 발명은, 플라이 휠이 초기 시동시 스타트 모터에 의해 회전되는 링기어를 외주면으로 압입 고정하면서 엔진의 초기 구동력 발생하는 크랭크축으로 링기어의 회전력을 전달하도록 크랭크축에 결합된 지지플레이트와, 상기 지지플레이트에 고정되면서 지지플레이트로부터 일정간격을 두도록 절곡되어 그 내부로 수용공간을 형성하는 커버플레이트, 상기 커버플레이트의 내부 수용공간인 안쪽에서 고정되는 인서트플레이트 및 상기 커버플레이트내에서 인서트플레이트에 접촉되도록 삽입·고정되어 클러치 조작 시 클러치어셈블리의 클러치플레이트에 접촉되면서 마찰력을 발생하는 마찰플레이트로 이루어진 것을 특징으로 한다.

발명의 구성 및 작용

이하 본 발명의 실시예를 첨부된 예시도면을 참조로 상세히 설명한다.

도 4는 본 발명에 따른 플라이휠의 구성 단면도를 도시한 것인바, 본 발명은 피스톤(2)의 왕복 운동을 회전운동으로 변환하는 크랭크축(5)에 결합되면서 클러치어셈블리(20)가 장착되는 플라이휠(10)이, 초기 시동시에 스타트 모터의 피니언 기어와 맞물려 회전력을 전달받는 링기어(11)가 외주 측에 열 박음으로 압입됨과 더불어 보울트등의 고정부재를 통해 크랭크축(5)에 결합되는 지지플레이트(10a)와, 이 지지플레이트(10a)에 보울트등의 고정부재를 통해 고정된 일단으로부터 서로 간격을 두도록 절곡되어 그 내부로 수용공간을 형성하는 컵형상의 커버플레이트(10b), 이 커버플레이트(10b)의 안쪽에서 보울트등의 고정부재를 통해 고정되는 인서트플레이트(10c) 및 이 인서트플레이트(10c)에 접촉됨과 더불어 커버플레이트(10b)내에 삽입·고정되어 클러치 조작 시 클러치어셈블리(20)의 클러치플레이트(22)와 접촉되어 클램핑로드(F')가 작용되는 마찰플레이트(10d)로 이루어진다.

여기서, 상기 지지플레이트(10a)와 인서트플레이트(10c)는 주철로 이루어지는 반면, 상기 커버플레이트(10b)는 강(Steel)재질로 이루어져 플라이휠(10)에 작용하는 버스트 포스(F)를 자체적인 탄성력을 통해 흡수·완화시켜주게 된다.

또한, 상기 인서트플레이트(10c)는 클러치 조작에 따라 마찰플레이트(10d)에서 발생되는 마찰열의 전달시 열팽창에 의해 커버플레이트(10b)와의 간섭을 방지하도록 약 1.00 - 1.50mm 정도의 갭(K)을 유지하게 된다.

그리고, 상기 마찰플레이트(10d)는 클러치 조작에 따른 클러치플레이트(22)와의 마찰열을 외부로 발산하도록 그 측면 테두리를 따라 다수의 슬랏(10d')이 형성되어진다.

이하 본 발명의 작동을 첨부된 도면을 참조로 상세히 설명한다.

본 발명은 플라이휠(10)이 일체로 이루어지는 대신 여러 부품들로 이루어지는데 즉, 지지플레이트(10a)에 초기 시동시에 스타트 모터의 피니언 기어와 맞물려 회전력을 전달받는 링기어(11)를 열 박음으로 압입시킨 상태에서 커버플레이트

(10b)의 안쪽으로 보울트등의 고정부재를 이용해 인서트플레이트(10c)와 마찰플레이트(10d) 순차적으로 고정한 후, 상기 커버플레이트(10b)와 지지플레이트(10a)를 보울트등의 고정부재를 매개로 결합시킨 상태에서 크랭크축(5)에 결합하여 플라이휠(10)을 조립하게 된다.

이때, 상기 지지플레이트(10a)와 크랭크축(5)사이에는 파이롯 베어링(Pilot Bearing)이 개재됨은 물론이다.

이어, 상기 플라이휠(10)에 클러치어셈블리(20)를 결합하여 클러치 조작시 클러치플레이트(22)에 의해 플라이휠(10)의 마찰플레이트(10d)쪽으로 강하게 작용하는 클램핑로드(F')를 통해 엔진과 변속기사이의 회전력을 단속하게 된다.

이때, 본 발명의 플라이휠(10)은 클러치의 조작시 엔진의 회전에 의해 플라이휠(10)에 발생하는 버스트 포스(F)가 강제 질로 이루어진 커버플레이트(10b)의 탄성력에 의해 흡수·완화되거나 또는 제거되고 이로 인해, 상기 커버플레이트(10b)의 모서리부위인 노치 부위로 집중되는 버스트 포스(F)에 의한 균열의 발생을 예방할 수 있게 된다.

또한, 본 발명의 플라이휠(10)은 클러치의 빈번한 조작에 의한 클램핑로드(F')에 의해 클러치플레이트(22)와 마찰플레이트(10d)사이에서 마찰열이 발생되면, 상기 마찰플레이트(10d)에 접촉된 인서트플레이트(10c)로 전달됨과 더불어 마찰플레이트(10d)의 측면 테두리를 따라 형성된 다수의 슬롯(10d')을 통해 보다 빠른 열의 방출이 이루어지게 된다.

이때, 상기 인서트플레이트(10c)로 전달된 마찰열이 방열되는 것보다 적층되는 것이 클 경우에는 인서트플레이트(10c)가 팽창하게 되지만, 이때 상기 인서트플레이트(10c)가 이를 수용한 커버플레이트(10b)내에서 약 1.00 - 1.50mm 정도의 갭(K)을 유지하므로 열팽창에 의한 커버플레이트(10b)와의 간섭을 방지할 수 있게 됨은 물론이다.

발명의 효과

이상 설명한 바와 같이 본 발명에 의하면, 플라이휠이 링기어가 외주 측에 압입되면서 크랭크축에 결합되는 부분과 클러치 조작에 따라 클러치플레이트로부터 클램핑 로드를 받는 부분으로 분리 제작한 후 서로 조립·결합시켜, 플라이휠이 적용되는 차종에 관계없이 플라이휠에서 발생하는 버스트포스에 의한 크랙과 클램핑로드에 따른 마찰열의 방열성을 향상시킬 수 있어 내구성을 강화할 수 있는 효과가 있게 된다.

(57) 청구의 범위

청구항 1.

초기 시동시 스타트 모터에 의해 회전되는 링기어(11)를 외주면으로 압입 고정하면서 엔진의 초기 구동력 발생하는 크랭크축(5)으로 링기어(11)의 회전력을 전달하도록 크랭크축(5)에 결합된 지지플레이트(10a)와,

상기 지지플레이트(10a)에 고정되면서 지지플레이트(10a)로부터 일정간격을 두도록 절곡되어 그 내부로 수용공간을 형성하는 커버플레이트(10b),

상기 커버플레이트(10b)의 내부 수용공간인 안쪽에서 고정되는 인서트플레이트(10c) 및

상기 커버플레이트(10b)내에서 인서트플레이트(10c)에 접촉되도록 삽입·고정되어 클러치 조작 시 클러치어셈블리(20)의 클러치플레이트(22)에 접촉되면서 마찰력을 발생하는 마찰플레이트(10d)로 이루어진 방열과 강성 강화용 플라이 휠.

청구항 2.

제 1항에 있어서, 상기 지지플레이트(10a)와 인서트플레이트(10c)는 주철로 이루어지고, 상기 커버플레이트(10b)는 탄성을 갖는 강재질로 이루어진 것을 특징으로 하는 방열과 강성 강화용 플라이 휠.

청구항 3.

제 1항에 있어서, 상기 인서트플레이트(10c)는 열팽창에 의해 커버플레이트(10b)와의 간섭을 방지하도록 1.00 - 1.50mm 정도의 갭(K)을 형성하는 것을 특징으로 하는 방열과 강성 강화용 플라이 휠.

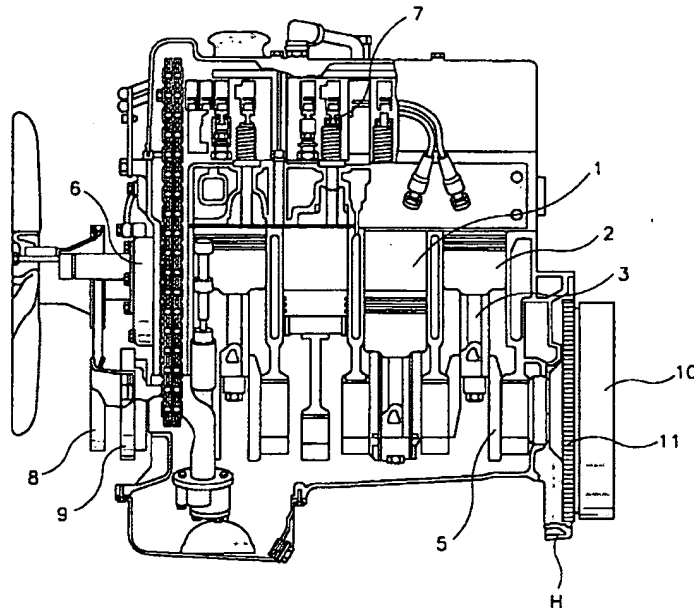
청구항 4.
삭제

청구항 5.

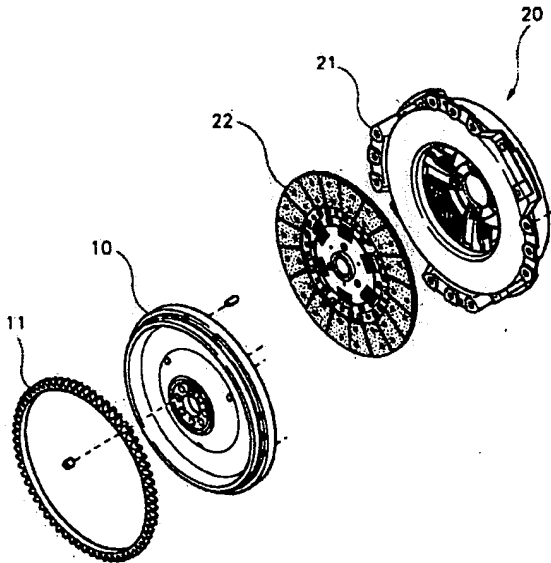
제 1항에 있어서, 상기 마찰플레이트(10d)는 클러치 조작에 따른 클러치플레이트(22)와의 마찰열을 외부로 발산하도록 그 측면 테두리를 따라 다수의 슬롯(10d')이 형성되어진 것을 특징으로 하는 방열과 강성 강화용 플라이 휠.

도면

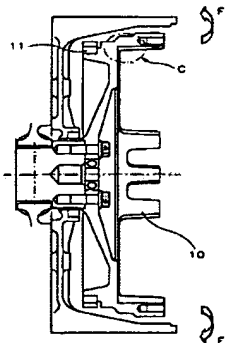
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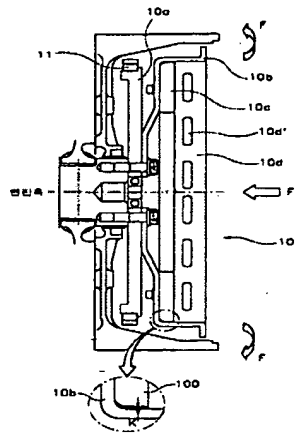
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도면4



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(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
1 November 2001 (01.11.2001)

PCT

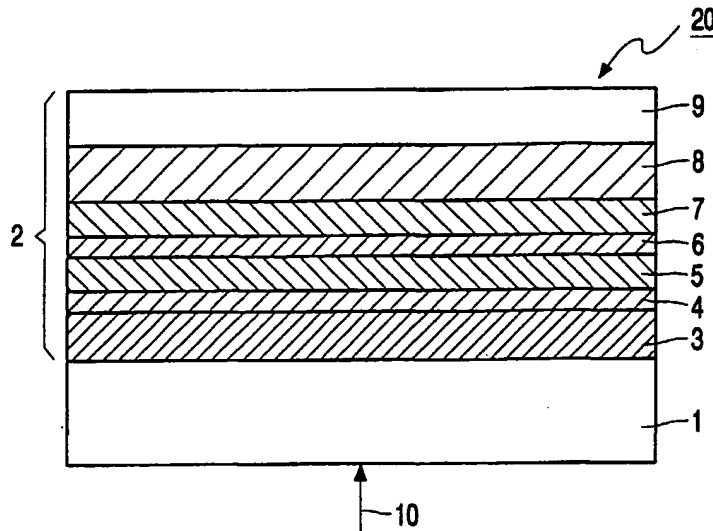
(10) International Publication Number
WO 01/82297 A1

- (51) International Patent Classification⁷: G11B 7/24 (74) Agent: DEGUELLE, Wilhelmus, H., G.; Internationaal Octrooibureau B.V., Prof. Holstlaan 6, NL-5656 AA Eindhoven (NL).
- (21) International Application Number: PCT/EP01/04028
- (22) International Filing Date: 9 April 2001 (09.04.2001) (81) Designated States (national): CA, CN, JP, KR, MX, RU.
- (25) Filing Language: English (84) Designated States (regional): European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LÜ, MC, NL, PT, SE, TR).
- (26) Publication Language: English
- (30) Priority Data: 00201488.4 20 April 2000 (20.04.2000) EP
Published:
— with international search report
- (71) Applicant: KONINKLIJKE PHILIPS ELECTRONICS N.V. [NL/NL]; Groenewoudseweg 1, NL-5621 BA Eindhoven (NL).
For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.
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WO 01/82297 A1

(54) Title: OPTICAL RECORDING MEDIUM AND USE OF SUCH OPTICAL RECORDING MEDIUM



(57) Abstract: The optical recording medium (20) has a substrate (1) and a stack (2) of layers provided thereon. A phase change recording layer (5), having a melting point T_{mp} is sandwiched between a first (3) and a second (7) dielectric layer. A crystallization-accelerating layer (4, 6) is being interposed in contact with the recording layer (5). Further a reflective layer (8) is present and an optional cover layer (9). The crystallization-accelerating layer (4, 6) consists of a binary metal alloy or a semiconductor and has a melting point T_{mg} at least 250 °C higher than the melting point T_{mp} of the recording layer (5) and has a crystal structure similar to the crystalline state of the recording layer (5).

Optical recording medium and use of such optical recording medium

The invention relates to an optical recording medium having a substrate and a stack of layers provided thereon, the stack comprising a recording layer, having a melting point T_{mp} and being able to change between an amorphous and a crystalline state, sandwiched between a first and a second dielectric layer, the first being adjacent to the substrate, a
5 crystallization accelerating layer being interposed in contact with the recording layer, and a reflective layer.

The invention also relates to the use of such an optical recording medium.

10 An optical recording medium of the type mentioned in the opening paragraph is known from Japanese patent application JP-09161316 A. In the known medium the state of the recording layer locally changes from crystalline to amorphous when data are optically recorded.

Optical data storage based on the phase change principle is attractive, because
15 it combines the possibilities of direct overwrite (DOW) and high storage density with easy compatibility with read-only optical data storage systems. Phase-change optical recording involves the formation of submicrometer-sized amorphous recording marks in a crystalline film using a focused relatively high power laser-light beam. During recording information, the medium is moved with respect to the focused laser-light beam that is modulated in
20 accordance with the information to be recorded. Due to this, quenching takes place in the phase-change recording layer and causes the formation of amorphous information bits in the exposed areas of the recording layer that remains crystalline in the unexposed areas. Erasure of written amorphous marks is realized by recrystallizing through heating with the same laser at an intermediate power level, without melting the recording layer. The amorphous marks
25 represent the data bits, which can be read, e.g. via the substrate, by a low-power focused laser-light beam. Reflection differences of the amorphous marks with respect to the crystalline recording layer bring about a modulated laser-light beam which is subsequently converted by a detector into a modulated photocurrent in accordance with the recorded digital information.

One of the most important demands in phase-change optical recording is a high data rate, which means that data can be written in and read from the medium with a rate of at least 30Mbits/s. A high data rate requires the recording layer to have a high crystallization rate, i.e. a short crystallization time. To ensure that the previously recorded amorphous marks can be crystallized during direct overwrite, the recording layer should have a proper crystallization time to match the velocity of the medium relative to the laser-light beam. If the crystallization speed is not high enough to match the velocity of the medium relative to the laser-light beam the amorphous marks from the previous recording, representing old data, cannot be completely erased, meaning recrystallized, during DOW. This causes a high noise level. A high crystallization speed is particularly required in high-density recording and high data rate applications, such as disc-shaped DVD+RW, DVR-red and blue which are abbreviations of new generation high density Digital Versatile Disc+RW, where RW refers to the rewritability of such discs, and Digital Video Recording optical storage discs, where red and blue refer to the used laser wavelength. For these new discs the complete erasure time (CET) has to be at most 60 ns. CET is defined as the minimum duration of the erasing pulse for complete crystallization of a written amorphous mark in a crystalline environment, which is measured statically. For DVD+RW, which has a 4.7 GB recording density per 120 mm disk, a user data bit rate of 33 Mbits/s is needed, and for DVR-red said rate is 35 Mbits/s. For rewritable phase change optical recording systems such as DVR-blue, a user data rate higher than 50 Mbits/s is required.

The known medium of the phase-change type comprises a disc-shaped substrate carrying a stack of layers consisting, in succession, of a first dielectric layer, a Sb_2Te_3 layer as crystallization accelerating layer, a Sb layer as a composition correcting layer, a recording layer of a phase-change $Sb_{72}Te_{28}$ alloy, a second dielectric layer and a metal reflective layer. Such a stack of layers can be referred to as an INP'PIM structure, wherein M represents a reflective or mirror layer, I represents a dielectric layer and P represents a phase-change recording layer while P' represents a composition correction layer which mixes with the recording layer at first recording. A crystallization accelerating layer N of Sb_2Te_3 , has been arranged between the first dielectric layer and the correction and the recording layer to achieve a fast crystallization of the medium during erasing information in the medium by means of a laser-light beam. In the known recording medium the N layer has a melting point of 618°C, only 68°C higher than the melting point 550°C of the P layer. The melting point of the known N layer is relatively close to the melting point of the P layer causing the N layer to dissolve in the correction and recording layer P'P after one or at best a few recording/erasure

cycles, whereafter the crystallization accelerating layer N is no longer present and its crystallization-accelerating action is lost.

For complete erasure of an amorphous mark, two processes occur, i.e. nucleation and grain (crystallite) growth. An investigation of the known recording medium has revealed that the known crystallization-accelerating layer N is merely a nucleation-promoting layer.

It is a disadvantage of the known medium that its crystallization accelerating layer only functions for at most a few recording and erasing cycles. This is not sufficient for modern erasable media, which require a stable performance for at least a thousand of recording and erasing cycles.

It is an object of the invention to provide an optical recording medium of the kind described in the opening paragraph, which is suitable for high speed rewritable optical recording, having a CET-value of at most 60 ns.

It is another object of the invention to provide an optical recording medium of the kind described in the opening paragraph, which is suitable for rewritable optical recording, having a stable performance for at least 10^3 recording and erasing cycles.

This object is achieved in accordance with the invention by an optical recording medium as described in the opening paragraph, which is characterized in that the crystallization accelerating layer

- comprises a material selected from the group consisting of binary metal alloys, semiconductor elements and semiconductor alloys and
- has a melting point T_{mg} at least 250°C higher than the melting point T_{mp} of the recording layer and
- has a crystal structure similar to the crystalline state of the recording layer.

The crystallization accelerating layer according to the invention, which will also be abbreviated as G, yields a high crystallization speed of the recording layer because the amorphous marks of the recording layer are in contact with the G-layer. This accelerates the crystallite growth process, leading to a higher crystallization speed. Especially because the crystal structure of the G-layer is similar to, or even the same as, the structure of the crystalline state of the recording layer the crystallization rate of amorphous marks is advantageously increased. The crystal structure of the G-layer then serves as a very good grain growth initialization or nucleation layer for crystallite growth in the recording layer.

The G-layer is always present adjacent to a thermally isolating layer, here the first or second dielectric layer, because a stack having a G-layer between the recording layer and the substrate or the reflection layer cannot realize the desired thermal properties.

An advantage of the G-layer, comprising a material selected from the group
5 consisting of binary metal alloys, semiconductors and semiconductor alloys, is that it has a high melting point. This counteracts dissolving of the G-layer in the recording layer and maintenance of the crystalline structure for a large number of recording and erasing cycles. During recording, the maximum temperature in the recording layer is about 800°C, which is about 1.4 times T_{mp} for a recording layer with a T_{mp} of 550°C. This may be deduced from a
10 temperature calculation based on the presented laser energy during recording and the physical properties of the stack. The melting temperature T_{mg} of the G-layer has to be above this maximum temperature so that the G-layer remains in crystalline state when the recording layer is melted.

Therefore, the melting temperature difference between recording layer and G-
15 layer should be 250°C or larger, but preferably 300°C or larger, taking into consideration a safety margin.

Preferred materials, which may be used as G-layer, are PbTe, Ag₂Te, CrTe, Ge and Si.

In an embodiment of the recording medium the G-layer is arranged between
20 the recording layer and the second dielectric layer. The thickness of the G-layer may be chosen between 0.1 and 10 nm. The thermal conductivity of the crystallization-accelerating layer is generally comparable to that of the recording layer, which is an alloy of metals. However this only has a small effect on the thermal behaviour of the stack because the thickness of the G-layer is generally relatively small compared to the other layers in the
25 stack. This facilitates the thermal design of the stack.

In another embodiment the crystallization time is reduced further in that a second G-layer is arranged between the recording layer and the first dielectric layer. Thus a G-layer is arranged on both sides of the recording layer. The second G-layer may be of a material similar or identical to the material of the other G-layer. The crystallization time is
30 reduced because now a crystalline layer, which accelerates the crystallite growth process, is present against the recorded amorphous mark on both sides. The thicknesses of the G-layers are between 0.1 and 10nm, preferably lower than 5 nm.

In a specific embodiment the two G-layers present on either side of the recording layer are substantially equal both in thickness and in composition. Equal in

thickness means to within 10% of each other. The equality of the thicknesses is advantageous in the manufacturing of the medium. In general the stack is deposited by evaporation or by sputtering in a vacuum chamber, where substrates move stepwise along a series of stations having targets of different compositions. The dwell time at each station is about equal, and the thickness of the layer deposited at a station is determined in part by switching the deposition process on and off. Consequently, the deposition of a relatively thin layer may require less time than available at a station, whereas the deposition of a relatively thick layer may even require two adjacent stations having the same target. It is therefore advantageous to choose to replace a relatively thick layer and a relatively thin layer by two layers of about equal thickness and composition, thereby reducing the number of deposition stations and the manufacturing time of a stack.

In a specific embodiment the recording layer comprises an alloy of Q, In, Sb and Te, wherein Q is selected from the group consisting of Ag and Ge.

The preferred composition comprises $Q_a In_b Sb_c Te_d$ (in atomic percentages), wherein Q is selected from the group consisting of Ag and Ge;

$$2 \leq a \leq 9$$

$$0 < b \leq 6$$

$$55 \leq c \leq 80$$

$$16 \leq d \leq 30; a + b + c + d = 100.$$

In another specific embodiment the recording layer comprises a compound of Ge, Sb and Te. The preferred composition of this compound is defined by the formula

$$Ge_{50x}Sb_{40-40x}Te_{60-10x} \text{ (in atomic percentages), wherein } 0.166 \leq x \leq 0.444;$$

the recording layer having a thickness of 5 to 35 nm;

This composition exists on a part of the line connecting the compounds GeTe and Sb_2Te_3 in the triangular Ge-Sb-Te composition diagram and includes the stoichiometric compounds $Ge_2Sb_2Te_5$ ($x = 4/9$), $GeSb_2Te_4$ ($x = 2/7$) and $GeSb_4Te_7$ ($x = 1/6$). Especially these ternary stoichiometric compounds are preferred, because these materials crystallize rapidly since no segregation is required during crystallization.

The first and second dielectric layers are preferably made of a mixture of ZnS and SiO_2 , e.g. $(ZnS)_{80}(SiO_2)_{20}$. The layers may alternatively be made of SiO_2 , TiO_2 , Ta_2O_5 , ZnS, AlN and/or Si_3N_4 . The dielectric layer through which the laser light enters the stack preferably has a thickness of 70 to $(70 + \lambda/2n)$ nm wherein n is the refractive index of the first dielectric layer and λ is the wavelength of the read/write laser-light beam. If the total

thickness is smaller than 70 nm, the cyclability is reduced considerably. The cyclability is measured by the relative change of the optical contrast M_0 after a large number of DOW-cycles, e.g. 10^3 . The optical contrast M_0 is defined as $|R_C - R_A|/R_C$, where R_C and R_A are the reflections of the recording material in the crystalline and amorphous state respectively.

- 5 Another way to define cyclability is related to jitter increase of the medium. Jitter is a measure of the distortion of the shape of a recording mark, and is measured as a time shift of rising and falling edges in the information signal. The jitter of the medium should be at a low, constant level during at least 10^3 DOW-cycles.

As mentioned above the total thickness of the first dielectric layer is preferably
10 smaller than $(70 + \lambda/2n)$ nm. A larger total thickness does not further increase the cyclability and is more expensive to make. If for example the wavelength is equal to 630 nm and the refractive index is 1.5, the thickness range extends from 70 nm to 280 nm.

The dielectric layer, which is closest to the reflective layer, has a thickness of
15 10 to 40 nm. Preferably the thickness of the dielectric layer adjacent to the reflective layer is larger than or equal to 15 nm. A smaller thickness results in an increased cooling rate of the recording layer and, consequently, an undesirable increase in the write power. The thickness is preferably smaller than 40 nm. A larger thickness decreases the thermal contact between the recording layer and the reflective layer too much, resulting in too low a cooling rate of the recording layer and a worse recording performance.

20 The reflective layer may comprise metals such as Al, Ti, Au, Ni, Cu, Ag and Cr, and alloys of these metals. The reflective layer preferably has a thickness of 60 to 120 nm.

Both the reflective layers and the dielectric layers generally have been provided by vapour deposition or sputtering.

25 Optionally an outermost layer may be present on the stack as a cover layer that protects the underlying layers from the environment. The cover layer is made of, for example, an UV light-cured poly(meth)acrylate.

Another specific embodiment is characterized in that the reflective layer is present between the substrate and the first dielectric layer. Optionally a cover layer, that is
30 transparent for laser-light and has a surface which allows optical recording of information into and reading of information from the underlying recording layer with a focused laser-light beam is present on top of the stack. Thus in this embodiment the optical recording medium is written in and read out through the cover layer. This method is used in the new DVR discs that were mentioned above. The cover layer of a DVR disc has a thickness of about 100

micrometers. This cover layer allows the use, in optical disc recorders, of a read/write lens with a high numerical aperture that is necessary for high density recording and reading. Because the laser light enters the medium through the cover layer it may be necessary to adjust the thicknesses of the layers of the stack in order to optimize for optimal optical contrast between recorded and unrecorded areas.

The term high-speed recording, which was mentioned above, is to be understood to mean in this context a linear velocity of the medium relative to the laser-light beam of at least 7.2 m/s, which is six times the speed according to the Compact Disc standard. The use of an optical recording medium according to the invention is therefore advantageous because the crystallization rate is fast enough to permit at least this recording velocity. The important parameter is the CET (in ns), which is defined above. The CET is inversely proportional to the crystallization rate.

The substrate of the information medium generally is transparent to the laser wavelength, and is made, for example, of polycarbonate, polymethyl methacrylate (PMMA), amorphous polyolefin or glass. In a typical example, the substrate is disc-shaped and has a diameter of 120 mm and a thickness of 1.2 mm, 0.6 mm or 0.1 mm for respectively low, medium and high information density applications.

Alternatively, the substrate may be in the form of a synthetic resin flexible tape, made e.g. from a polyester film. This flexible tape, with a stack of layers deposited thereon, is called an optical tape and can be suited for use in an optical tape recorder, which is for example based on a fast spinning polygon. In such a device the reflected laser-light beam scans transversely across the tape surface.

The surface of the disc-shaped substrate on the side of the recording layer is, preferably, provided with a servotrack that can be scanned optically. This servotrack is often constituted by a spiral-shaped groove and is formed in the substrate by means of a mould during injection moulding or pressing. This groove can be alternatively formed in a replication process in a synthetic resin layer, for example, of an UV light-cured layer of acrylate, which is separately provided on the substrate. In high-density recording such a groove has a pitch e.g. of 0.5 - 0.8 μm and a width of about half the pitch.

High-density recording and erasing can be achieved by using a short-wavelength laser, e.g. with a wavelength of 675 nm or shorter (red to blue).

The phase change recording layer as well as the G-layer can be applied by vapour depositing or sputtering of a suitable target. The recording layer thus deposited is amorphous and exhibits a low reflection. In order to constitute a suitable recording layer

having a high reflection, this layer must first be completely crystallized, which is commonly referred to as initialization. For this purpose, the recording layer can be heated in a furnace to a temperature just above the crystallization temperature of the e.g. Ge-In-Sb-Te or Ge-Sb-Te compound, e.g. 200°C. A synthetic resin substrate, such as polycarbonate, to which a high
5 temperature may cause damage, can alternatively be heated by a laser-light beam of sufficient power. This can be realized, e.g. in a recorder, in which case a laser beam scans the moving recording layer. The amorphous layer is then locally heated to the temperature required for crystallizing the layer, without the substrate being subjected to a disadvantageous heat load.

If desired, an additional, optically transparent, metal layer M' can be
10 interposed in the stack, thereby forming a so called MIRIM'-structure, wherein R represents a layer stack comprising a recording layer and at least one crystallization accelerating layer according to the present invention. Although the structure becomes more complicated, the additional metal layer increases the cooling rate of the recording layer as well as the optical contrast M_0 .

15 Embodiments of the optical recording medium of the invention will be described with reference to the drawings.

In the drawings:

20 Fig. 1 shows a schematic cross sectional view of a first embodiment of the optical recording medium.

Fig. 2. shows a view as shown in FIG.1 of a second embodiment.

Fig. 3 shows a view as in FIG.2 of a third embodiment.

25

In Fig. 1 the optical recording medium 20 has a substrate 1 and a stack 2 of layers provided thereon. The substrate 1 may be made of, for example, a sheet of plastic, e.g. polycarbonate, or glass. In Fig. 1 the stack 2 comprises a phase change recording layer 5, having a melting point T_{mp} and being able to change between an amorphous and a crystalline
30 state, that is sandwiched between a first 3 and a second 7 dielectric layer, the first 3 being adjacent to the substrate 1. In this embodiment both the first dielectric layer 3 and the second dielectric layer 7 are made of the material $(ZnS)_{80}(SiO_2)_{20}$ and have a thickness of 125 nm and 20 nm respectively. A crystallization accelerating layer 6, abbreviated as G-layer, is interposed in contact with the recording layer 5, which comprises an alloy of Q, In, Sb and

Te, wherein Q is selected from the group consisting of Ag and Ge. A reflective layer 8 is present on top of the stack 2. Reflective layer 8 is a 100 nm layer of Al or an aluminium alloy, e.g. AlCr or AlTi. The crystallization accelerating layer 6 comprises a binary metal alloy or a semiconductor element or semiconductor alloy and has a melting point T_{mg} at least
5 250°C higher than the melting point T_{mp} of the recording layer 5. The crystal structure of these materials is similar to the crystalline state of the recording layer 5. In this embodiment the recording layer 5 is made of $Ge_{6.2}In_{3.2}Sb_{71.1}Te_{19.6}$, which has a thickness of 12 nm. The embodiment shown has a cover layer 9 that may be made of an organic material, e.g. a UV-cured resin. A focused laser-light beam with a wavelength $\lambda=405$ nm enters the medium
10 through the substrate 1. This beam is diagrammatically illustrated by means of an arrow 10 in Fig. 1.

In this embodiment, when using PbTe as G-layer, which has a thickness of 3 nm, the CET has been measured to be equal to 40 ns, which is sufficiently short to allow high-speed recording. When no G-layer is present a minimal CET value of 48 ns can be
15 obtained. Other preferred materials as G-layer are Ag_2Te , CrTe, Ge or Si. The melting points T_{mg} of bulk PbTe, Ag_2Te , CrTe, Ge and Si are 914, 960, 1292, 936 and 1414°C respectively. The write power for the medium is relatively low and is 9 mW at the entrance face of the medium at a relative speed between the radiation beam and the medium of 7.2 m/s. The R_A and R_C are measured to be 4.3% and 23% respectively.

20 The cyclability is measured as the number of rewrite cycles where the jitter has increased to 12% of the clock time T_C . The jitter is the standard deviation of the difference between the rising and falling edges in the information signal and the data clock recovered from the information signal. As an example, for a standard CD format written with the so-called EFM code at the CD speed of 1.2 m/s and clock time of 230 ns, the jitter should
25 be lower than 28 ns. The number of overwrite cycles before deterioration of the medium becomes noticeable, e.g. the jitter has increased to 12% of the clock time, is larger than 10^3 . The jitter of a pattern read from the medium as a function of the overwrite cycle does not show a large overshoot.

During writing, the recording layer 5 of $Ge_{6.2}In_{3.2}Sb_{71.1}Te_{19.6}$ is heated to a
30 temperature of about 750°C, well above its melting temperature, which is about 550°C. The temperature during recording is below the melting temperature of the G-layer 6 comprising PbTe. The high melting temperature of the material used for the G-layer neighbouring the recording layer 5 therefore results in an increased cyclability of the recording medium.

In application JP-09161316 A the crystallization accelerating layer N of Sb_2Te_3 , which has a melting point of 618°C , is thus heated above its melting temperature, causing the atoms in the layer to become mobile. These atoms are then able to diffuse into the recording layer. The properties of the recording layer are affected by the influx of foreign
5 atoms, resulting in a deterioration of the recording process.

In Fig. 2 and Fig. 3 corresponding reference numerals denote the same layers as in Fig. 1.

In Fig. 2 a second crystallization accelerating layer 4 similar to the crystallization accelerating layer 6 is arranged between the recording layer 5 and the first
10 dielectric layer 3. Now G-layers 4, 6 are present on both sides adjacent to the recording layer 5. The G-layer 6 is made of PbTe and has a thickness of 1.5 nm. The second G-layer 4 is substantially equal both in thickness and in composition to the G-layer 6. The recording layer 5 has a thickness of 10 nm. Further the characteristics of the stack 2 are the same as in Fig. 1. The CET is measured to be 36 ns. The CET in this embodiment is smaller than in the
15 embodiment with only one G-layer. The R_A and R_C are measured to be 4.6% and 22% respectively.

In Fig. 3 the reflective layer 8 is present between the substrate 1 and the first dielectric layer 3. In this embodiment the laser light 10 is entering the stack 2 through the cover layer 9 which has a thickness of 100 μm . The cover layer 9 has a uniform thickness,
20 thereby improving the optical read and write performance in underlying recording layers when the read or write laser beam passes through said cover layer 9. For example a 100 μm cover layer 9 is used for the new 60 mm radius Digital Video Recording (DVR) disc. This disc is recorded in and read out through this cover layer 9, which therefore has to be of good optical quality. Preferably, the cover layer 9 is $100 \pm 3 \mu\text{m}$ thick up to radius 58.5 mm. The
25 cover layer 9 is made from a UV-cured resin. Dielectric layer 3 and 7 have a thickness of 20 nm and 125 nm respectively and are made of the same dielectric material as in Fig. 1. G-layer 4, 6 are made of the same material as in Fig. 2 and both have a thickness of 1.5 nm. Recording layer 5 has a thickness of 10 nm. For characteristics that are not specifically mentioned reference is made to the description of Fig. 1.

30 Preferably, for all embodiments, the surface of the disc-shaped substrate 1 on the side of the stack 2 is provided with a servotrack that can be scanned optically. This servotrack is often constituted by a spiral-shaped groove and is formed in the substrate by means of a mould during injection moulding or pressing. This groove can be alternatively formed in a replication process in a synthetic resin layer, for example, of an UV light-cured

layer of acrylate, which is separately provided on the substrate 1. In high-density recording such a groove has a pitch e.g. of 0.5 - 0.8 μm and a width of about half the pitch.

In a modification of the recording medium of Fig. 3, the recording layer 5 comprises an alloy of Ge, Sb and Te, e.g. $\text{Ge}_2\text{Sb}_2\text{Te}_5$.

5 It should be noted that the above-mentioned embodiments illustrate rather than limit the invention, and that those skilled in the art will be able to design many alternative embodiments without departing from the scope of the appended claims. In the claims, any reference signs placed between parentheses shall not be construed as limiting the claim. The word "comprising" does not exclude the presence of elements or steps other than those listed
10 in a claim. The word "a" or "an" preceding an element does not exclude the presence of a plurality of such elements. The mere fact that certain measures are recited in mutually different dependent claims does not indicate that a combination of these measures cannot be used to advantage.

According to the invention an optical recording medium is provided, which is
15 suitable for high speed recording, e.g. with a possible data rate higher than 50 Mbits/s, and which is suitable for direct overwrite for a least 10^3 times.

CLAIMS:

1. An optical recording medium (20) having a substrate (1) and a stack (2) of layers provided thereon, the stack comprising a recording layer (5), having a melting point T_{mp} and being able to change between an amorphous and a crystalline state, sandwiched between a first (3) and a second (7) dielectric layer, the first (1) being adjacent to the substrate (1), a crystallization accelerating layer (4, 6) being interposed in contact with the recording layer (5), and a reflective layer (8), characterized in that the crystallization accelerating layer (4, 6)
- 5
- comprises a material selected from the group consisting of binary metal alloys, semiconductors elements and semiconductor alloys and
 - 10 - has a melting point T_{mg} at least 250°C higher than the melting point T_{mp} of the recording layer (5) and
 - has a crystal structure similar to the crystalline state of the recording layer (5).
2. An optical recording medium (20) as claimed in Claim 1 characterized in that
- 15 the crystallization accelerating layer (4, 6) comprises a material selected from the group consisting of PbTe, Ag₂Te, CrTe, Ge and Si.
3. An optical recording medium (20) as claimed in any of Claims 1 or 2, characterized in that the crystallization accelerating layer (6) is arranged between the
- 20 recording layer (5) and the second dielectric layer (7).
4. An optical recording medium (20) as claimed in Claim 3, characterized in that a second crystallization accelerating layer (4) similar to the crystallization accelerating layer (6) is arranged between the recording layer (5) and the first dielectric layer (3).
- 25
5. An optical recording medium (20) as claimed in Claim 4, characterized in that the second crystallization accelerating layer (4) is substantially equal both in thickness and in composition to the crystallization accelerating layer (6).

6. An optical recording medium (20) as claimed in Claim 1, characterized in that the recording layer (5) comprises an alloy of Q, In, Sb and Te, wherein Q is selected from the group consisting of Ag and Ge
- 5 7. An optical recording medium (20) as claimed in Claim 1, characterized in that the recording layer (5) comprises an alloy of Ge, Sb and Te.
8. An optical recording medium (20) as claimed in Claim 1, characterized in that the reflective layer (8) is present between the substrate (1) and the first dielectric layer (3).
- 10 9. The use of an optical recording medium (20), which medium is claimed in any one of the preceding claims, characterized in that the linear velocity of the medium relative to a laser-light beam (10) is at least 7.2 m/s.

1/1

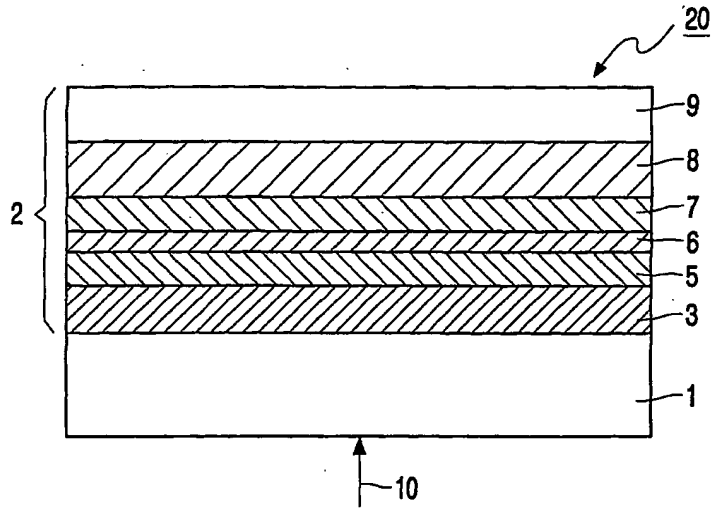


FIG. 1

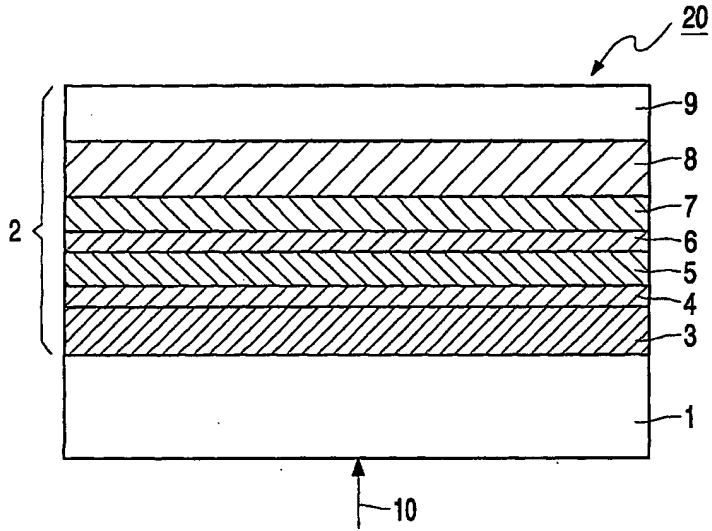


FIG. 2

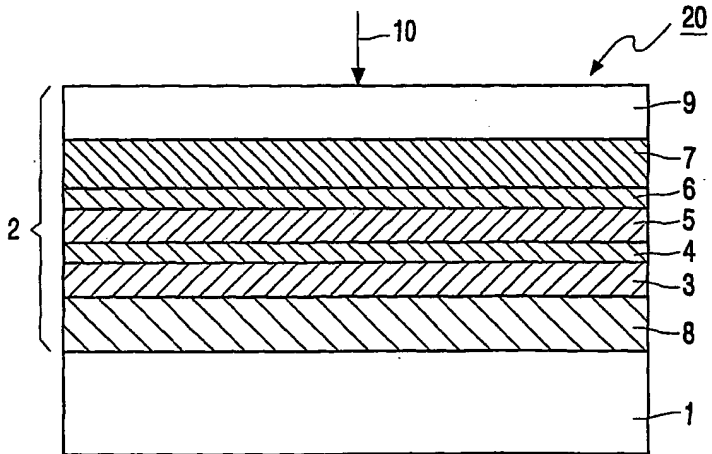


FIG. 3

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 01/04028

A. CLASSIFICATION OF SUBJECT MATTER IPC 7 G11B7/24		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols) IPC 7 G11B		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the International search (name of data base and, where practical, search terms used) EPO-Internal, PAJ, WPI Data		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 980 068 A (ASAHI CHEMICAL IND) 16 February 2000 (2000-02-16) abstract paragraph '0012! - paragraph '0019! paragraph '0038!; figure 1	1,2,7
Y	---	8
A	---	3,4,9
Y	EP 0 431 489 A (HITACHI LTD) 12 June 1991 (1991-06-12) column 16, line 28 - line 56; figure 18 --- -/-	8
<input checked="" type="checkbox"/> Further documents are listed in the continuation of box C. <input checked="" type="checkbox"/> Patent family members are listed in annex.		
* Special categories of cited documents :		
A document defining the general state of the art which is not considered to be of particular relevance *E* earlier document but published on or after the international filing date *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) *O* document referring to an oral disclosure, use, exhibition or other means *P* document published prior to the international filing date but later than the priority date claimed *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. *8* document member of the same patent family		
Date of the actual completion of the international search 8 August 2001		Date of mailing of the international search report 16/08/2001
Name and mailing address of the ISA European Patent Office, P.B. 5616 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016		Authorized officer Annibal, P

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 01/04028

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 945 860 A (MATSUSHITA ELECTRIC IND CO LTD) 29 September 1999 (1999-09-29) abstract paragraph '0017! paragraph '0018! paragraph '0034! paragraph '0035! paragraph '0050! paragraph '0084!; figures 1,2	1,3-7,9
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X	EP 0 843 874 A (PHILIPS ELECTRONICS NV) 27 May 1998 (1998-05-27) column 25, line 10 -column 26, line 38; examples 10,11	1,7

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/EP 01/04028

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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PATENT APPLICATION FEE DETERMINATION RECORD
Effective December 8, 2004

10/954182

CLAIMS AS FILED - PART I

	(Column 1)	(Column 2)
TOTAL CLAIMS		
FOR	NUMBER FILED	(NUMBER EXTRA
TOTAL CHARGEABLE CLAIMS	minus 20 =	
INDEPENDENT CLAIMS	minus 3 =	
MULTIPLE DEPENDENT CLAIM PRESENT <input type="checkbox"/>		

SMALL ENTITY TYPE

OR OTHER THAN SMALL ENTITY

RATE	FEE	OR	RATE	FEE
BASIC FEE	150.00	OR	BASIC FEE	300.00
X\$ 25=		OR	X\$50=	
X100=		OR	X200=	
+180=		OR	+360=	
TOTAL		OR	TOTAL	

* If the difference in column 1 is less than zero, enter "0" in column 2

CLAIMS AS AMENDED - PART II

	(Column 1)	(Column 2)	(Column 3)
AMENDMENT A	12-2-05	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR
Total	31	minus	61
Independent	4	minus	9
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

SMALL ENTITY OR OTHER THAN SMALL ENTITY

RATE	ADDITIONAL FEE	OR	RATE	ADDITIONAL FEE
X\$ 25=	/	OR	X\$50=	/
X100=	/	OR	X200=	/
+180=	/	OR	+360=	/
TOTAL ADDIT. FEE		OR	TOTAL ADDIT. FEE	

	(Column 1)	(Column 2)	(Column 3)
AMENDMENT B	8/9/06	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR
Total	57	minus	61
Independent	5	minus	9
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

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X100=	/	OR	X200=	/
+180=	/	OR	+360=	/
TOTAL ADDIT. FEE		OR	TOTAL ADDIT. FEE	

	(Column 1)	(Column 2)	(Column 3)
AMENDMENT C	12/06/06	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR
Total	37	minus	61
Independent	2	minus	9
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

RATE	ADDITIONAL FEE	OR	RATE	ADDITIONAL FEE
X\$ 25=	/	OR	X\$50=	/
X100=	/	OR	X200=	/
+180=	/	OR	+360=	/



03-05-07

Handwritten initials and a signature.

PATENT
Customer No. 22,852
Attorney Docket No. 9140.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)
)
 ZHANG, Hongmei et al.) Group Art Unit: 2823
)
 Application No.: 10/954,182) Examiner: ESTRADA, Michelle
)
 Filed: October 1, 2004)
)
 For: BIASED PULSE DC REACTIVE) Confirmation No.: 9873
 \ SPUTTERING OF OXIDE FILMS)

MAIL STOP AMENDMENT
 Commissioner for Patents
 P.O. Box 1450
 Alexandria, VA 22313-1450

Sir:

SEVENTH SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT
UNDER 37 C.F.R. § 1.97(c)

Pursuant to 37 C.F.R. §§ 1.56 and 1.97(c), Applicant brings to the attention of the Examiner the documents on the attached listing. This Information Disclosure Statement is being filed after the events recited in Section 1.97(b) but, to the undersigned's knowledge, before the mailing date of either a Final action, Quayle action, or a Notice of Allowance. Under the provisions of 37 C.F.R. § 1.97(c), the Commissioner is hereby authorized to charge the fee of \$180.00 to Deposit Account No. 06-0916 for this Information Disclosure Statement.

Copies of the listed non-patent literature documents are attached. Copies of the U.S. patents and patent publications are not enclosed.

Applicant respectfully requests that the Examiner consider the listed documents and indicate that they were considered by making appropriate notations on the attached form.

This submission does not represent that a search has been made or that no better art exists and does not constitute an admission that each or all of the listed documents are material or constitute "prior art." If the Examiner applies any of the documents as prior art against any claims in the application and Applicant determines that the cited documents do not constitute "prior art" under United States law, Applicant reserves the right to present to the office the relevant facts and law regarding the appropriate status of such documents.

Applicant further reserves the right to take appropriate action to establish the patentability of the disclosed invention over the listed documents, should one or more of the documents be applied against the claims of the present application.

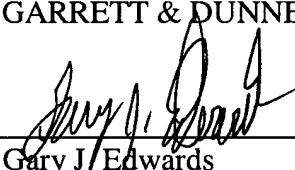
If there is any fee due in connection with the filing of this Statement, please charge the fee to our Deposit Account No. 06-0916.

Respectfully submitted,

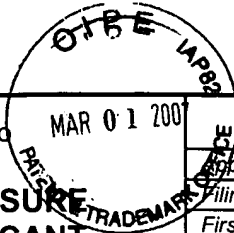
FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Date: March 1, 2007

By: _____


Gary J. Edwards
Reg. No. 41,008

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IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known		
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>			Application Number	10/954,182	
			Filing Date	October 1, 2004	
			First Named Inventor	ZHANG, Hongmei	
			Art Unit	2823	
			Examiner Name	ESTRADA, Michelle	
Sheet	1	of	2	Attorney Docket Number	9140.0016-01

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner Initials	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
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		US-2005/0175287 A1	08-11-2005	Pan et al.	

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

NON PATENT LITERATURE DOCUMENTS			
Examiner Initials	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁸
		BELKIND, A. et al., "Pulsed-DC Reactive Sputtering of Dielectrics: Pulsing Parameter Effects," 43rd Annual Technical Conference Proceedings-Denver: 86-90 (April 15-20, 2000).	
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		Office Action dated December 18, 2006, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Response to Office Action dated March 1, 2007, in U.S. Application No. 11/100,856 (Attorney Docket No. 9140.0015-01).	
		Response to Office Action dated February 6, 2007, in U.S. Application No. 10/101,863 (Attorney Docket No. 9140.0016-00).	
		Supplemental Preliminary Amendment dated February 6, 2007, in U.S. Application No. 11/228,834 (Attorney Docket No. 9140.0016-02).	
		Supplemental Preliminary Amendment dated February 6, 2007, in U.S. Application No. 11/191,643 (Attorney Docket No. 9140.0016-04).	
		Notice of Allowance dated February 21, 2007, in U.S. Application No. 10/789,953 (Attorney Docket No. 9140.0030-00).	
		Response to Office Action dated December 21, 2006, in U.S. Application No. 11/228,805 (Attorney Docket No. 9140.0030-01).	
		Response to Office Action dated January 26, 2007, in U.S. Application No.	

Examiner Signature		Date Considered	
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

**EXPRESS MAIL LABEL NO.
EV 977728687 US**

IDS Form PTO/SB/08: Substitute for form 1449A/PTO				Complete if Known		
INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Use as many sheets as necessary)				Application Number		10/954,182
				Filing Date		October 1, 2004
				First Named Inventor		ZHANG, Hongmei
				Art Unit		2823
				Examiner Name		ESTRADA, Michelle
Sheet	2	of	2	Attorney Docket Number		9140.0016-01

NON PATENT LITERATURE DOCUMENTS		
		10/851,542 (Attorney Docket No. 9140.0033-00).
		Notice of Allowance dated February 22, 2007, in U.S. Application No. 10/851,542 (Attorney Docket No. 9140.0033-00).
		Continuation application and Preliminary Amendment dated December 13, 2006 (Attorney Docket No. 9140.0042-01).

Examiner Signature		Date Considered	
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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/954,182	10/01/2004	Hongmei Zhang	09140-0016-01000	9873

22852 7590 03/14/2007
FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER
LLP
901 NEW YORK AVENUE, NW
WASHINGTON, DC 20001-4413

EXAMINER

ESTRADA, MICHELLE

ART UNIT PAPER NUMBER

2823

SHORTENED STATUTORY PERIOD OF RESPONSE	MAIL DATE	DELIVERY MODE
3 MONTHS	03/14/2007	PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

If NO period for reply is specified above, the maximum statutory period will apply and will expire 6 MONTHS from the mailing date of this communication.

Office Action Summary

Application No. 10/954,182	Applicant(s) ZHANG ET AL.	
Examiner Michelle Estrada	Art Unit 2823	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

- 1) Responsive to communication(s) filed on 06 December 2006.
- 2a) This action is **FINAL**.
- 2b) This action is non-final.
- 3) Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims

- 4) Claim(s) 41-58, 61, 62, 85 and 87-89 is/are pending in the application.
 - 4a) Of the above claim(s) _____ is/are withdrawn from consideration.
- 5) Claim(s) 85 and 87-89 is/are allowed.
- 6) Claim(s) 61 is/are rejected.
- 7) Claim(s) 41-58 and 61 is/are objected to.
- 8) Claim(s) _____ are subject to restriction and/or election requirement.

Application Papers

- 9) The specification is objected to by the Examiner.
- 10) The drawing(s) filed on _____ is/are: a) accepted or b) objected to by the Examiner.

Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

Priority under 35 U.S.C. § 119

- 12) Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
 - a) All b) Some * c) None of:
 - 1. Certified copies of the priority documents have been received.
 - 2. Certified copies of the priority documents have been received in Application No. _____.
 - 3. Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

* See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

- 1) Notice of References Cited (PTO-892)
- 2) Notice of Draftsperson's Patent Drawing Review (PTO-948)
- 3) Information Disclosure Statement(s) (PTO/SB/08)
Paper No(s)/Mail Date _____
- 4) Interview Summary (PTO-413)
Paper No(s)/Mail Date. _____
- 5) Notice of Informal Patent Application
- 6) Other: _____

DETAILED ACTION

After an updated search before allowance, the allowability of claim 61 is withdrawn in view of Moise et al. below.

Claim Rejections - 35 USC § 103

The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

Claim 61 is rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff et al. (6,117,279) in view of Moise et al. (2001/0034106).

Re claim 61, Smolanoff et al. disclose providing a process gas between the target (16) and a substrate (15); providing pulsed DC power to the target (Col. 5, lines 50-55); providing a magnetic field to the target (Col. 6, lines 1-7); and wherein a material is deposited on the substrate (Col. 5, lines 22-26); and an oxide film is formed by reactive sputtering (Col. 6, lines 15+).

Smolanoff et al. does not specifically disclose reconditioning the target.

Moise et al. disclose that it is important to control target conditioning ([0108]).

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff et al. and Moise et al. to enable the conditioning step of

Art Unit: 2823

Moise et al. to be performed in the process of Smolanoff et al. so that the layer results in a better target condition for the deposition of the next layer.

Allowable Subject Matter

Claims 85 and 87-89 allowed.

Claims 41-58 and 62 objected to as being dependent upon a rejected base claim, but would be allowable if rewritten in independent form including all of the limitations of the base claim and any intervening claims.

Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Michelle Estrada whose telephone number is 571-272-1858. The examiner can normally be reached on Monday through Friday.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Matthew Smith can be reached on 571-272-1907. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 571-272-2800.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR.

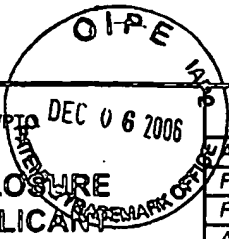
Art Unit: 2823

Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).



Michelle Estrada
Primary Examiner
Art Unit 2823

ME
March 5, 2007



IDS Form PTO/SB/08: Substitute for form 1449A/PTO **Complete if Known**

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet	1	of	3	Application Number	10/954,182
				Filing Date	October 1, 2004
				First Named Inventor	ZHANG, Hongmei
				Art Unit	2823
				Examiner Name	ESTRADA, Michelle
				Attorney Docket Number	9140.0016-01

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner Initials	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
ME		US-6,088,492	07-11-2000	Kaneko et al.	
ME		US-6,288,835 B1	09-11-2001	Nilsson et al.	
ME		US-6,452,717 B1	09-17-2002	Endo	
ME		US-2002/0191916 A1	12-19-2002	Frish et al.	
ME		US-2003/0044118 A1	03-06-2003	Zhou et al.	
ME		US-2003/0143853 A1	07-31-2003	Celii et al.	

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

FOREIGN PATENT DOCUMENTS						
Examiner Initials	Cite No. ¹	Foreign Patent Document	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ^o
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)				
ME		KR 2002-26187	04-06-2002	Hyundai Motor Co Ltd		Abstract
ME		WO 01/82297 A1	11-01-2001	Koninklijke Philips Electronics N.V.		

NON PATENT LITERATURE DOCUMENTS			
Examiner Initials	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ^o
ME		AGRAWAL, G.P., in: Fiber-Optic Communication Systems, 2nd Edition, John Wiley & Sons, New York, pp. 362-399 and 415 (1997).	
ME		MASUDA, H. & KAWAI, S., "Wide-band and gain-flattened hybrid fiber amplifier consisting of an EDFA and a multiwavelength pumped raman amplifier," IEEE Photonics Technology Lett. 11(6):647-649 (1999).	
ME		SNOEKS, E. et al., "Cooperative upconversion in erbium-implanted soda-lime silicate glass optical waveguides," J. Opt. Soc. Am. B 12(8):1468-1474 (1995).	
ME		Final Office Action dated October 12, 2006, in U.S. Application No. 10,291,179 (Attorney Docket No. 9140.0001-00).	
ME		Response to Final Office Action mailed November 3, 2006, in U.S. Application	

Examiner Signature	/Michelle Estrada/	Date Considered	03/02/2007
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

EXPRESS MAIL LABEL NO.
EV 746096154 US

IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known		
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>			<i>Application Number</i>	10/954,182	
			<i>Filing Date</i>	October 1, 2004	
			<i>First Named Inventor</i>	ZHANG, Hongmei	
			<i>Art Unit</i>	2823	
			<i>Examiner Name</i>	ESTRADA, Michelle	
Sheet	2	of	3	<i>Attorney Docket Number</i>	9140.0016-01

NON PATENT LITERATURE DOCUMENTS		
		No. 10,291,179 (Attorney Docket No. 9140.0001-00).
ME		Office Action dated December 1, 2006, in U.S. Application No. 10,291,179 (Attorney Docket No. 9140.0001-00).
		Notice of Allowance mailed March 25, 2004 for US Patent No. 6,827,826 (Atty. Docket No. 09140.0002-02).
		Notice of Allowance issued on October 8, 2002, in U.S. Patent No. 6,533,907 (Atty. Docket No. 09140-0004-00).
		Amendment dated October 19, 2006, in U.S. Application No. 09/903,081 (Atty. Docket No. 09140.0014-00).
		Notice of Allowance issued on October 21, 2004, in U.S. Application No. 10/101,492 (Atty. Docket No. 09140-0015-00).
		Response to Office Action filed September 11, 2006 in U.S. Application No. 11/100,856 (Atty. Docket No. 09140.0015-01.)
		Office Action mailed December 1, 2006, in U.S. Application No. 11/100,856 (Attorney Docket No. 09140.0015-01).
		Office Action mailed September 6, 2006, in U.S. Appl. No. 10/101,863 (Atty. Docket No. 09140.0016-00).
		Final Office Action mailed October 19, 2006, in U.S. Application No. 10/650,461 (Attorney Docket No. 09140.0025-00).
		Voluntary Amendment filed July 26, 2006 in TW Appl. No. 92123625 (Atty. Docket No. 09140.0025-00270).
		Response to Final Office Action filed August 3, 2006, in U.S. Application No. 10/789,953 (Atty. Docket No. 09140.0030-00).
		Notice of Allowance mailed October 23, 2006, in U.S. Application No. 10/789,953 (Atty. Docket No. 09140.0030-00).
		Office Action dated October 12, 2006, for U.S. Application No. 11/228,805 (Attorney Docket No. 09140.0030-01000).
		Office Action dated September 22, 2006 from Korean Patent Office in Appl. No. 10-2005-7016055 (Atty. Docket No. 09140.0030-00202)
		Response to Office Action mailed November 8, 2006, to the Korean Patent Office in Application No. 10-2005-7016055 (Attorney Docket No. 09140.0030-00202).
ME		Response to Office Action from Singapore Patent Office in Appl. No. 200505388-9, dated August 11, 2006 (Atty. Docket No. 9140.0030-00256).

Examiner Signature	/Michelle Estrada/	Date Considered	03/02/2007
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IDS Form PTO/SB/08: Substitute for form 1449A/PTO				Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>				<i>Application Number</i>	10/954,182
				<i>Filing Date</i>	October 1, 2004
				<i>First Named Inventor</i>	ZHANG, Hongmei
				<i>Art Unit</i>	2823
				<i>Examiner Name</i>	ESTRADA, Michelle
Sheet	3	of	3	<i>Attorney Docket Number</i>	9140.0016-01

NON PATENT LITERATURE DOCUMENTS		
ME		Final Office Action dated October 26, 2006, in U.S. Application No. 10/851,542 (Atty. Docket No. 09140.0033-00).
ME		Preliminary Amendment filed July 21, 2006, in U.S. Application No. 11/297,057 (Attorney Docket No. 9140.0042-00).
ME		Supplemental Preliminary Amendment, Substitute Specification, and replacement drawing filed December 6, 2006, in U.S. Application No. 11/297,057 (Attorney Docket No. 9140.0042-00).
ME		Voluntary Amendment filed August 15, 2006 in TW Appl. No. 94143175 (Atty. Docket No. 09140.0042-00270).
ME		PCT International Search Report and Written Opinion for Application No. PCT/US05/44781 dated October 3, 2006 (Attorney Docket No. 9140.0042-00304).

Examiner Signature	/Michelle Estrada/	Date Considered	03/02/2007
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

EXPRESS MAIL LABEL NO. EV 746096154 US

Notice of References Cited	Application/Control No. 10/954,182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.	
	Examiner Michelle Estrada	Art Unit 2823	Page 1 of 1

U.S. PATENT DOCUMENTS

*	Document Number Country Code-Number-Kind Code	Date MM-YYYY	Name	Classification	
*	A	US-2001/0034106	10-2001	Moise et al.	438/396
	B	US-			
	C	US-			
	D	US-			
	E	US-			
	F	US-			
	G	US-			
	H	US-			
	I	US-			
	J	US-			
	K	US-			
	L	US-			
	M	US-			

FOREIGN PATENT DOCUMENTS

*	Document Number Country Code-Number-Kind Code	Date MM-YYYY	Country	Name	Classification
	N				
	O				
	P				
	Q				
	R				
	S				
	T				

NON-PATENT DOCUMENTS

*	Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)
	U
	V
	W
	X

*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)
Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.



PATENT
Customer No. 22,852
Attorney Docket No. 9140.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
)	
ZHANG, Hongmei et al.)	Group Art Unit: 2823
)	
Application No.: 10/954,182)	Examiner: ESTRADA, Michelle
)	
Filed: October 1, 2004)	
)	Confirmation No.: 9873
For: BIASED PULSE DC REACTIVE)	
SPUTTERING OF OXIDE FILMS)	

MAIL STOP AMENDMENT
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

AMENDMENT AND RESPONSE TO OFFICE ACTION

In reply to the Office Action mailed March 14, 2007, please amend the above-identified application as follows:

Amendments to the Claims are reflected in the listing of claims that begins on page 2 of this paper.

Remarks/Arguments follow the amendment sections on page 9 of this paper.

04/02/2007 EEKUBAY1 00000017 060916 10954182
01 FC:1201 3400.00 DA

AMENDMENTS TO THE CLAIMS:

This listing of claims will replace all prior versions and listings of claims in the application:

Claims 1-40 (Canceled).

Claim 41 (Currently amended): The method of claims ~~61~~ or 85 wherein the target is a metallic target and the process gas includes oxygen.

Claim 42 (Currently amended): The method of claims ~~61~~ or 85 wherein the target is a metallic target and the process gas includes one or more of a set consisting of N₂, NH₃, CO, NO, CO₂, halide containing gasses.

Claim 43 (Currently amended): The method of claims ~~61~~ or 85 wherein the target is a ceramic target.

Claim 44 (Canceled).

Claim 45 (Currently amended): The method of claims ~~61~~ or 85 wherein the magnetic field is provided by a moving magnetron.

Claim 46 (Currently amended) The method of claims ~~61~~ or 85 further including holding the temperature of the substrate substantially constant.

Claim 47 (Currently amended): The method of claims ~~61~~ or 85 wherein the process gas includes a mixture of Oxygen and Argon.

Claim 48 (Currently amended): The method of claims ~~61~~ or 85 wherein the Oxygen flow is adjusted to adjust the index of refraction of the film.

Claim 49 (Currently amended): The method of claims ~~61~~ or 85 wherein the process gas

further includes nitrogen.

Claim 50 (Currently amended): The method of claims ~~61 or~~ 85 wherein providing pulsed DC power to a target includes providing pulsed DC power to a target which has an area larger than that of the substrate.

Claim 51 (Currently amended): The method of claims ~~61 or~~ 85, further including uniformly sweeping the target with a magnetic field.

Claim 52 (Previously presented): The method of claim 51 wherein uniformly sweeping the target with a magnetic field includes sweeping a magnet in one direction across the target where the magnet extends beyond the target in the opposite direction.

Claim 53 (Currently Amended): The method of claims ~~61 or~~ 85 wherein the target is an alloyed target.

Claim 54 (Previously presented): The method of claim 53 wherein the alloyed target includes one or more rare-earth ions.

Claim 55 (Previously presented): The method of claim 53 wherein the alloyed target includes Si and Al.

Claim 56 (Previously presented): The method of claim 53 wherein the alloyed target includes one or more elements taken from a set consisting of Si, Al, Er, Yb, Zn, Ga, Ge, P, As, Sn, Sb, Pb, Ag, Au, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Tm, and Lu.

Claim 57 (Previously presented): The method of claim 53 wherein the alloyed target is a tiled target.

Claim 58 (Previously presented): The method of claim 57 wherein each tile of the tiled target is formed by prealloy atomization and hot isostatic pressing of a powder.

Claims 59-61 (Canceled).

Claim 62 (Currently Amended): ~~The method of claim 61, wherein reconditioning the target, includes~~ A method of depositing a film on a substrate, comprising:

providing a process gas between a conductive target and a substrate;

providing pulsed DC power to the target;

providing a magnetic field to the target; and

reconditioning the target;

wherein reconditioning the target includes reactive sputtering in the metallic mode and then reactive sputtering in the poison mode.

Claims 63-84 (Canceled).

Claim 85 (Previously presented): A method of depositing a film on a substrate, comprising:

providing a process gas between a target and a substrate;

providing pulsed DC power to the target;

providing a magnetic field to the target; and

wherein a material is deposited on the substrate, and an oxide film is formed by reactive sputtering in a mode between a metallic mode and a poison mode.

Claim 86 (Canceled).

Claim 87 (Previously presented): The method according to claims ~~61, 62,~~ or 85, further including a narrow band-rejection filter that rejects power at an RF frequency, and further including

providing an RF bias to the substrate at the RF frequency.

Claim 88 (Previously presented): The method according to claim 87, wherein the narrow band-rejection filter has a bandwidth of about 100 kHz.

Claim 89 (Previously presented): The method according to claim 87, wherein the RF frequency is about 2 MHz.

Claims 90-92 (Canceled).

Claim 93 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the target is a metallic target and the process gas includes oxygen.

Claim 94 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the target is a metallic target and the process gas includes one or more of a set consisting of N₂, NH₃, CO, NO, CO₂, halide containing gasses.

Claim 95 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the target is a ceramic target.

Claim 96 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;

wherein the magnetic field is provided by a moving magnetron.

Claim 97 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
further including holding the temperature of the substrate substantially constant.

Claim 98 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the process gas includes a mixture of Oxygen and Argon.

Claim 99 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the Oxygen flow is adjusted to adjust the index of refraction of the film.

Claim 100 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the process gas further includes nitrogen.

Claim 101 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;

providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;

wherein providing pulsed DC power to a target includes providing pulsed DC power to a target which has an area larger than that of the substrate.

Claim 102 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
further including uniformly sweeping the target with a magnetic field.

Claim 103 (New): The method of claim 102 wherein uniformly sweeping the target with a magnetic field includes sweeping a magnet in one direction across the target where the magnet extends beyond the target in the opposite direction.

Claim 104 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the target is an alloyed target.

Claim 105 (New): The method of claim 104 wherein the alloyed target includes one or more rare-earth ions.

Claim 106 (New): The method of claim 104 wherein the alloyed target includes Si and Al.

Claim 107 (New): The method of claim 104 wherein the alloyed target includes one or more elements taken from a set consisting of Si, Al, Er, Yb, Zn, Ga, Ge, P, As, Sn, Sb, Pb, Ag,

Au, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy Ho, Tm, and Lu.

Claim 108 (New): The method of claim 104 wherein the alloyed target is a tiled target.

Claim 109 (New): The method of claim 108 wherein each tile of the tiled target is formed by prealloy atomization and hot isostatic pressing of a powder.

REMARKS

Claims 41-43, 45-58, 61, 62, 85, and 87-89 are pending in present application. The Examiner has rejected claim 61, objected to claims 41-43, 45-58, and 62, and allowed claims 85 and 87-89. With this response, Applicants have amended claims 41-43, 45-53, and 62, canceled claim 61, and added new claims 93-109.

Upon entry of the present amendments, claims 41-43, 45-58, 62, 85, 87-89, and 93-109 will be pending in this application.

Claim Rejections Under 35 U.S.C. § 103

The Examiner rejected claim 61 under 35 U.S.C. § 103(a) as allegedly being unpatentable over Smolanoff et al. (6,117,279) in view of Moise et al. (2001/0034106). OA, ¶3.

To facilitate prosecution, and not in acquiescence to the rejection, Applicants have canceled claim 61.

Objected Claims

The Examiner has allowed claim 85 and objected to claims 41-43, 45-58, and 62. Claims 41-43 and 45-58 have been amended to remove their dependency on rejected claim 61. Claims 41-43 and 45-58, as amended, now depend only on claim 85, which has been allowed. Therefore, claims 41-43 and 45-58 are allowable for at least the same reasons as is claim 85.

Claim 62 has been amended to include all of the limitations of claim 61, from which it depends. Claim 62 is therefore allowable.

Claims 93-109 correspond to claims 41-43 and 45-58, respectively. Each of claims 93-104 include the limitations of claim 61. Claims 93-104, as indicated by the Examiner, are now allowable. Claims 105-108 depend from claim 104 and are allowable for at least the same

reason as is claim 104. Claim 109 depends from claim 108 and is allowable for at least the same reason as is claim 108.

Information Disclosure Statement

Applicant notes the Examiner's acknowledgement of the Supplemental Information Disclosure Statement ("Supplemental IDS") filed on December 6, 2006, in which Applicant thanks the Examiner. Applicant also notes the Supplemental Information Disclosure Statement filed on March 1, 2007. Applicants respectfully request that the Examiner consider the cited documents on the March 1, 2007, Supplemental Information Disclosure Statement, and the Supplemental Information Disclosure Statement filed concurrently herewith. Applicants remind the Examiner of their duty to disclosure any art that is relevant to the above-referenced application. To this effect, the MPEP 2001.06(b) states:

The individuals covered by 37 CFR 1.56 have a duty to bring to the attention of the examiner, or other Office official involved with the examination of a particular application, information within their knowledge as to other copending United States applications which are "material to patentability" of the application in question. As set forth by the court in *Armour & Co. v. Swift & Co.*, 466 F.2d 767, 779, 175 USPQ 70, 79 (7th Cir. 1972):

Accordingly, the individuals covered by 37 CFR 1.56 cannot assume that the examiner of a particular application is necessarily aware of other applications which are "material to patentability" of the application in question, but must instead bring such other applications to the attention of the examiner. *See Dayco Prod., Inc. v. Total Containment, Inc.*, 329 F.3d 1358, 1365-69, 66 USPQ2d 1801, 1806-08 (Fed. Cir. 2003). For example, if a particular inventor has different applications pending in which similar subject matter but patentably indistinct claims are present that fact must be disclosed to the examiner of each of the involved applications. Similarly, the prior art references from one application must be made of record in another subsequent application if such prior art references are "material to patentability" of the subsequent application. *See Dayco Prod.*, 329 F.3d at 1369, 66 USPQ2d at 1808.

By acknowledging the Supplemental IDS and considering the references listed on the PTO/SB/08 forms submitted along with the Supplemental IDS, the Examiner has assisted Applicant in fulfilling their duty to disclose information in conjunction with the guidelines noted above. Applicant duly appreciates the Examiner's assistance in this matter.

Conclusion

In view of the foregoing amendments and remarks, Applicants respectfully request reconsideration and reexamination of this application and the timely allowance of the pending claims.

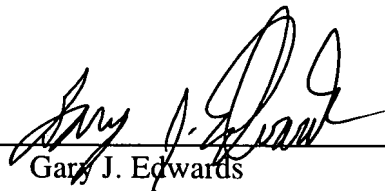
Please grant any extensions of time required to enter this response and charge any additional required fees to Deposit Account No. 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Date: March 30, 2007

By: _____



Gary J. Edwards
Reg. No. 41,008

**EXPRESS MAIL LABEL NO.
EV 977728506 US**



PATENT
Customer No. 22,852
Attorney Docket No. 9140.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)
)
ZHANG, Hongmei et al.) Group Art Unit: 2823
)
Application No.: 10/954,182) Examiner: ESTRADA, Michelle
)
Filed: October 1, 2004)
) Confirmation No.: 9873
For: BIASED PULSE DC REACTIVE)
SPUTTERING OF OXIDE FILMS)

MAIL STOP AMENDMENT
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

EIGHTH SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT
UNDER 37 C.F.R. § 1.97(c)

Pursuant to 37 C.F.R. §§ 1.56 and 1.97(c), Applicant brings to the attention of the Examiner the documents on the attached listing. This Information Disclosure Statement is being filed after the events recited in Section 1.97(b) but, to the undersigned's knowledge, before the mailing date of either a Final action, Quayle action, or a Notice of Allowance. Under the provisions of 37 C.F.R. § 1.97(c), the Commissioner is hereby authorized to charge the fee of \$180.00 to Deposit Account No. 06-0916 for this Information Disclosure Statement.

Copies of the listed non-patent literature documents are attached. Copies of the U.S. patent and patent publication are not enclosed.

Applicant respectfully requests that the Examiner consider the listed documents and indicate that they were considered by making appropriate notations on the attached form.

This submission does not represent that a search has been made or that no better art exists and does not constitute an admission that each or all of the listed documents are material or constitute "prior art." If the Examiner applies any of the documents as prior art against any claims in the application and Applicant determines that the cited documents do not constitute "prior art" under United States law, Applicant reserves the right to present to the office the relevant facts and law regarding the appropriate status of such documents.

Applicant further reserves the right to take appropriate action to establish the patentability of the disclosed invention over the listed documents, should one or more of the documents be applied against the claims of the present application.

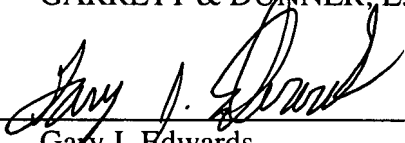
If there is any fee due in connection with the filing of this Statement, please charge the fee to our Deposit Account No. 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Date: March 30, 2007

By: _____


Gary J. Edwards
Reg. No. 41,008

**EXPRESS MAIL LABEL NO.
EV 977728506 US**

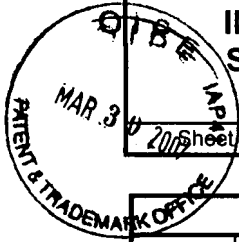
Complete if Known

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Application Number	10/954,182
Filing Date	October 1, 2004
First Named Inventor	ZHANG, Hongmei
Art Unit	2823
Examiner Name	ESTRADA, Michelle
Attorney Docket Number	9140.0016-01

Sheet 1 of 1



U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS

Examiner Initials	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
		US-6,391,166 B1	05-21-2002	Wang	
		US-20070053139 A1	03-08-2007	Zhang et al.	

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

NON PATENT LITERATURE DOCUMENTS

Examiner Initials	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
		Response to Office Action dated March 19, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Response to Office Action dated February 20, 2007, in U.S. Application No. 10/650,461 (Attorney Docket No. 9140.0025-00).	
		Office Action dated March 6, 2007, in U.S. Application No. 10/650,461 (Attorney Docket No. 9140.0025-00).	
		Notice of Allowance dated March 26, 2007, in U.S. Application No. 11/228,805 (Attorney Docket No. 9140.0030-01).	
		Supplemental Notice of Allowance dated March 15, 2007, in U.S. Application No. 10/851,542 (Attorney Docket No. 9140.0033-00).	
		Voluntary Amendment dated March 8, 2007, in TW Appl. No. 93114518 (Attorney Docket No. 9140.0033-00270).	
		Application filed March 22, 2007 (Attorney Docket No. 9140.0033-01).	

Examiner Signature		Date Considered	
--------------------	--	-----------------	--

EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

**EXPRESS MAIL LABEL NO.
EV 977728506 US**

04-02-07

Law



PATENT
Customer No. 22,852
Attorney Docket No. 9140.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)
)
 ZHANG, Hongmei et al.) Group Art Unit: 2823
)
 Application No.: 10/954,182) Examiner: ESTRADA, Michelle
)
 Filed: October 1, 2004)
) Confirmation No.: 9873
 For: BIASED PULSE DC REACTIVE)
 SPUTTERING OF OXIDE FILMS)

MAIL STOP AMENDMENT

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

TRANSMITTAL LETTER

Enclosed is a reply to the Office Action of March 14, 2007. The item(s) checked below are appropriate:

The claims are calculated below:

	Claims Remaining After Amendment		Highest Number Previously Paid	Present Extra	Rate	Additional Fee
Total	39	-	45	0	x \$ 50	\$ 0
Indep.	19	-	2	17	x \$200	3,400.00
<input type="checkbox"/> First Presentation of Multiple Dep. Claim(s)					+\$360	0
Subtotal						\$ 3,400.00
Reduction by 1/2 if small entity						- 0
TOTAL						\$ 3,400.00

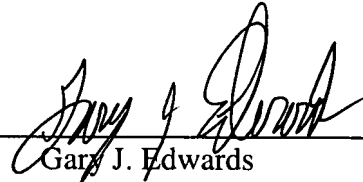
The Commissioner is hereby authorized to charge the fee of \$3,580.00 to Deposit Account No. 06-0916 to cover:

\$3,400.00 for the claim fees;
\$ 180.00 for the Supplemental Information Disclosure Statement.

Please grant any extensions of time required to enter this response and charge any additional required fees to Deposit Account 06-0916.

Dated: March 30, 2007

By: _____



Gary J. Edwards
Reg. No. 41,008

**EXPRESS MAIL LABEL NO.
EV 977728506 US**

Cont. Page

PATENT APPLICATION FEE DETERMINATION RECORD

Effective December 8, 2004

10954/82

CLAIMS AS AMENDED - PART II

3/30/07

AMENDMENT A	(Column 1)	(Column 2)	(Column 3)
	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
Total	• 39	Minus	•• 61
Independent	• 13	Minus	••• 9
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

SMALL ENTITY OR		OTHER THAN SMALL ENTITY	
RATE	ADDITIONAL FEE	RATE	ADDITIONAL FEE
X\$ 25=		X\$50=	-
X100=		X200=	800
+180=		+360=	
TOTAL ADDIT. FEE		TOTAL ADDIT. FEE 800	

AMENDMENT B	(Column 1)	(Column 2)	(Column 3)
	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
Total	•	Minus	••
Independent	•	Minus	•••
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

SMALL ENTITY OR		OTHER THAN SMALL ENTITY	
RATE	ADDITIONAL FEE	RATE	ADDITIONAL FEE
X\$ 25=		X\$50=	
X100=		X200=	
+180=		+360=	
TOTAL ADDIT. FEE		TOTAL ADDIT. FEE	

AMENDMENT C	(Column 1)	(Column 2)	(Column 3)
	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
Total	•	Minus	••
Independent	•	Minus	•••
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

SMALL ENTITY OR		OTHER THAN SMALL ENTITY	
RATE	ADDITIONAL FEE	RATE	ADDITIONAL FEE
X\$ 25=		X\$50=	
X100=		X200=	
+180=		+360=	
TOTAL ADDIT. FEE		TOTAL ADDIT. FEE	

AMENDMENT C	(Column 1)	(Column 2)	(Column 3)
	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
Total	•	Minus	••
Independent	•	Minus	•••
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

SMALL ENTITY OR		OTHER THAN SMALL ENTITY	
RATE	ADDITIONAL FEE	RATE	ADDITIONAL FEE
X\$ 25=		X\$50=	
X100=		X200=	
+180=		+360=	
TOTAL ADDIT. FEE		TOTAL ADDIT. FEE	

• If the entry in column 1 is less than the entry in column 2, write "0" in column 3.
 • If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20."
 • If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3."
 The "Highest Number Previously Paid For" (Total or Independent) is the highest number found in the appropriate box in column 1.



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
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P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/954,182	10/01/2004	Hongmei Zhang	09140-0016-01000	9873

22852 7590 04/12/2007
FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER
LLP
901 NEW YORK AVENUE, NW
WASHINGTON, DC 20001-4413

EXAMINER

ESTRADA, MICHELLE

ART UNIT PAPER NUMBER

2823

SHORTENED STATUTORY PERIOD OF RESPONSE	MAIL DATE	DELIVERY MODE
30 DAYS	04/12/2007	PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

If NO period for reply is specified above, the maximum statutory period will apply and will expire 6 MONTHS from the mailing date of this communication.

ED

Notice of Non-Compliant Amendment (37 CFR 1.121)	Application No.	Applicant(s)	
	10954182	Examiner	Art Unit

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

The amendment document filed on 30 March 2007 is considered non-compliant because it has failed to meet the requirements of 37 CFR 1.121 or 1.4. In order for the amendment document to be compliant, correction of the following item(s) is required.

THE FOLLOWING MARKED (X) ITEM(S) CAUSE THE AMENDMENT DOCUMENT TO BE NON-COMPLIANT:

- 1. Amendments to the specification:
 - A. Amended paragraph(s) do not include markings.
 - B. New paragraph(s) should not be underlined.
 - C. Other _____.
- 2. Abstract:
 - A. Not presented on a separate sheet. 37 CFR 1.72.
 - B. Other _____.
- 3. Amendments to the drawings:
 - A. The drawings are not properly identified in the top margin as "Replacement Sheet," "New Sheet," or "Annotated Sheet" as required by 37 CFR 1.121(d).
 - B. The practice of submitting proposed drawing correction has been eliminated. Replacement drawings showing amended figures, without markings, in compliance with 37 CFR 1.84 are required.
 - C. Other _____.
- 4. Amendments to the claims:
 - A. A complete listing of all of the claims is not present.
 - B. The listing of claims does not include the text of all pending claims (including withdrawn claims)
 - C. Each claim has not been provided with the proper status identifier, and as such, the individual status of each claim cannot be identified. Note: the status of every claim must be indicated after its claim number by using one of the following status identifiers: (Original), (Currently amended), (Canceled), (Previously presented), (New), (Not entered), (Withdrawn) and (Withdrawn-currently amended).
 - D. The claims of this amendment paper have not been presented in ascending numerical order.
 - E. Other: see claim 87.
- 5. Other (e.g., the amendment is unsigned or not signed in accordance with 37 CFR 1.4):

For further explanation of the amendment format required by 37 CFR 1.121, see MPEP § 714.

TIME PERIODS FOR FILING A REPLY TO THIS NOTICE:

1. Applicant is given **no new time period** if the non-compliant amendment is an after-final amendment, an amendment filed after allowance, or a drawing submission (only). If applicant wishes to resubmit the non-compliant after-final amendment with corrections, the **entire corrected amendment** must be resubmitted.
2. Applicant is given **one month**, or thirty (30) days, whichever is longer, from the mail date of this notice to supply the correction, if the non-compliant amendment is one of the following: a preliminary amendment, a non-final amendment (including a submission for a request for continued examination (RCE) under 37 CFR 1.114), a supplemental amendment filed within a suspension period under 37 CFR 1.103(a) or (c), and an amendment filed in response to a *Quayle* action. If any of above boxes 1. to 4. are checked, the correction required is only the **corrected section** of the non-compliant amendment in compliance with 37 CFR 1.121.

Extensions of time are available under 37 CFR 1.136(a) only if the non-compliant amendment is a non-final amendment or an amendment filed in response to a *Quayle* action.

Failure to timely respond to this notice will result in:

Abandonment of the application if the non-compliant amendment is a non-final amendment or an amendment filed in response to a *Quayle* action; or

Non-entry of the amendment if the non-compliant amendment is a preliminary amendment or supplemental amendment.

/Ruby Johnson/

571-272-4359

Legal Instruments Examiner (LIE), if applicable

Telephone No.



04-25-07

DW

PATENT
Customer No. 22,852
Attorney Docket No. 9140.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
)	
ZHANG, Hongmei et al.)	Group Art Unit: 2823
)	
Application No.: 10/954,182)	Examiner: ESTRADA, Michelle
)	
Filed: October 1, 2004)	
)	Confirmation No.: 9873
For: BIASED PULSE DC REACTIVE)	
SPUTTERING OF OXIDE FILMS)	

MAIL STOP AMENDMENT
 Commissioner for Patents
 P.O. Box 1450
 Alexandria, VA 22313-1450

Sir:

RESPONSE TO NOTICE OF NON-COMPLIANT AMENDMENT

This is in response to the Notice of Non-Compliant Amendment (“Notice”) dated April 12, 2007. Applicants respond to the reason for non-compliance as follows:

Amendments to the Claims are reflected in the listing of claims that begins on page 2 of this paper.

Remarks follow the amendment sections on page 9 of this paper.

AMENDMENTS TO THE CLAIMS:

This listing of claims will replace all prior versions and listings of claims in the application:

Claims 1-40 (Canceled).

Claim 41 (Currently amended): The method of claims ~~61-85~~ wherein the target is a metallic target and the process gas includes oxygen.

Claim 42 (Currently amended): The method of claims ~~61-85~~ wherein the target is a metallic target and the process gas includes one or more of a set consisting of N₂, NH₃, CO, NO, CO₂, halide containing gasses.

Claim 43 (Currently amended): The method of claims ~~61-85~~ wherein the target is a ceramic target.

Claim 44 (Canceled).

Claim 45 (Currently amended): The method of claims ~~61-85~~ wherein the magnetic field is provided by a moving magnetron.

Claim 46 (Currently amended) The method of claims ~~61-85~~ further including holding the temperature of the substrate substantially constant.

Claim 47 (Currently amended): The method of claims ~~61-85~~ wherein the process gas includes a mixture of Oxygen and Argon.

Claim 48 (Currently amended): The method of claims ~~61-85~~ wherein the Oxygen flow is adjusted to adjust the index of refraction of the film.

Claim 49 (Currently amended): The method of claims ~~61-85~~ wherein the process gas

further includes nitrogen.

Claim 50 (Currently amended): The method of claims ~~61~~ or 85 wherein providing pulsed DC power to a target includes providing pulsed DC power to a target which has an area larger than that of the substrate.

Claim 51 (Currently amended): The method of claims ~~61~~ or 85, further including uniformly sweeping the target with a magnetic field.

Claim 52 (Previously presented): The method of claim 51 wherein uniformly sweeping the target with a magnetic field includes sweeping a magnet in one direction across the target where the magnet extends beyond the target in the opposite direction.

Claim 53 (Currently amended): The method of claims ~~61~~ or 85 wherein the target is an alloyed target.

Claim 54 (Previously presented): The method of claim 53 wherein the alloyed target includes one or more rare-earth ions.

Claim 55 (Previously presented): The method of claim 53 wherein the alloyed target includes Si and Al.

Claim 56 (Previously presented): The method of claim 53 wherein the alloyed target includes one or more elements taken from a set consisting of Si, Al, Er, Yb, Zn, Ga, Ge, P, As, Sn, Sb, Pb, Ag, Au, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Tm, and Lu.

Claim 57 (Previously presented): The method of claim 53 wherein the alloyed target is a tiled target.

Claim 58 (Previously presented): The method of claim 57 wherein each tile of the tiled target is formed by prealloy atomization and hot isostatic pressing of a powder.

Claims 59-61 (Canceled).

Claim 62 (Currently amended): ~~The method of claim 61, wherein reconditioning the target, includes~~ A method of depositing a film on a substrate, comprising:

providing a process gas between a conductive target and a substrate;

providing pulsed DC power to the target;

providing a magnetic field to the target; and

reconditioning the target;

wherein reconditioning the target includes reactive sputtering in the metallic mode and then reactive sputtering in the poison mode.

Claims 63-84 (Canceled).

Claim 85 (Previously presented): A method of depositing a film on a substrate, comprising:

providing a process gas between a target and a substrate;

providing pulsed DC power to the target;

providing a magnetic field to the target; and

wherein a material is deposited on the substrate, and an oxide film is formed by reactive sputtering in a mode between a metallic mode and a poison mode.

Claim 86 (Canceled).

Claim 87 (Currently amended): The method according to claims ~~61, 62,~~ or 85, further including a narrow band-rejection filter that rejects power at an RF frequency, and further including

providing an RF bias to the substrate at the RF frequency.

Claim 88 (Previously presented): The method according to claim 87, wherein the narrow band-rejection filter has a bandwidth of about 100 kHz.

Claim 89 (Previously presented): The method according to claim 87, wherein the RF frequency is about 2 MHz.

Claims 90-92 (Canceled).

Claim 93 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the target is a metallic target and the process gas includes oxygen.

Claim 94 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the target is a metallic target and the process gas includes one or more of a set consisting of N₂, NH₃, CO, NO, CO₂, halide containing gasses.

Claim 95 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the target is a ceramic target.

Claim 96 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;

wherein the magnetic field is provided by a moving magnetron.

Claim 97 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
further including holding the temperature of the substrate substantially constant.

Claim 98 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the process gas includes a mixture of Oxygen and Argon.

Claim 99 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the Oxygen flow is adjusted to adjust the index of refraction of the film.

Claim 100 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the process gas further includes nitrogen.

Claim 101 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;

providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;

wherein providing pulsed DC power to a target includes providing pulsed DC power to a target which has an area larger than that of the substrate.

Claim 102 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
further including uniformly sweeping the target with a magnetic field.

Claim 103 (New): The method of claim 102 wherein uniformly sweeping the target with a magnetic field includes sweeping a magnet in one direction across the target where the magnet extends beyond the target in the opposite direction.

Claim 104 (New): A method of depositing film on a substrate, comprising:
providing a process gas between a conductive target and a substrate;
providing pulsed DC power to the target;
providing a magnetic field to the target; and
reconditioning the target;
wherein the target is an alloyed target.

Claim 105 (New): The method of claim 104 wherein the alloyed target includes one or more rare-earth ions.

Claim 106 (New): The method of claim 104 wherein the alloyed target includes Si and Al.

Claim 107 (New): The method of claim 104 wherein the alloyed target includes one or more elements taken from a set consisting of Si, Al, Er, Yb, Zn, Ga, Ge, P, As, Sn, Sb, Pb, Ag,

Au, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy Ho, Tm, and Lu.

Claim 108 (New): The method of claim 104 wherein the alloyed target is a tiled target.

Claim 109 (New): The method of claim 108 wherein each tile of the tiled target is formed by prealloy atomization and hot isostatic pressing of a powder.

REMARKS

In response to the Notice of Non-Compliant Amendment dated April 12, 2007,
Applicants add the proper status identifier to claim 87.

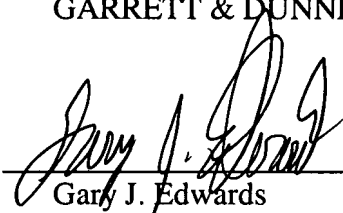
Please grant any extensions of time required to enter this response and charge any
additional required fees to Deposit Account No. 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Date: April 23, 2007

By: _____



Gary J. Edwards
Reg. No. 41,008

**EXPRESS MAIL LABEL NO.
EV 977728395 US**

Cont. Page
PATENT APPLICATION FEE DETERMINATION RECORD
 Effective December 8, 2004

10954182

CLAIMS AS AMENDED - PART II

3/30/07

AMENDMENT A	(Column 1)	(Column 2)	(Column 3)
	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
Total	39	61	-
Independent	13	9	4
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

SMALL ENTITY OR		OTHER THAN SMALL ENTITY	
RATE	ADDITIONAL FEE	RATE	ADDITIONAL FEE
X\$ 25=		X\$50=	-
X100=		X200=	800
+180=		+360=	
TOTAL ADDIT. FEE		TOTAL ADDIT. FEE 800	

AMENDMENT B	(Column 1)	(Column 2)	(Column 3)
	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
Total	39	61	0
Independent	13	13	0
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

SMALL ENTITY OR		OTHER THAN SMALL ENTITY	
RATE	ADDITIONAL FEE	RATE	ADDITIONAL FEE
X\$ 25=		X\$50=	0
X100=		X200=	6
+180=		+360=	0
TOTAL ADDIT. FEE		TOTAL ADDIT. FEE 0	

AMENDMENT C	(Column 1)	(Column 2)	(Column 3)
	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
Total			
Independent			
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

SMALL ENTITY OR		OTHER THAN SMALL ENTITY	
RATE	ADDITIONAL FEE	RATE	ADDITIONAL FEE
X\$ 25=		X\$50=	
X100=		X200=	
+180=		+360=	
TOTAL ADDIT. FEE		TOTAL ADDIT. FEE	

AMENDMENT C	(Column 1)	(Column 2)	(Column 3)
	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA
Total			
Independent			
FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <input type="checkbox"/>			

SMALL ENTITY OR		OTHER THAN SMALL ENTITY	
RATE	ADDITIONAL FEE	RATE	ADDITIONAL FEE
X\$ 25=		X\$50=	
X100=		X200=	
+180=		+360=	
TOTAL ADDIT. FEE		TOTAL ADDIT. FEE	

* If the entry in column 1 is less than the entry in column 2, write "0" in column 3.
 * If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20."
 * If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3."
 The "Highest Number Previously Paid For" (Total or Independent) is the highest number found in the appropriate box in column 1.



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www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
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10/954,182	10/01/2004	Hongmei Zhang	09140-0016-01000	9873
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22852 7590 07/24/2007
 FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER
 LLP
 901 NEW YORK AVENUE, NW
 WASHINGTON, DC 20001-4413

EXAMINER

ESTRADA, MICHELLE

ART UNIT	PAPER NUMBER
----------	--------------

2823

MAIL DATE	DELIVERY MODE
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07/24/2007

PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

DETAILED ACTION

Claim Rejections - 35 USC § 103

The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

Claims 93, 94, 96, 98, 100, 102 and 103 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff et al. (6,117,279) in view of Moise et al. (2001/0034106).

Re claims 93 and 94, Smolanoff et al. disclose providing a process gas between the target (16) and a substrate (15); providing pulsed DC power to the target (Col. 5, lines 50-55); providing a magnetic field to the target (Col. 6, lines 1-7); and wherein a material is deposited on the substrate (Col. 5, lines 22-26); and an oxide film is formed by reactive sputtering (Col. 6, lines 15+); wherein the process gas includes oxygen; wherein the process gas includes N₂ or NH₃.

Smolanoff et al. does not specifically disclose reconditioning the target.

Moise et al. disclose that it is important to control target conditioning ([0108]).

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff et al. and Moise et al. to enable the conditioning step of Moise et al. to be performed in the process of Smolanoff et al. so that the layer results in a better target condition for the deposition of the next layer.

Re claim 96, Smolanoff et al. disclose wherein the magnetic field is provided by a moving magnetron (Col. 5, lines 39-49).

Re claim 98, Smolanoff et al. disclose wherein the process gas includes a mixture of oxygen and argon (Col. 7, lines 22-27).

Re claim 100, Smolanoff et al. disclose wherein the process gas further includes nitrogen (Col. 7, lines 25-26).

Re claim 102, Smolanoff et al. disclose further including uniformly sweeping the target with a magnetic field (Col. 6, lines 1-6).

Re claim 103, Smolanoff et al. disclose wherein sweeping the target with a magnetic field includes sweeping a magnet in one direction across the target where the magnet extends beyond the target in the opposite direction (Col. 6, lines 1-6).

Claims 97, 99 and 101 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff et al. in view of Moise et al. as applied to claims 93, 94, 96, 98, 100, 102 and 103 above, and further in view of Chen et al. (2004/0077161).

The combination does not disclose holding the temperature of the substrate substantially constant.

Re claim 97, Chen et al. disclose further including holding the temperature of the substrate substantially constant (Page 3, Paragraph [0046]).

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff et al., Moise et al. and Chen et al. to enable the temperature step of the combination to be performed according to the teachings of Chen et al.

because one of ordinary skill in the art would have been motivated to look to alternative suitable methods of performing the disclosed temperature step of the combination and art recognized suitability for an intended purpose has been recognized to be motivation to combine. See MPEP 2144.07. Furthermore, Chen discloses that the temperature is maintained or controlled at a desired temperature, which is suitable for a particular process.

Re claim 99, Chen et al. disclose wherein the oxygen flow is adjusted by the mass flow controllers; thereby it will adjust the index refraction of the film.

Re claim 101, Chen et al. disclose wherein providing pulsed DC power to a target includes providing pulsed DC power to a target, which has an area larger than that of the substrate (See fig. 3).

Claims 95 and 104-109 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff et al. in view of Moise et al. as applied to claims 93, 94, 96, 98, 100, 102 and 103 above, and further in view of Milonopoulou et al. (2003/0175142).

Re claim 95, the combination of Smolanoff et al. and Moise et al. does not disclose wherein the target is a ceramic target.

Milonopoulou et al. disclose forming a coating layer on a substrate; providing a target (12), which is ceramic (Abstract).

Art Unit: 2823

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff et al., Moise et al. and Milonopoulou et al. to enable the target material of Smolanoff et al. to be the same according to the teachings of Milonopoulou et al. because one of ordinary skill in the art would have been motivated to look to alternative suitable target materials of the disclosed target of Smolanoff et al. and art recognized suitability for an intended purpose has been recognized to be motivation to combine. See MPEP 2144.07.

Using a specific type of filter (narrow band-rejection) is a matter of design choice depending on the quality of product needed, and it is obvious that the filter is going to work at certain frequencies.

Re claim 104, Milonopoulou et al. disclose wherein the target is an alloyed target (Abstract).

Re claim 105, Milonopoulou et al. disclose wherein the alloyed target includes one or more rare earth ions.

Re claim 106, Milonopoulou et al. disclose wherein the alloyed target includes Si and Al.

Re claim 107, Milonopoulou et al. disclose wherein the alloyed target includes one or more elements taken from a set consisting of Si, Al, Er and Yb.

Re claim 108, Milonopoulou et al. disclose wherein the alloyed target is a tiled target.

Re claim 109, Milonopoulou et al. disclose wherein each tiled target is formed by pre-alloy atomization and hot isostatic pressing of a powder (Page 2, Paragraph [0020]).

Allowable Subject Matter

Claims 41-43, 45-58, 62, 85 and 87-89 are allowed.

Conclusion

Applicant's amendment necessitated the new ground(s) of rejection presented in this Office action. Accordingly, **THIS ACTION IS MADE FINAL**. See MPEP § 706.07(a). Applicant is reminded of the extension of time policy as set forth in 37 CFR 1.136(a).

A shortened statutory period for reply to this final action is set to expire **THREE MONTHS** from the mailing date of this action. In the event a first reply is filed within **TWO MONTHS** of the mailing date of this final action and the advisory action is not mailed until after the end of the **THREE-MONTH** shortened statutory period, then the shortened statutory period will expire on the date the advisory action is mailed, and any extension fee pursuant to 37 CFR 1.136(a) will be calculated from the mailing date of the advisory action. In no event, however, will the statutory period for reply expire later than **SIX MONTHS** from the date of this final action.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Michelle Estrada whose telephone number is 571-272-1858. The examiner can normally be reached on Monday through Friday.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Matthew Smith can be reached on 571-272-1907. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

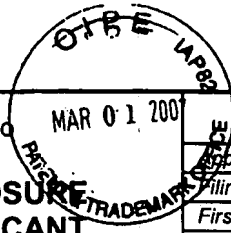
Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 571-272-2800.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).



Michelle Estrada
Primary Examiner
Art Unit 2823

ME
July 16, 2007



IDS Form PTO/SB/08: Substitute for form 1449A/PTO		Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Use as many sheets as necessary)		Application Number	10/954,182
		Filing Date	October 1, 2004
		First Named Inventor	ZHANG, Hongmei
		Art Unit	2823
		Examiner Name	ESTRADA, Michelle
Sheet	1	of	2
		Attorney Docket Number	9140.0016-01

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner Initials	Cite No. ¹	Document Number	Issue or Publication Date	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)	MM-DD-YYYY		
<i>[Signature]</i>		US-6,154,582	11-28-2000	Bazylenko et al.	
		US-2005/0175287 A1	08-11-2005	Pan et al.	

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

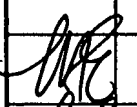

NON PATENT LITERATURE DOCUMENTS			
Examiner Initials	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
<i>[Signature]</i>		BELKIND, A. et al., "Pulsed-DC Reactive Sputtering of Dielectrics: Pulsing Parameter Effects," 43rd Annual Technical Conference Proceedings-Denver: 86-90 (April 15-20, 2000).	
<i>[Signature]</i>		SCHOLL, R., "Power Supplies for Pulsed Plasma Technologies: State-Of-The-Art And Outlook," Advances Energy Industries, Inc., pages 1-8 (1999).	
		SCHOLL, R., "Power Systems for Reactive Sputtering of Insulating Films," Advances Energy Industries, Inc., pages 1-8 (August 2001).	
		Response to Office Action and Terminal Disclaimer dated March 1, 2007, in U.S. Application No. 10/291,179 (Attorney Docket No. 9140.0001-00).	
		Office Action dated December 18, 2006, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Response to Office Action dated March 1, 2007, in U.S. Application No. 11/100,856 (Attorney Docket No. 9140.0015-01).	
		Response to Office Action dated February 6, 2007, in U.S. Application No. 10/101,863 (Attorney Docket No. 9140.0016-00).	
		Supplemental Preliminary Amendment dated February 6, 2007, in U.S. Application No. 11/228,834 (Attorney Docket No. 9140.0016-02).	
		Supplemental Preliminary Amendment dated February 6, 2007, in U.S. Application No. 11/191,643 (Attorney Docket No. 9140.0016-04).	
		Notice of Allowance dated February 21, 2007, in U.S. Application No. 10/789,953 (Attorney Docket No. 9140.0030-00).	
		Response to Office Action dated December 21, 2006, in U.S. Application No. 11/228,805 (Attorney Docket No. 9140.0030-01).	
<i>[Signature]</i>		Response to Office Action dated January 26, 2007, in U.S. Application No.	

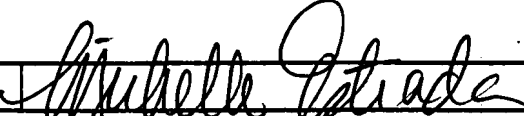
Examiner Signature <i>[Signature]</i>	Date Considered 7/16/07
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

EXPRESS MAIL LABEL NO.
EV 97728687 US

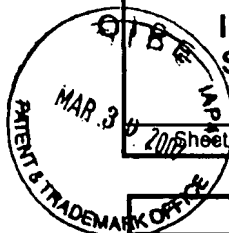
IDS Form PTO/SB/08: Substitute for form 1449A/PTO				Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>				<i>Application Number</i>	10/954,182
				<i>Filing Date</i>	October 1, 2004
				<i>First Named Inventor</i>	ZHANG, Hongmei
				<i>Art Unit</i>	2823
				<i>Examiner Name</i>	ESTRADA, Michelle
Sheet	2	of	2	<i>Attorney Docket Number</i>	9140.0016-01

NON PATENT LITERATURE DOCUMENTS		
	10/851,542 (Attorney Docket No. 9140.0033-00).	
	Notice of Allowance dated February 22, 2007, in U.S. Application No. 10/851,542 (Attorney Docket No. 9140.0033-00).	
	Continuation application and Preliminary Amendment dated December 13, 2006 (Attorney Docket No. 9140.0042-01).	

Examiner Signature		Date Considered	7/16/07
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

EXPRESS MAIL LABEL NO.
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IDS Form PTO/SB/08: Substitute for form 1449A/PTO		Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Use as many sheets as necessary)		Application Number	10/954,182
		Filing Date	October 1, 2004
		First Named Inventor	ZHANG, Hongmei
		Art Unit	2823
		Examiner Name	ESTRADA, Michelle
		Attorney Docket Number	9140.0016-01
Sheet	1	of	1

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner Initials	Cite No. ¹	Document Number	Issue or Publication Date	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)	MM-DD-YYYY		
[Signature]		US-6,391,166 B1	05-21-2002	Wang	
		US-20070053139 A1	03-08-2007	Zhang et al.	


Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

NON PATENT LITERATURE DOCUMENTS			
Examiner Initials	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁹
[Signature]		Response to Office Action dated March 19, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Response to Office Action dated February 20, 2007, in U.S. Application No. 10/650,461 (Attorney Docket No. 9140.0025-00).	
		Office Action dated March 6, 2007, in U.S. Application No. 10/650,461 (Attorney Docket No. 9140.0025-00).	
		Notice of Allowance dated March 26, 2007, in U.S. Application No. 11/228,805 (Attorney Docket No. 9140.0030-01).	
		Supplemental Notice of Allowance dated March 15, 2007, in U.S. Application No. 10/851,542 (Attorney Docket No. 9140.0033-00).	
		Voluntary Amendment dated March 8, 2007, in TW Appl. No. 93114518 (Attorney Docket No. 9140.0033-00270).	
		Application filed March 22, 2007 (Attorney Docket No. 9140.0033-01).	

Examiner Signature	<i>Michelle Estrada</i>	Date Considered	7/16/07
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

EXPRESS MAIL LABEL NO.
EV 977728506 US

Index of Claims 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

✓	Rejected
=	Allowed


-	Cancelled
÷	Restricted

N	Non-Elected
I	Interference

A	Appeal
O	Objected

Claims renumbered in the same order as presented by applicant
 CPA
 T.D.
 R.1.47


CLAIM		DATE							
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Index of Claims 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

✓	Rejected	-	Cancelled	N	Non-Elected	A	Appeal
=	Allowed	÷	Restricted	I	Interference	O	Objected

Claims renumbered in the same order as presented by applicant
 CPA
 T.D.
 R.1.47

CLAIM		DATE							
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Index of Claims 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

✓	Rejected
=	Allowed


-	Cancelled
÷	Restricted

N	Non-Elected
I	Interference

A	Appeal
O	Objected

Claims renumbered in the same order as presented by applicant
 CPA
 T.D.
 R.1.47

CLAIM		DATE							
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Index of Claims 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

✓	Rejected	-	Cancelled	N	Non-Elected	A	Appeal
=	Allowed	÷	Restricted	I	Interference	O	Objected

Claims renumbered in the same order as presented by applicant
 CPA
 T.D.
 R.1.47

CLAIM		DATE							
Final	Original	07/16/2007							
	109	✓							

10-26-07

RCE

REQUEST FOR CONTINUED EXAMINATION (RCE) TRANSMITTAL

Address to:
Mail Stop RCE
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450



Application Number: 10/954,182	Confirmation Number: 9873
Filing Date: October 1, 2004	
First Named Inventor: ZHANG, Hongmei et al.	
Group Art Unit: 2823	
Examiner: ESTRADA, Michelle	
Attorney Docket Number: 10655.0016-01	

JPW

This is a Request for Continued Examination (RCE) under 37 C.F.R. § 1.114 of the above-identified application.
Request for Continued Examination (RCE) practice under 37 C.F.R. § 1.114 does not apply to any utility or plant application filed prior to June 8, 1995, or to any design application.

1. **Submission required under 37 C.F.R. § 1.114: Note: If the RCE is proper, any previously filed unentered amendments and amendments enclosed with the RCE will be entered in the order in which they were filed unless applicant instructs otherwise. If applicant does not wish to have any previously filed unentered amendment(s) entered, applicant must request non-entry of such amendment.**

a. Previously submitted. If a final Office action is outstanding, any amendments filed after the final Office action may be considered as a submission even if this box is not checked.

i. Consider the arguments in the Appeal Brief or Reply Brief previously filed on _____.

ii. Other _____

b. **DO NOT ENTER** the amendment(s) previously filed on _____. An alternate submission is attached.

c. Enclosed submission: .

i. Amendment/Reply

ii. Affidavit(s)/Declaration(s)

iii. Information Disclosure Statement

iv. Other _____

2. Miscellaneous

a. Suspension of action on the above-mentioned application is requested under 37 C.F.R. § 1.103(c) for a period of [number] months. (Period of suspension shall not exceed 3 months; fee under 37 C.F.R. § 1.17(i) required.)

b. Other _____

3. Fees

a. The filing fee is calculated as follows:

i. \$810.00 RCE fee required under 37 C.F.R. § 1.17(e)

ii. Petition for extension of time for (Months) \$

iii. Other _____

b. The Commissioner is authorized to charge the fee of \$810.00 to Deposit Account No. 06-0916.

c. The Commissioner is authorized to charge any deficiencies in the filing fees, or credit any overpayments to Deposit Account 06-0916.

Signature of Applicant, Attorney, or Agent Required

Name: Gary J. Edwards	(650) 849-6622	Reg. No.: 41,008
Signature:		Date: October 24, 2007

EXPRESS MAIL LABEL NO. EM 100825779 US



PATENT
Customer No. 22,852
Attorney Docket No. 10655.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
)	
ZHANG, Hongmei et al.)	Group Art Unit: 2823
)	
Application No.: 10/954,182)	Examiner: ESTRADA, Michelle
)	
Filed: October 1, 2004)	Confirmation No.: 9873
)	
For: BIASED PULSE DC REACTIVE)	
SPUTTERING OF OXIDE FILMS)	

MAIL STOP RCE
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

AMENDMENT

This response is being filed with a Request for Continued Examination and is in reply to the Final Office Action mailed July 24, 2007, the period for response having been extended to October 24, 2007. Applicants amend the application as follows:

Amendments to the Claims are reflected in the listing of claims in this paper beginning on page 2.

Remarks follow the amendment sections of this paper beginning on page 6.

AMENDMENTS TO THE CLAIMS:

This listing of claims will replace all prior versions and listings of claims in the application:

Claims 1-40 (Canceled).

Claim 41 (Previously presented): The method of claim 85 wherein the target is a metallic target and the process gas includes oxygen.

Claim 42 (Previously presented): The method of claim 85 wherein the target is a metallic target and the process gas includes one or more of a set consisting of N₂, NH₃, CO, NO, CO₂, halide containing gasses.

Claim 43 (Previously presented): The method of claim 85 wherein the target is a ceramic target.

Claim 44 (Canceled).

Claim 45 (Previously presented): The method of claim 85 wherein the magnetic field is provided by a moving magnetron.

Claim 46 (Previously presented) The method of claim 85 further including holding the temperature of the substrate substantially constant.

Claim 47 (Previously presented): The method of claim 85 wherein the process gas includes a mixture of Oxygen and Argon.

Claim 48 (Previously presented): The method of claim 85 wherein the Oxygen flow is adjusted to adjust the index of refraction of the film.

Claim 49 (Previously presented): The method of claim 85 wherein the process gas further includes nitrogen.

Claim 50 (Previously presented): The method of claim 85 wherein providing pulsed DC power to a target includes providing pulsed DC power to a target which has an area larger than that of the substrate.

Claim 51 (Previously presented): The method of claim 85, further including uniformly sweeping the target with a magnetic field.

Claim 52 (Previously presented): The method of claim 51 wherein uniformly sweeping the target with a magnetic field includes sweeping a magnet in one direction across the target where the magnet extends beyond the target in the opposite direction.

Claim 53 (Previously presented): The method of claim 85 wherein the target is an alloyed target.

Claim 54 (Previously presented): The method of claim 53 wherein the alloyed target includes one or more rare-earth ions.

Claim 55 (Previously presented): The method of claim 53 wherein the alloyed target includes Si and Al.

Claim 56 (Previously presented): The method of claim 53 wherein the alloyed target includes one or more elements taken from a set consisting of Si, Al, Er, Yb, Zn, Ga, Ge, P, As, Sn, Sb, Pb, Ag, Au, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Tm, and Lu.

Claim 57 (Previously presented): The method of claim 53 wherein the alloyed target is a tiled target.

Claim 58 (Previously presented): The method of claim 57 wherein each tile of the tiled target is formed by prealloy atomization and hot isostatic pressing of a powder.

Claims 59-61 (Canceled).

Claim 62 (Previously presented): A method of depositing a film on a substrate, comprising:

providing a process gas between a conductive target and a substrate;

providing pulsed DC power to the target;

providing a magnetic field to the target; and

reconditioning the target;

wherein reconditioning the target includes reactive sputtering in the metallic mode and then reactive sputtering in the poison mode.

Claims 63-84 (Canceled).

Claim 85 (Previously presented): A method of depositing a film on a substrate, comprising:

providing a process gas between a target and a substrate;

providing pulsed DC power to the target;

providing a magnetic field to the target; and

wherein a material is deposited on the substrate, and an oxide film is formed by reactive sputtering in a mode between a metallic mode and a poison mode.

Claim 86 (Canceled).

Claim 87 (Previously presented): The method according to claims 62 or 85, further including a narrow band-rejection filter that rejects power at an RF frequency, and further including

providing an RF bias to the substrate at the RF frequency.

Claim 88 (Previously presented): The method according to claim 87, wherein the narrow band-rejection filter has a bandwidth of about 100 kHz.

Claim 89 (Previously presented): The method according to claim 87, wherein the RF frequency is about 2 MHz.

Claims 90-109 (Canceled).

REMARKS

Claims 41-43, 45-58, 62, 85, 87-89, and 93-109 are pending in the above-identified application. The Examiner has rejected claims 93-109 and allowed claims 41-43, 45-58, 62, 85 and 87-89. With this response, Applicants have retained claims 41-43, 45-58, 62, 85 and 87-89, and canceled claims 93-109 without prejudice.

Upon entry of the present amendment, only allowed claims 41-43, 45-58, 62, 85 and 87-89 will be pending in this application. Applicants retain the right to file the currently rejected claims, claims 93-109, in a different continuation application.

Claim Rejections Under 35 U.S.C. § 103(a)

Claims 93-109 have been rejected over various combinations of Smolanoff (U.S. Patent No. 6,117,279), Moise et al. (U.S. Publication No. 2001/0034106), Chen (U.S. Publication No. 2004/0077161), and Milonopoulou et al. (U.S. Publication No. 2003/0175142). Without agreeing with or acquiescing in the Examiner's analysis of claims 93-109 and the cited art, Applicants have canceled claims 93-109. Applicants, therefore, request that the Examiner remove this rejection.

Allowable Subject Matter

Applicants thank the Examiner for indicating that claims 41-43, 45-58, 62, 85 and 87-89 are allowable over the prior art.

Conclusion

Applicants respectfully request that this Amendment be entered by the Examiner, placing claims 41-43, 45-58, 62, 85 and 87-89 in condition for allowance, since the final action by the Examiner has indicated that claims 41-43, 45-58, 62, 85 and 87-89 are allowable over the prior art.

In view of the foregoing amendments and remarks, Applicants request the entry of this Amendment, the Examiner's reconsideration and reexamination of the application, and the timely allowance of the pending claims.

Please grant any extensions of time required to enter this response and charge any additional required fees to Deposit Account 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Dated: October 24, 2007

By: 

Gary J. Edwards
Reg. No. 41,008

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PATENT
Customer No. 22,852
Attorney Docket No. 10655.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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ZHANG, Hongmei et al.) Group Art Unit: 2823
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Application No.: 10/954,182) Examiner: ESTRADA, Michelle
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For: BIASED PULSE DC REACTIVE) Confirmation No.: 9873
SPUTTERING OF OXIDE FILMS)

MAIL STOP RCE
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

NINTH SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT
UNDER 37 C.F.R. § 1.97(b)

Pursuant to 37 C.F.R. §§ 1.56 and 1.97(b), Applicants bring to the attention of the Examiner the listed documents on the attached PTO SB/08 Form. This Supplemental Information Disclosure Statement is being filed before the mailing date of a first Office Action after the filing of a Request for Continued Examination in the above-referenced application.

Copies of the listed foreign and non-patent literature documents are attached. A copy of the U.S. patents are not enclosed.

Applicants respectfully request that the Examiner consider the listed documents and indicate that they were considered by making appropriate notations on the attached form.

This submission does not represent that a search has been made or that no better art exists and does not constitute an admission that each or all of the listed documents are material or

constitute "prior art." If the Examiner applies any of the documents as prior art against any claim in the application and Applicants determine that the cited documents do not constitute "prior art" under United States law, Applicants reserve the right to present to the U.S. Patent and Trademark Office the relevant facts and law regarding the appropriate status of such documents.

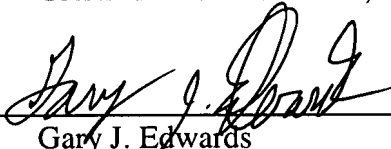
Applicants further reserve the right to take appropriate action to establish the patentability of the disclosed invention over the listed documents, should one or more of the documents be applied against the claims of the present application.

If there is any fee due in connection with the filing of this Statement, please charge the fee to Deposit Account 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Dated: October 24, 2007

By: 
Gary J. Edwards
Reg. No. 41,008

**EXPRESS MAIL LABEL NO.
EM 100825779 US**



IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known		
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>			Application Number	10/954,182	
			Filing Date	October 1, 2004	
			First Named Inventor	ZHANG, Hongmei	
			Art Unit	2823	
			Examiner Name	ESTRADA, Michelle	
Sheet	1	of	2	Attorney Docket Number	10655.0016-01

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner Initials ¹	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
		US-6,391,166 B1	05-21-2002	Wang	
		US-7,262,131 B2	08-28-2007	Narasimhan et al.	

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

FOREIGN PATENT DOCUMENTS						
Examiner Initials ¹	Cite No. ¹	Foreign Patent Document	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁶
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)				
		WO 2007/027535 A2	03-08-2007	Symmorphix, Inc.		

NON PATENT LITERATURE DOCUMENTS			
Examiner Initials ¹	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
		Office Action dated May 21, 2007, in U.S. Application No. 10/291,179 (Attorney Docket No. 9140.0001-00).	
		Final Office Action dated April 13, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Amendment/RCE filed August 9, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Office Action dated September 5, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Final Office Action dated September 7, 2007, in U.S. Application No. 11/100,856 (Attorney Docket No. 9140.0015-01).	
		Final Office Action dated May 2, 2007, in U.S. Application No. 10/101,863 (Attorney Docket No. 9140.0016-00).	
		Response to Final Office Action filed October 2, 2007, in U.S. Application No. 10/101,863 (Attorney Docket No. 9140.0016-00).	
		Response to Office Action filed July 9, 2007, in U.S. Application No. 10/650,461 (Attorney Docket No. 9140.0025-00).	
		Final Office Action dated October 10, 2007, in U.S. Application No. 10/650,461 (Attorney Docket No. 9140.0025-00).	
		Corrected Notice of Allowance dated June 7, 2007, in U.S. Application No. 11/228,805 (Attorney Docket No. 9140.0030-01).	
		Supplemental Notice of Allowance dated July 5, 2007, in U.S. Application No. 11/228,805 (Attorney Docket No. 9140.0030-01).	
		Second Supplemental Preliminary Amendment filed May 31, 2007, in U.S. Application No. 11/297,057 (Attorney Docket No. 9140.0042-00).	

Examiner Signature		Date Considered	
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

**EXPRESS MAIL LABEL NO.
EM 100825779 US**

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
8 March 2007 (08.03.2007)

PCT

(10) International Publication Number
WO 2007/027535 A2

- (51) International Patent Classification:
H01G 4/06 (2006.01)
- (21) International Application Number:
PCT/US2006/033315
- (22) International Filing Date: 24 August 2006 (24.08.2006)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:
11/218,652 2 September 2005 (02.09.2005) US
- (71) Applicant (for all designated States except US): **SYM-MORPHIX, INC.** [US/US]; 1278 Reamwood Avenue, Sunnyvale, California 94089-2233 (US).

(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

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Published:
— without international search report and to be republished upon receipt of that report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.



WO 2007/027535 A2

(54) Title: DEPOSITION OF PEROVSKITE AND OTHER COMPOUND CERAMIC FILMS FOR DIELECTRIC APPLICATIONS

PEROVSKITE	~ 302
SUBSTRATE	~ 301

(57) Abstract: In accordance with the present invention, deposition of perovskite material, for example barium strontium titanite (BST) film, by a pulsed-dc physical vapor deposition process or by an RF sputtering process is presented. Such a deposition can provide a high

deposition rate deposition of a layer of perovskite. Some embodiments of the deposition address the need for high rate deposition of perovskite films, which can be utilized as a dielectric layer in capacitors, other energy storing devices and micro-electronic applications. Embodiments of the process according to the present invention can eliminate the high temperature (>700 °C) anneal step that is conventionally needed to crystallize the BST layer.

Deposition of Perovskite and Other Compound Ceramic Films for Dielectric Applications

FIELD OF THE INVENTION

[001] The present invention is related to production and application of dielectric thin-films and, in particular, the deposition of perovskites such as Barium Strontium Titanate (BST) films and other ceramic oxides for dielectric applications.

DISCUSSION OF RELATED ART

[002] Perovskite films, for example Barium Strontium Titanate (BST) films, are one of the attractive materials to use in capacitors for high density device applications because of its relatively high dielectric constant, low leakage current density, high dielectric breakdown strength, and ferroelectric perovskite phase that does not exhibit fatigue. However, electric properties of the perovskite films are greatly dependent on the deposition process, the substrate, the post-processing, and the related film structure. For all of the potential, thin film perovskites have rarely been utilized in manufacture primarily because of difficulties in controlling physical and chemical properties of the crystalline and amorphous phases of perovskite thin-film materials and their interactions with metallic and conductive electrodes.

[003] Solid-state thin-film devices are typically formed by stacking thin films of metal and dielectric on a substrate. The thin films typically include two metallic electrodes with a dielectric layer in between. The thin films can be deposited utilizing a number of deposition processes, including sputtering, electroplating, chemical vapor deposition, sol gel, or oxidation. Substrates suitable for these applications have conventionally been high temperature materials capable of withstanding at least one high temperature anneal process to at least 650-750 °C so as to crystallize the perovskite dielectric film in order to increase its

dielectric constant. Such a substrate can be any suitable material with appropriate structural and material properties, for example a semiconductor wafer, refractory metallic sheet (e.g., titanium, zirconium, or stainless steel), ceramic such as alumina, or other material capable of withstanding subsequent high temperature processing.

[004] However, conventional materials and production processes can limit the types of materials that can be used in device manufacture. Typically, the dielectric material is deposited in amorphous form and then the material is heated in an anneal process to form the crystalline material. Conventional formation of perovskite layers, for example, require an anneal at or above 650°C to transform the deposited amorphous film to a crystalline form. Such a high temperature anneal, however, severely limits the materials that can be utilized as the substrate, and often requires the use of expensive noble metals such as platinum to protect the substrate from reaction with the electrode material. Such high heat-treat temperatures are incompatible with standard semiconductor or MEM device processing, and limit the choice of substrate materials on which the layers can be formed, increasing the cost, and decreasing the yield of such devices formed with the layers.

[005] Therefore, there is a need for a low temperature process for depositing crystalline material, for example perovskite material and other ceramic oxides, onto a substrate.

SUMMARY

[006] In accordance with the present invention, deposition of layers in a pulsed-DC physical vapor deposition process from a conductive ceramic target is presented. In some embodiments, the deposition can provide a low-temperature, high deposition-rate deposition of a dense amorphous layer of BST from a conductive BST target, which can be annealed at much lower temperature to yield crystalline BST. Some embodiments of the deposition address the need for low temperature, high rate deposition of perovskite films, for example

BST films, which can be utilized as the dielectric layer in high specific capacitance devices as, for example, de-coupling capacitors, energy storage devices, voltage tunable capacitors, or other micro-electronic devices.

[007] A method of depositing a perovskite or ceramic oxide layer according to some embodiments of the present invention includes placing a substrate in a reactor; flowing a gaseous mixture, for example argon and oxygen, through the reactor; and applying pulsed-DC power to a target formed of conductive perovskite or ceramic oxide material, such as BST, positioned opposite the substrate.

[008] In some embodiments the perovskite layer can be formed utilizing radio frequency (RF) sputtering. The perovskite is deposited by RF sputtering of a wide area target in the presence of a sputtering gas under a condition of uniform target erosion. The substrate is positioned opposite a planar target formed of perovskite, for example BST, the area of the target being larger than the area of the substrate. A central area of the target of the same size as the substrate and overlying the substrate is exposed to a uniform plasma condition, which provides a condition of uniform target erosion. A uniform plasma condition can be created without magnetic enhancement, termed diode sputtering, or by providing a time-averaged uniform magnetic field by scanning a magnet across the target in a plane parallel to the plane of the target.

[009] A film produced utilizing a pulsed dc, bias PVD process with a conductive ceramic target can be deposited at much higher rates than an insulating ceramic process, which requires an RF sputtering process. Further, deposition occurs with much less oxygen present in the gas flow to provide a fully oxidized film as opposed to a metallic target. The resulting film is much higher density than the low rate films. The films can be stoichiometric, uniform, highly dense, with low sintering temperatures and resulting high dielectric properties.

[010] In some embodiments, the substrate is preheated. The substrate can be heated to a temperature of about 400°C or below during deposition for low temperature perovskite deposition, or to higher temperatures for perovskite deposition on substrates capable of withstanding such temperature regime. Substrates suitable for low temperature perovskite deposition include glass, plastic, metal foil, stainless steel, and copper. A perovskite layer of thickness up to several microns thick can be deposited, although layers of any thickness can be formed.

[011] In some embodiments the perovskite layer formed on the substrate is later annealed. The anneal temperature can be as low as 400°C for low temperature anneal, and higher for perovskite deposition on substrates capable of withstanding such higher temperature regime. In some embodiments the perovskite target can be doped with transition metal dopants, for example manganese, transition elements, lanthanides (including the rare earth ions) and/or amphoteric elements.

[012] In some embodiments, a stacked capacitor structure can be formed. The stacked capacitor structure includes one or more capacitor stacks deposited on a thin substrate, wherein each capacitor stack includes: a bottom electrode layer, a perovskite, for example BST, dielectric layer deposited over the bottom electrode layer; and a top electrode layer deposited over the dielectric layer. A top conducting layer can be deposited over the capacitor stacks.

[013] In some embodiments, a capacitor structure can be formed in a cluster tool. An exemplary method of producing a capacitor in a cluster tool includes loading a substrate into the cluster tool; depositing an electrode layer over the substrate in a first chamber of the cluster tool; depositing a perovskite dielectric layer over the electrode layer in a second chamber of the cluster tool; depositing a second electrode layer over the dielectric layer in a

third chamber. In some embodiments the first and the second electrode layers can be deposited in the same chamber.

[014] A fixture for holding a thin substrate can include a top portion; and a bottom portion, wherein the thin substrate is held when the top portion is attached to the bottom portion.

[015] In some embodiments, the ceramic layer can be deposited on a substrate coated with iridium or other refractory conductive material to provide a low temperature anneal processed capacitive structure.

[016] These and other embodiments of the invention are further discussed below with reference to the following figures. It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory only and are not restrictive of the invention, as claimed. Further, specific explanations or theories regarding the deposition or performance of materials according to the present invention are presented for explanation only and are not to be considered limiting with respect to the scope of the present disclosure or the claims.

BRIEF DESCRIPTION OF THE DRAWINGS

[017] Figures 1A and 1B illustrate a pulsed-DC biased reactive deposition apparatus that can be utilized in the methods of depositing according to the present invention.

[018] Figure 1C illustrates an RF sputtering deposition apparatus.

[019] Figure 2 shows an example of a target that can be utilized in the reactor illustrated in Figures 1A, 1B, and 1C.

[020] Figures 3A and 3B illustrate a thin-film capacitor design according to some embodiments of the present invention.

[021] Figures 4A, 4B, 4C, and 4D illustrate a thin substrate mount and mask arrangement that can be utilized in the deposition of dielectric perovskite layers, for example BST films, deposited according to some embodiments of the present invention.

[022] Figure 5 illustrates a cluster tool that can be utilized to form batteries with dielectric perovskite layers deposited according to some embodiments of the present invention.

[023] Figure 6 illustrates an example of stacked capacitor structure with dielectric perovskite layers deposited according to some embodiments of the present invention.

[024] In the figures, elements having the same designation have the same or similar functions.

DETAILED DESCRIPTION

[025] In accordance with embodiments of the present invention, dielectric perovskite films or other ceramic oxide films are deposited on a substrate by a pulsed-DC physical vapor deposition (PVD) process utilizing a conductive ceramic target. In some embodiments, the film can be deposited by RF sputtering.

[026] In some embodiments, a dielectric perovskite layer, for example BST material, is deposited directly on the substrate with only low temperature anneal, eliminating the need of a subsequent high temperature anneal to crystallize the film. Removing the high temperature anneal allows for formation of capacitor structures on light-weight, low temperature, and low cost substrates such as copper foil and plastic sheet, reducing both the weight and the cost of capacitors while maintaining the high dielectric constant of the perovskite, for example BST, high-density dielectric film.

[027] Deposition of materials by pulsed-DC, RF biased reactive ion deposition is described in U.S. Patent Application Serial No. 10/101,863, entitled "Biased Pulse DC Reactive Sputtering of Oxide Films," to Hongmei Zhang, et al., filed on March 16, 2002.

Preparation of targets is described in U.S. Patent Application Serial No. 10/101,341, entitled "Rare-Earth Pre-Alloyed PVD Targets for Dielectric Planar Applications," to Vassiliki Milonopoulou, et al., filed on March 16, 2002. U.S. Patent Application Serial No. 10/101,863 and U.S. Patent Application Serial No. 10/101,341 are each assigned to the same assignee as is the present disclosure and each is incorporated herein in their entirety.

Deposition of oxide materials by RF sputtering has also been described in U.S. Patent No. 6,506,289, which is also herein incorporated by reference in its entirety. Transparent oxide films can be deposited utilizing processes similar to those specifically described in U.S. Patent No. 6,506,289 and U.S. Application Serial No. 10/101,863.

[028] Figure 1A shows a schematic of a reactor apparatus 10 for sputtering material from a target 12 according to the present invention. In some embodiments, apparatus 10 may, for example, be adapted from an AKT-1600 PVD (400 X 500 mm substrate size) system from Applied Komatsu or an AKT-4300 (600 X 720 mm substrate size) system from Applied Komatsu, Santa Clara, CA. The AKT-1600 reactor, for example, has three deposition chambers connected by a vacuum transport chamber. These AKT reactors can be modified such that pulsed DC power is supplied to the target and RF power is supplied to the substrate during deposition of a material film.

[029] Apparatus 10 includes target 12, which is electrically coupled through a filter 15 to a pulsed DC power supply 14. In some embodiments, target 12 is a wide area sputter source target, which provides material to be deposited on a substrate 16. Substrate 16 is positioned parallel to and opposite target 12. Target 12 functions as a cathode when power is applied to it from the pulsed DC power supply 14 and is equivalently termed a cathode. Application of power to target 12 creates a plasma 53. Substrate 16 is capacitively coupled to an electrode 17 through an insulator 54. Electrode 17 can be coupled to an RF power supply 18. A magnet 20 is scanned across the top of target 12.

[030] For pulsed reactive DC magnetron sputtering, as performed by apparatus 10, the polarity of the power supplied to target 12 by power supply 14 oscillates between negative and positive potentials. During the positive period, the insulating layer on the surface of target 12 is discharged. To obtain arc free deposition, the pulsing frequency exceeds a critical frequency that can depend on target material, cathode current and reverse time. High quality films can be made using reactive pulse DC magnetron sputtering as shown in apparatus 10.

[031] Pulsed DC power supply 14 can be any pulsed DC power supply, for example an AE Pinnacle plus 10K by Advanced Energy, Inc. With this DC power supply, up to 10 kW of pulsed DC power can be supplied at a frequency of between 0 and 350 kHz. The reverse voltage can be 10% of the negative target voltage. Utilization of other power supplies can lead to different power characteristics, frequency characteristics and reverse voltage percentages. The reverse time on this embodiment of power supply 14 can be adjusted between 0 and 5 μ s.

[032] Filter 15 prevents the RF bias power from power supply 18 from coupling into pulsed DC power supply 14. In some embodiments, power supply 18 can be a 2 MHz RF power supply, for example a Nova-25 power supply made by ENI, Colorado Springs, Co.

[033] In some embodiments, filter 15 can be a 2 MHz sinusoidal band rejection filter. In some embodiments, the band width of the filter can be approximately 100 kHz. Filter 15, therefore, prevents the 2 MHz power from the bias to substrate 16 from damaging power supply 14 while allowing the full bandwidth of the pulsed DC power supply to pass filter 15.

[034] Pulsed DC deposited films are not fully dense and may have columnar structures. Columnar structures can be detrimental to thin film applications such as barrier films and dielectric films, where high density is important, due to the boundaries between the

columns. The columns act to lower the dielectric strength of the material, but may provide diffusion paths for transport or diffusion of electrical current, ionic current, gas, or other chemical agents such as water.

[035] In the AKT-1600 based system, for example, target 12 can have an active size of about 675.70 X 582.48 by 4 mm in order to deposit films on substrate 16 that have dimension about 400 X 500 mm. The temperature of substrate 16 can be adjusted to between -50 °C and 500 °C. The distance between target 12 and substrate 16 can be between about 3 and about 9 cm. Process gas can be inserted into the chamber of apparatus 10 at a rate up to about 200 sccm while the pressure in the chamber of apparatus 10 can be held at between about .7 and 6 milliTorr. Magnet 20 provides a magnetic field of strength between about 400 and about 600 Gauss directed in the plane of target 12 and is moved across target 12 at a rate of less than about 20-30 sec/scan. In some embodiments utilizing the AKT 1600 reactor, magnet 20 can be a race-track shaped magnet with dimensions about 150 mm by 600 mm.

[036] In some embodiments of the present invention a perovskite layer is deposited by RF sputtering with a wide area target and a condition of uniform target erosion. An example apparatus 30 for RF sputtering is illustrated schematically in FIG. 1C. Apparatus 30 includes an RF power supply 60 coupled to wide area sputter source target 12 which provides material to be deposited on substrate 16. Substrate 16 is positioned parallel to and opposite target 12. Target 12 functions as a cathode when RF power is applied to it and is equivalently termed the cathode. In the present disclosure, target 12 can be formed from a perovskite material, for example BST, for deposition of dielectric perovskite film. Substrate 16 is a solid, smooth surface. Substrate 16 typically is supported on a holder or carrier sheet 17 that may be larger than substrate 16.

[037] In some embodiments, a feature of the RF sputtering method is that the area of wide area target 12 is greater than the area on the carrier sheet on which physically and

chemically uniform deposition is accomplished. Secondly, a central region on target 12, overlying the substrate 16, can be provided with a very uniform condition of sputter erosion of the target material. Uniform target erosion is a consequence of a uniform plasma condition. In the following discussion, all mention of uniform condition of target erosion is taken to be equivalent to uniform plasma condition. Uniform target erosion is evidenced by the persistence of film uniformity throughout an extended target life. A uniform deposited film is defined as a film having a nonuniformity in thickness, when measured at representative points on the entire surface of a substrate wafer, of less than about 5%. Thickness nonuniformity is defined, by convention, as the difference between the minimum and maximum thickness divided by twice the average thickness. If films deposited from a target from which more than about 20% of the weight of the target has been removed under constant process conditions continue to exhibit thickness uniformity, then the sputtering process is judged to be in a condition of uniform target erosion for all films deposited during the target life.

[038] Thus, a uniform plasma condition can be created in the region between the target and the substrate overlying the substrate. The region of uniform plasma condition is indicated in the exploded view of FIG. 1B. A plasma is created in the region denoted 51, which extends under the entire target 12. The central region of the target 52 experiences the condition of uniform sputter erosion. As discussed further below, a layer deposited on a substrate placed anywhere below central region 52 will have uniform film thickness.

[039] In addition, the region in which deposition provides uniform film thickness is larger than the area in which deposition provides a film with uniform physical or optical properties such as chemical composition or index of refraction. In the present invention the target can be planar or approximately planar for the formation of a film on a planar substrate which is to be coated with the material of the target. In practice, planarity of the target means

that all portions of the target surface in region 52 are within a few millimeters of an ideal planar surface, typically within 0.5 mm.

[040] Figure 2 illustrates an example of target 12. A film deposited on a substrate positioned on carrier sheet 17 directly opposed to region 52 of target 12 has good thickness uniformity. Region 52 is the region shown in Figure 1B that is exposed to a uniform plasma condition. In some implementations, carrier 17 can be coextensive with region 52. Region 24 shown in Figure 2 indicates the area below which both physically and chemically uniform deposition can be achieved, for example where physical and chemical uniformity provide refractive index uniformity. Figure 2 indicates region 52 of target 12 that provides thickness uniformity, which is, in general, larger than region 24 of target 12 providing thickness and chemical uniformity to the deposited film. In optimized processes, however, regions 52 and 24 may be coextensive.

[041] In some embodiments, magnet 20 extends beyond area 52 in one direction, for example the Y direction in Figure 2, so that scanning is necessary in only one direction, for example the X direction, to provide a time averaged uniform magnetic field. As shown in Figures 1A and 1B, magnet 20 can be scanned over the entire extent of target 12, which is larger than region 52 of uniform sputter erosion. Magnet 20 is moved in a plane parallel to the plane of target 12.

[042] The combination of a uniform target 12 with a target area 52 larger than the area of substrate 16 can provide films of highly uniform thickness. Further, the material properties of the film deposited can be highly uniform. The conditions of sputtering at the target surface, such as the uniformity of erosion, the average temperature of the plasma at the target surface and the equilibration of the target surface with the gas phase ambient of the process are uniform over a region which is greater than or equal to the region to be coated with a uniform film thickness. In addition, the region of uniform film thickness is greater

than or equal to the region of the film which is to have highly uniform optical properties such as index of refraction, density, transmission, or absorption.

[043] In the present disclosure, target 12 can be formed from perovskite material, such as BST, for deposition of dielectric perovskite film. In some embodiments of the present invention the perovskite target is doped with transition metal dopants, for example Manganese, transition elements, lanthanides (including the rare earth ions) and/or amphoteric elements. In some embodiments of the present invention the percentage of the dopant in the perovskite target is from 0.1 to several percent.

[044] In some embodiments of the invention, material tiles are formed. These tiles can be mounted on a backing plate to form a target for apparatus 10. A wide area sputter cathode target can be formed from a close packed array of smaller tiles. Target 12, therefore, may include any number of tiles, for example between 2 and 20 individual tiles. Tiles can be finished to a size so as to provide a margin of non-contact, tile to tile, less than about 0.010" to about 0.020" or less than half a millimeter so as to eliminate plasma processes that may occur between adjacent ones of tiles 30. The distance between tiles of target 12 and the dark space anode or ground shield 19 in Figure 1B can be somewhat larger so as to provide non contact assembly or to provide for thermal expansion tolerance during process chamber conditioning or operation.

[045] As shown in Figure 1B, a uniform plasma condition can be created in the region between target 12 and substrate 16 in a region overlying substrate 16. A plasma 53 can be created in region 51, which extends under the entire target 12. A central region 52 of target 12 can experience a condition of uniform sputter erosion. As discussed further below, a layer deposited on a substrate placed anywhere below central region 52 can then be uniform in thickness and other properties (i.e., dielectric, optical index, or material concentrations). In addition, in region 52 the deposition provides uniformity of deposited film that can be larger

than the area in which the deposition provides a film with uniform physical or optical properties such as chemical composition or index of refraction. In some embodiments, target 12 is substantially planar in order to provide uniformity in the film deposited on substrate 16. In practice, planarity of target 12 can mean that all portions of the target surface in region 52 are within a few millimeters of a planar surface, and can be typically within 0.5 mm of a planar surface.

[046] Reactive gases that provide a constant supply of oxygen to keep the target surface oxidized can be provided to expand the process window. Some examples of the gases that can be utilized for controlling surface oxidation are O₂, water vapor, hydrogen, N₂O, fluorine, helium, and cesium. Additionally, a feedback control system can be incorporated to control the oxygen partial pressure in the reactive chamber. Therefore, a wide range of oxygen flow rates can be controlled to keep a steady oxygen partial pressure in the resulting plasma. Other types of control systems such as target voltage control and optical plasma emission control systems can also be utilized to control the surface oxidation of the target. In some embodiments, power to target 12 can be controlled in a feedback loop at supply 14. Further, oxygen partial pressure controller 20 can control either oxygen or argon partial pressures in plasma 53. In some embodiments of the present invention, oxygen flow or partial pressure can be utilized to maintain a constant voltage of discharge from target 12.

[047] Figures 3A and 3B show a capacitor structure with a dielectric perovskite layer deposited according to some embodiments of the present invention. As shown in Figure 3A, a dielectric perovskite layer 302 is deposited on a substrate 301. In some embodiments, the dielectric layer 302 can be patterned in various ways before deposition of a substrate 301. In some embodiments, a first electrode layer 303 can be deposited on the substrate and the dielectric layer 302 is deposited over the first electrode layer. The second electrode layer 304 is then deposited over the dielectric layer 302. In some embodiments of

the invention, the dielectric perovskite layer 302 is crystalline and has sufficiently high dielectric constant without the necessity of a high temperature anneal. Therefore, substrate 301 can be a silicon wafer, titanium metal, alumina, or other conventional high temperature substrate, but may also be a low temperature material such as plastic, glass, or other material that may be susceptible to damage from the high temperature anneal. This feature can have the great advantage of decreasing the expense and weight of capacitor structures formed by the present invention. The low temperature deposition of perovskite material allows for successive depositions of perovskite and electrode layers, one upon another. Such a process would have the advantage that successive layers of capacitor structure would be obtained in a stacked condition without the inclusion of a substrate layer. The stacked layered capacitor would provide higher capacitance and higher energy storage than single layer devices with a smaller surface area. Additionally, a capacitor with a lower inductance can be obtained.

[048] In accordance with the present invention, perovskite films can be deposited on substrate 302 with a pulsed-DC biased PVD system as was described above. In particular, an AKT 1600 PVD system can be modified to provide an RF bias and an Advanced Energy Pinnacle plus 10K pulsed DC power supply can be utilized to provide power to a target. The pulsing frequency of the power supply can vary from about 0 to about 350 KHz. The power output of the power supply is between 0 and about 10 kW.

[049] A target of Barium Strontium Titanate with resistivity in the range of less than about megaohms can be utilized with high rate pulsed-dc sputtering. As discussed above, the target can be mounted on a monolithic backing plate as described in U.S. Provisional Application {Attorney Docket No. 09140.6013}, filed on August 26, 2005, which is also herein incorporated by reference in its entirety.

[050] In general, target 12 can be a dielectric material having a resistivity of less than about a megaohm, and therefore can be described as a conducting ceramic target. Target

12, which is formed of a dielectric perovskite material that may not be inherently conducting, is made conducting by formulation so as to contain an excess of metallic composition or by addition of a dopant that provides sufficient conductivity. Examples of suitable dopants include boron, antimony, arsenic, phosphorous, or other dopants. In the example of a BST target, the sintering process can be conducted in the presence of a reducing ambient to achieve a sufficiently conductive target material. Utilization of a conducting ceramic target material can be sputtered at high rates utilizing reactive pulsed-DC techniques so as to form dense stoichiometric dielectric films.

[051] Gas flows containing Oxygen and Argon can be utilized. In some embodiments, the Oxygen to Argon ratio ranges from 0 to about 50% with a total gas flow of between about 60 to about 80 sccm. The pulsing frequency ranges from about 200 kHz to about 350 kHz during deposition. RF bias can also be applied to the substrate. In many trials, the deposition rates varied from about 2 Angstrom/(kW sec) to about 1 Angstrom/(kW sec) depending on the O₂/Ar ratio as well as substrate bias.

[052] Figure 3A illustrates a layer of perovskite material 302 deposited on a thin substrate 301 according to some embodiments of the present invention. Substrate 301 can be formed of a thin metallic sheet (e.g., copper, titanium, stainless steel, or other suitable thin metallic sheet), can be formed of a high temperature plastic material, or may be formed of a ceramic, glass, or polymer material.

[053] Depositing materials on a thin substrate involves holding and positioning the substrate during deposition. Figures 4A, 4B, 4C, and 4D illustrate a reusable fixture 400 for holding a thin film substrate. As shown in Figure 4A, reusable fixture 400 includes a top portion 401 and a bottom portion 402 that are fastened together to secure the substrate. Thin substrate 301 is positioned between top portion 401 and bottom portion 402. As shown in Figure 7B, top portion 701 and bottom portion 702 are such that substrate 301 is brought into

a planar condition and subsequently clamped as top portion 401 is closed into bottom portion 402. Substrate 301 can be easily held by fixture 400 so that substrate 301 can be handled and positioned. In some embodiments, the corners of substrate 301, areas 403, are removed so that substrate 301 is more easily stretched by avoiding "wrap-around" corner clamping effects when top portion 401 is closed into bottom portion 402.

[054] As shown in Figure 4C, a mask 412 can be attached to fixture 400. In some embodiments, fixture 400 includes guides in order to align fixture 400 with respect to mask 412. In some embodiments, mask 412 may be attached to fixture 400 and travel with fixture 400. Mask 412 can be positioned at any desired height above substrate 301 in fixture 400. Therefore, mask 412 can function as either a contact or proximity mask. In some embodiments, mask 412 is formed of another thin substrate mounted in a fixture similar to fixture 400.

[055] As shown in Figure 4C and 4D, fixture 400 and mask 412 can be positioned relative to mount 410. Mount 410, for example, can be a susceptor, mount, or an electrostatic chuck of a processing chamber such as that shown in Figures 1A and 1B. Fixture 400 and mask 412 can have features that allow for ready alignment with respect to each other and with respect to mount 410. In some embodiments, mask 412 is resident in the processing chamber and aligned with fixture 400 during positioning of fixture 400 on mount 410, as shown in Figure 4D.

[056] Utilizing fixture 400 as shown in Figures 4A, 4B, 4C, and 4D allows processing of a thin film substrate in a processing chamber. In some embodiments, thin film substrates can be about 1 μm or more. Further, thin film substrate 301, once mounted within fixture 400, can be handled and moved from process chamber to process chamber. Therefore, a multiprocessor chamber system can be utilized to form stacks of layers,

including one or more layers of perovskite film deposited according to embodiments of the present invention.

[057] Figure 5 illustrates a cluster tool 500 for processing thin film substrates. Cluster tool 500 can, for example, include load lock 502 and load lock 503, through which mounted thin film substrate 301 is loaded and a resultant device is removed from cluster tool 500. Chambers 504, 505, 506, 507, and 508 are processing chambers for depositions of materials, heat treatments, etching, or other processes. One or more of chambers 504, 505, 506, 507, and 508 can be a pulsed-DC or RF PVD chamber such as discussed above with respect to Figures 1A, 1B, and 1C and within which a dielectric perovskite film may be deposited according to embodiments of the present invention.

[058] Processing chambers 504, 505, 506, 507, and 508 as well as load locks 502 and 503 are coupled by transfer chamber 501. Transfer chamber 501 includes substrate transfer robotics to shuttle individual wafers between processing chambers 504, 505, 506, 507, and 508 and load locks 502 and 503.

[059] In production of a thin-film capacitor, substrates are loaded into load lock 503. An electrode layer can be deposited in chamber 504, followed by a perovskite deposition performed in chamber 505. The substrate can then be removed through load lock 503 for an in-air heat treatment external to cluster tool 500. The treated wafer can then be reloaded into cluster tool 500 through load lock 502. The wafer can then again be removed from cluster tool 500 for deposition of a second electrode layer, or sometimes chamber 506 can be adapted to deposition of the second electrode layer. The process can be repeated to form a capacitor stack. The finished capacitor structure is then off-loaded from cluster tool 500 in load lock 502. Wafers are shuttled from chamber to chamber by robotics in transfer chamber 501.

[060] A capacitor structure produced according to the present invention could utilize thin film substrates loaded in a fixture such as fixture 400. Fixture 400 is then loaded into

load lock 503. Chamber 504 may still include deposition of the electrode layer. Chamber 505 then includes deposition of a perovskite layer according to embodiments of the present invention. A second electrode layer can then be deposited in chamber 506. In this process, only low temperature anneal is utilized to increase crystallinity and the dielectric constant of the perovskite layer.

[061] Another advantage of a thin film capacitor process is the ability to stack capacitor structures. In other words, substrates loaded into cluster tool 500 may traverse process chambers 504, 505, 506, 507, and 508 multiple times in order to produce multiply stacked capacitor structures. Figures 6A and 6B illustrate such structures.

[062] Figure 6A illustrates a parallel coupled stacking. As shown in Figure 6A, a substrate 301, which for example can be a high temperature plastic substrate, such as polyimide, is loaded into load lock 503. Electrode layer 303, for example, can be deposited in chamber 504. A dielectric perovskite layer 302 is then deposited on electrode layer 303. Perovskite layer 302 can be about 0.1 to 1 μm and can be deposited in chamber 505 according to embodiments of the present invention. The wafer can then be moved to chamber 506 where the next electrode layer 304 of thickness of about 0.1 μm or more is deposited. A second capacitor stack can then be deposited over the first capacitor stack formed by first electrode layer 303, perovskite layer 302, and second electrode layer 304. This capacitor stack includes second perovskite layer 305 and third electrode layer 306. In some embodiments, further stacks can be formed. In some embodiments, metal layers 303, 304, and 306 differ in the mask utilized in deposition so that tabs are formed for electrical coupling of layers.

[063] As discussed above, any number of individual capacitor stacks can be formed such that parallel capacitor formations are formed. Such a parallel arrangement of capacitor

stacking structure can be formed of alternating layers of electrode and perovskite dielectric layers and can have any number of dielectric layers.

[064] To form the structures shown in Figure 6, substrates are rotated again through the chambers of cluster tool 500 in order to deposit the multiple sets of capacitors. In general, a stack of any number of capacitors can be deposited in this fashion.

[065] Tables I and II illustrate some examples depositions of perovskite material, for example BST, according to the present invention. In these examples, the BST film is deposited using an AKT-1600 PVD (400 X 500 mm substrate size) system from Applied Komatsu. The power supply is an ENI 13.56 MHz RF power supply with a ENI matchbox. The target material is BST with resistivity in the range of $k\Omega$ s or less. The target material can, for example, be sintered. Silicon wafers are used for initial experiments. 0.1-1 microns of BST films are deposited on Si wafers with various bottom electrode materials such as: n++ Si, Ir, Pt, IrO_2 and also Ti_4O_7 , Ti_3O_5 , Nb, Os. The Oxygen to Argon ratio ranges from 0 to 50%. Process pressure ranges from 3-10 mT. RF bias is applied to substrates for some of the examples. The dielectric constant of as deposited film range from 13 to 123 and increases after post-deposition anneal to more than 1000.

[066] One skilled in the art will recognize variations and modifications of the examples specifically discussed in this disclosure. These variations and modifications are intended to be within the scope and spirit of this disclosure. As such, the scope is limited only by the following claims.

TABLE I

Example #	Film Thickness (nm)	Target Power (W) _r	Bias Power (W)	Ar/O ₂ Ratio	V _{bd}	E _{bd}	C (PF)	Dielectric Constant
BST 2	3679	1500	100	50/50	157	4.267464	167	13.35
BST 3	3736	1500	100	50/50	150	4.014989	168	13.64
BST3-N++ 550c	3736	1500	100		40	1.070664	1670	135.57
BST-Pt-1	2282	1500	100	50/25	47	2.059597	299.5	14.85
BST Pt-1 550C	2282	1500	100		16	0.701139	5722	283.74
BST-n++-4	2282	1500	100	50/25	120	5.258545	274	13.59
BST-n++550c	2282	1500	100		30	1.314636	1970	97.69
BST-IrO2-1	2310	1500	100	50/25	100	4.329004	296.2	14.87
BST-IrO2-1 750C	2310	1500	100	50/25	2.4	0.103896	17700	888.46
BST-Pt-2	2310	1500	100	50/25	100	4.329004	319	16.01
BST-Pt-2 650C	2310	1500	100		9.4	0.406926	9750	489.41
BST-Pt-3	2199	1500	100	75/25	7	0.318327	2580	123.28
BST-Pt-3 550	2199	1500	100	75/25	11.2	0.509322	10740	513.20
BST IrO2-2	2199	1500	100	75/25	16.7	0.759436	378	18.06
BST IrO2-2 550	2199	1500	100	75/25	1.4	0.063665	10400	496.95
BST IrO2-2 650	2199	1500	100	75/25	6.9	0.313779	11000	525.62
BST IrO2-2 750	2199	1500	100	75/25	1.4	0.063665	21950	1048.85
BST Pt 1 step	2918	2000	0	50/50			1239	78.56
BST Ir 1 step	2918	2000	0	50/50			1180	74.82
BST IrO2 1 step	2918	2000	0	50/50			567	35.95
BST Pt 2 steps	1689	2000	0	100/0-50/50			1220	44.78
BST Ir 2 steps	1689	2000	0	100/0-50/50			1230	45.14
BST IrO2 2 steps	1689	2000	0	100/0-50/50			684	25.10

Table II

Sample#	thickness	target power	bias power	Ar/O2	dep time (sec)	Vbd	Ebd	C(PF)	Dielectric Constant, k	Vbd (V)	C(PF)	Dielectric Constant, k (after 500C ^o anneal)
ALDOEN+-1	840.4					70	8.329367	380	6.939418			
	840.4							377	6.884633			
ALDOEN+-2	5767.2							60.5	7.581825			
experiment 109	1000							1200	0			
	840							1200	26.07562			
ebonex_BST (A)	1140	900	100		3600	75	8.928571	405	7.392438			
Ir Coated #2	2220	900	0	50/50	5400		0	508	0		16800	810.4302
	2220								24.50587		5000	
	2220										21530	1038.605
IrO2 Coated #2	2220	900	0	50/50	5400		0	365	17.60756	9	22000	1061.278
	2220						0				20000	964.7979
											19000	
											9000	
N++(1)	2220	900	0		5400	12	0.540541	290	13.98957	12	2512	121.1786
N++(2)	840	900	100	50/50	5400	10	1.190476	982	17.92438	10	2675	48.8266
N++(3)	can't meas	900	200		5400			377				
N++(4)	1490	900	100	50/25	5400		0	242	7.835289		537	17.38657
Ti4O7 (A)	910	900		50/50			0	3030	59.91525		450	8.898305
	910							2962	58.57062			
	910							2860	56.55367			
Ti4O7 (B)	1490			50/25			0	1988	64.36593		314	10.16645
	1490							2048	66.30856			
Ir # 3	650	900	1000 sec no bias/ 4400sec bias 80W	50/50	5400							very leaky

Ir #4	870	900	500 sec no bias/ 4900 sec 50w bias	50/50	5400														
Ir #5	2000	900	1500sec no bias/5700 sec 50W bias	50/50	7200														
IrO2 (Tsub=450c)	2000	900	no bias	50/50	5400	0.5	1390	60.40852	10	5972	259.5393								
	2000									6021	261.6688								
n++ (6)(Tsub=450c)	934	900	75 w bias	50/50	7200	1.284797	870	17.65711	12	2857	57.98431								
n++ (7) (room)	2541	900	no bias	50/50	5400		219		13	2210	122.0254								
n++(8) (room)	2504	900	75 w bias	50/50	7200		224	12.18809	22	2218	120.6839								
n++ (9) (room)	10000		75 w bias	50/50	28800		58	12.60322	55	954.7	207.4533								
n++ (10) (room)	5000		75w bias	50/50	14400														

WHAT IS CLAIMED IS:

1. A method of depositing a perovskite layer on a substrate, comprising:
 - placing the substrate into a reactor;
 - flowing a gaseous mixture through the reactor; and
 - providing power to a target formed of a perovskite material positioned opposite the substrate.
2. The method of claim 1, wherein providing power to the conducting target includes applying pulsed-DC power to the conducting target.
3. The method of claim 2 further including filtering the pulsed-DC power to protect a pulsed DC power supply from a bias power while allowing passage of the pulsed DC power through the filter.
4. The method of claim 2, further including supplying an RF bias power to the substrate.
5. The method of claim 1, wherein providing power to the conducting target includes applying RF power to the conducting target.
6. The method of claim 1, wherein a perovskite layer is formed on the substrate.
7. The method of claim 6, wherein the perovskite layer is a barium strontium titanite (BST) layer.
8. The method of claim 6, wherein the formed perovskite layer is more than about 0.1 micron thick.
9. The method of claim 6 wherein the formed perovskite layer is less than about 1 micron thick.
10. The method of claim 6, further comprising annealing the perovskite layer formed on the substrate.

11. The method of claim 10 wherein annealing the perovskite layer includes heating the perovskite layer to an anneal temperature of between about 500°C and about 800°C.
12. The method of claim 1, further comprising preheating the substrate before applying power to the conducting target.
13. The method of claim 12, wherein preheating the substrate including heating the substrate to a temperature of about 400 °C for low temperature perovskite deposition.
14. The method of claim 1, wherein the substrate is a low temperature substrate.
15. The method of claim 14, wherein the low temperature substrate is one of a set of substrates including glass, plastic, metal foil, copper, and stainless steel.
16. The method of claim 1 wherein the conducting target is doped with a transition metal dopant, transition element, lanthanide, and/or amphoteric elements.
17. The method of claim 16 wherein the target is doped with Manganese.
18. The method of claim 17 wherein a level of Manganese in the target is at least 0.1%.
19. The method of claim 1, wherein the perovskite target is a conductive target.
20. A capacitor structure, comprising:
 - a first conducting electrode layer;
 - a dielectric perovskite layer deposited over the first conducting electrode layer; and
 - a second conducting electrode layer deposited over the dielectric perovskite layer.
21. The capacitor of claim 20, wherein the first conducting layer is a copper sheet.
22. A stacked capacitor structure, comprising:

one or more capacitor stacks deposited on a substrate, wherein each capacitor stack comprises:

a bottom electrode layer,

a dielectric perovskite layer deposited over the electrode layer, and

a top electrode layer deposited over the one or more capacitor stacks.

23. The stacked capacitor structure of claim 22, wherein the capacitor stacks form a parallel stacked capacitor structure.
24. The stacked capacitor structure of claim 22, wherein the capacitor stacks form a series stacked capacitor structure.
25. A method of producing a capacitor, comprising:
 - loading a substrate into a cluster tool;
 - depositing a dielectric perovskite layer over a substrate in a chamber of the cluster tool.
26. The method of claim 25, wherein depositing the dielectric perovskite layer includes depositing perovskite film with a pulsed-DC PVD process.
27. The method of claim 25, wherein depositing the dielectric perovskite layer includes depositing perovskite film with an RF sputtering PVD process.
28. The method of claim 25, wherein depositing the dielectric perovskite layer includes depositing the perovskite material through a mask.
29. The method of claim 25, further including
 - depositing a bottom electrode layer on the substrate wherein the dielectric perovskite layer is deposited over the bottom electrode layer.
31. The method of claim 25, further including depositing a top electrode layer over the dielectric perovskite layer.
32. A fixture for holding a thin substrate, comprising:

a top portion; and
a bottom portion, wherein
the thin substrate is held when the top portion is attached to the bottom
portion.

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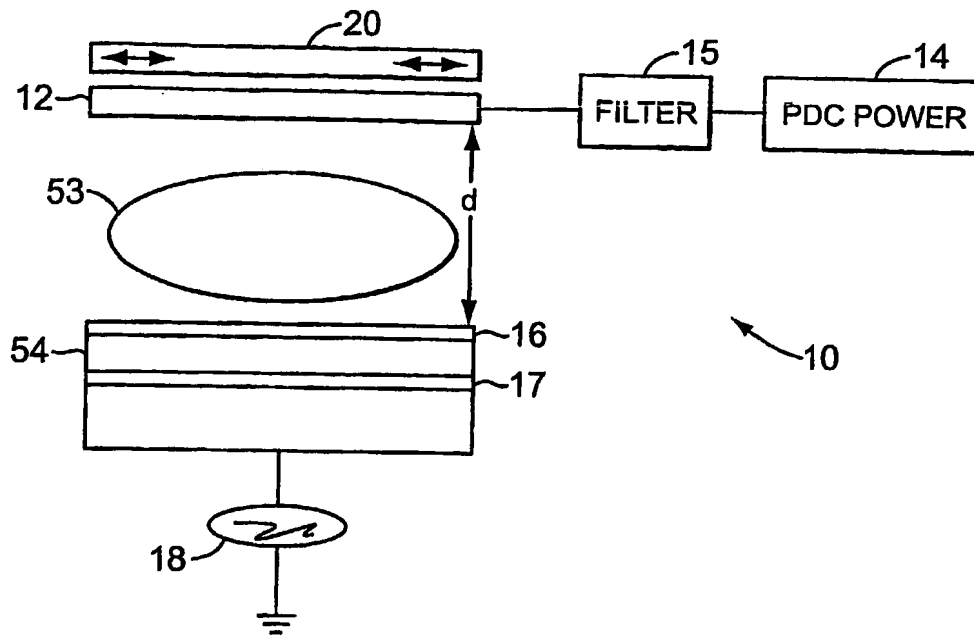


FIG. 1A

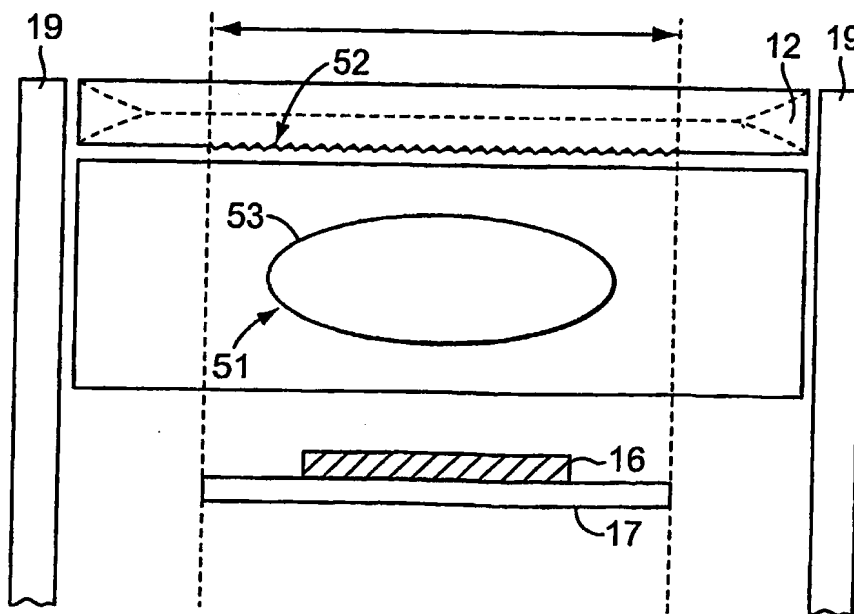


FIG. 1B

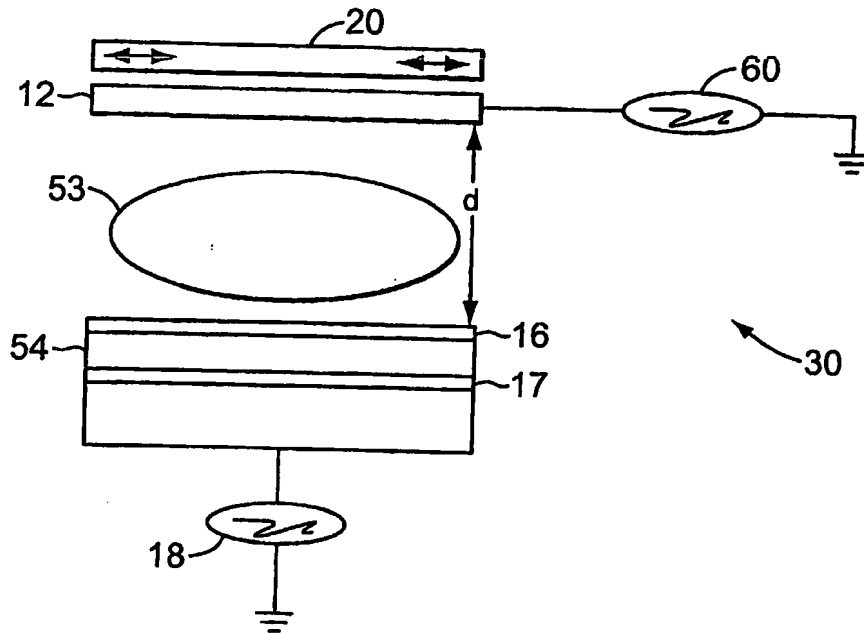


FIG. 1C

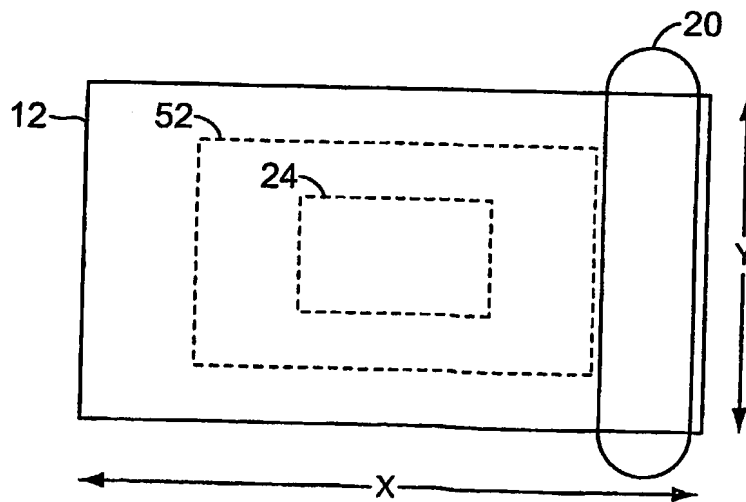


FIG. 2

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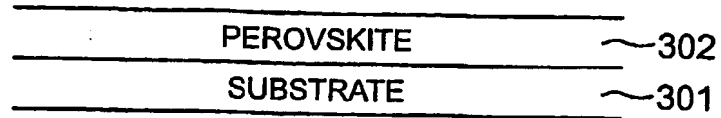


FIG. 3A

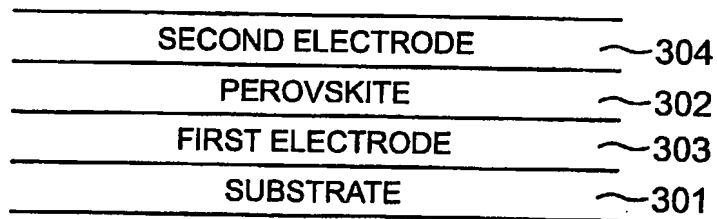


FIG. 3B

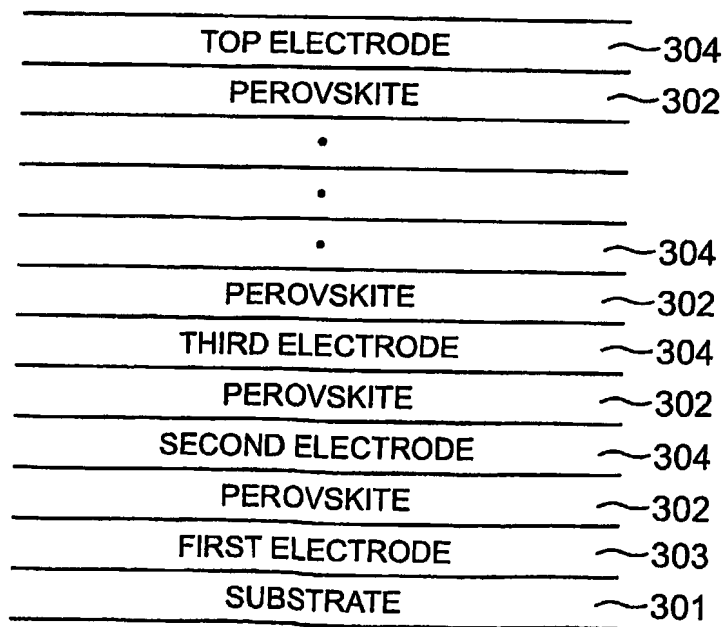


FIG. 6

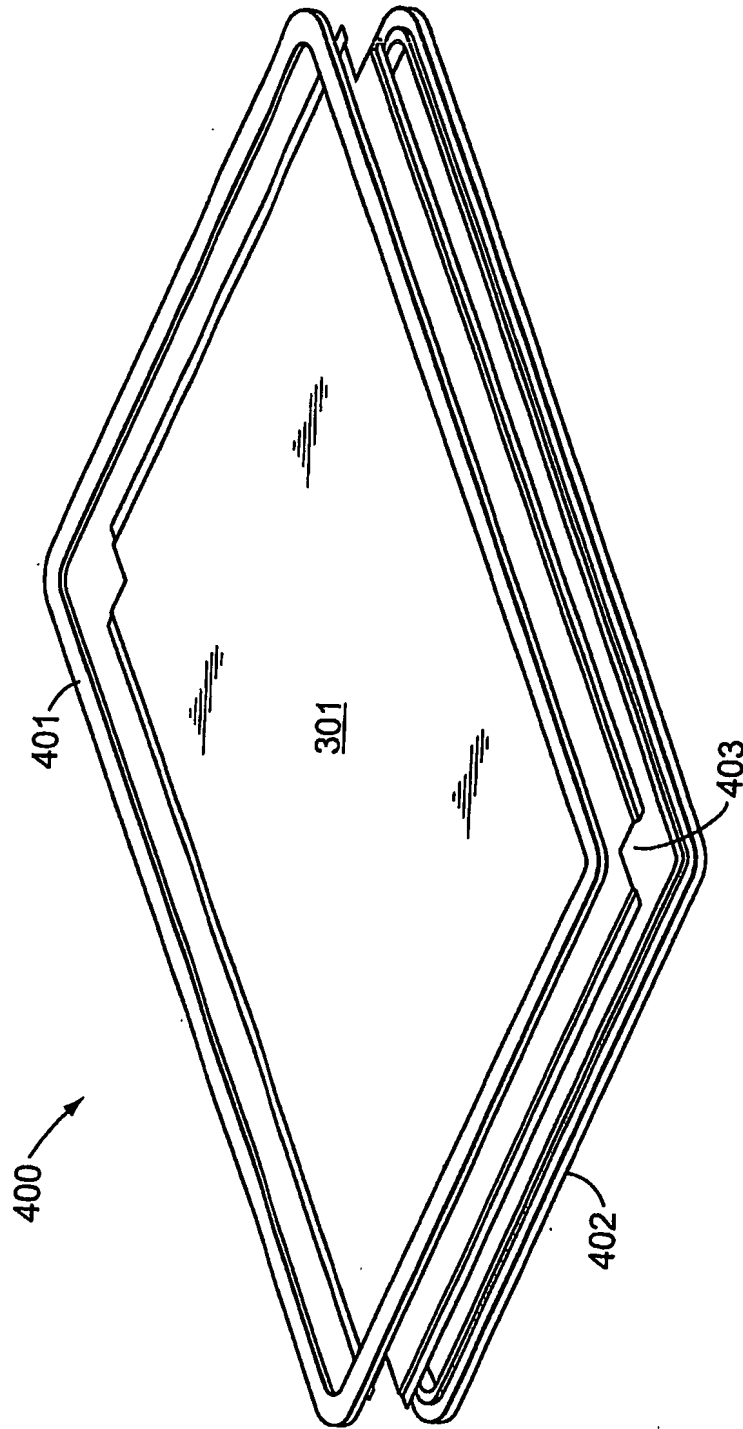


FIG. 4A

5/8

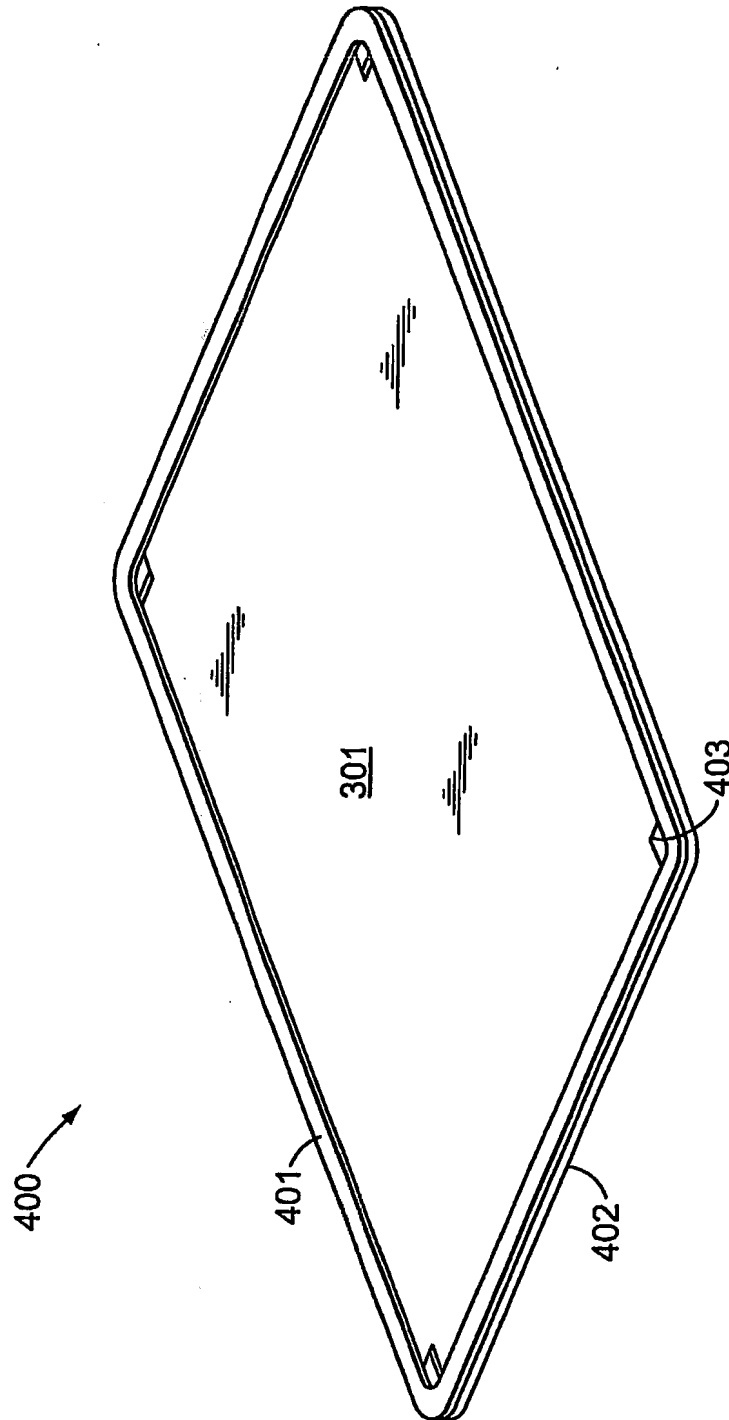


FIG. 4B

6/8

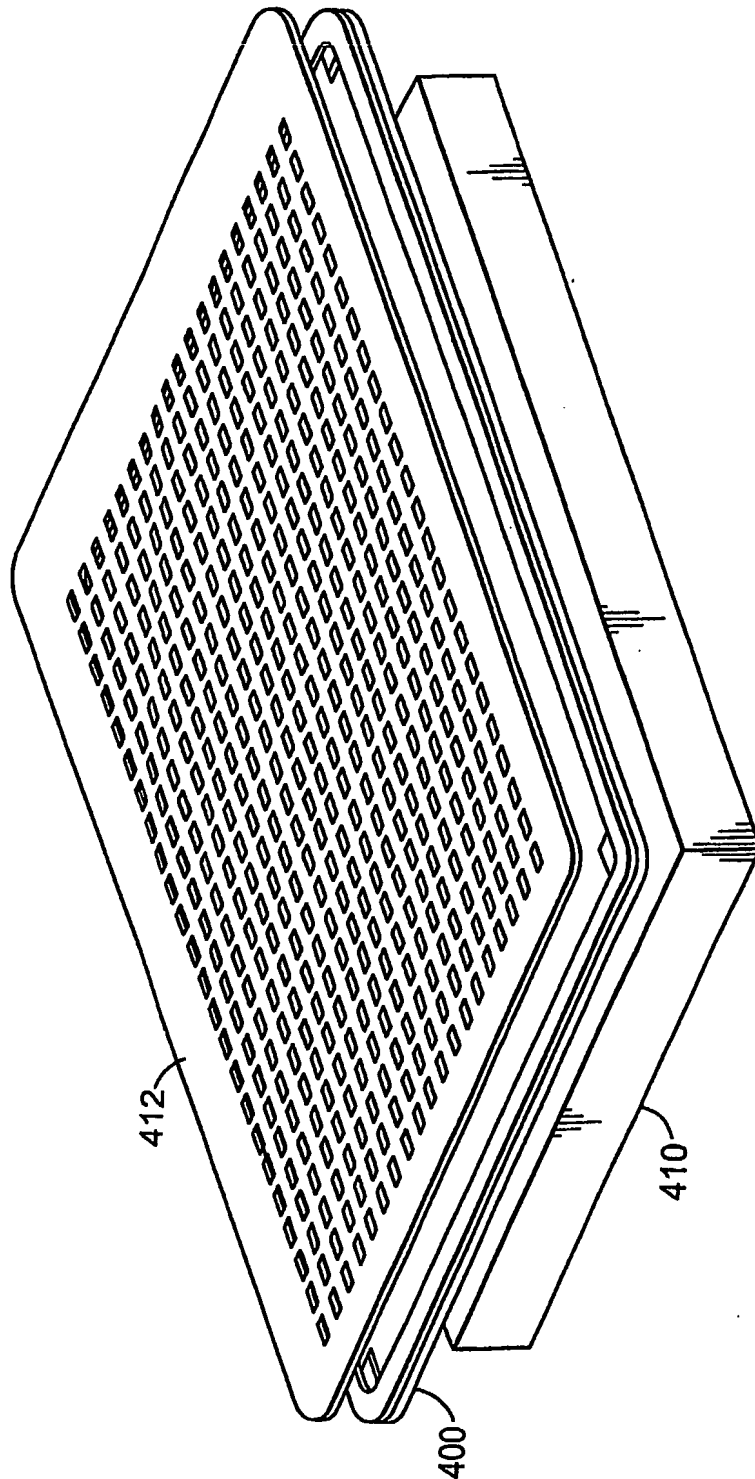


FIG. 4C

7/8

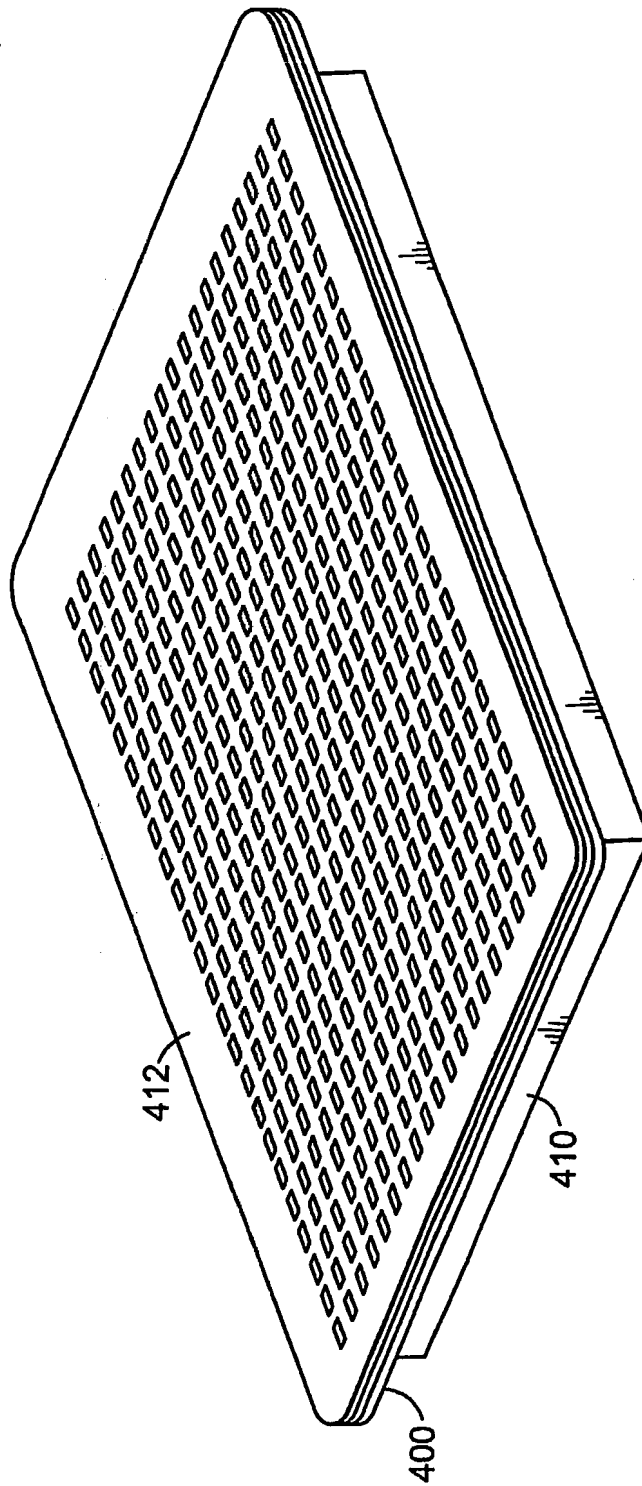


FIG. 4D

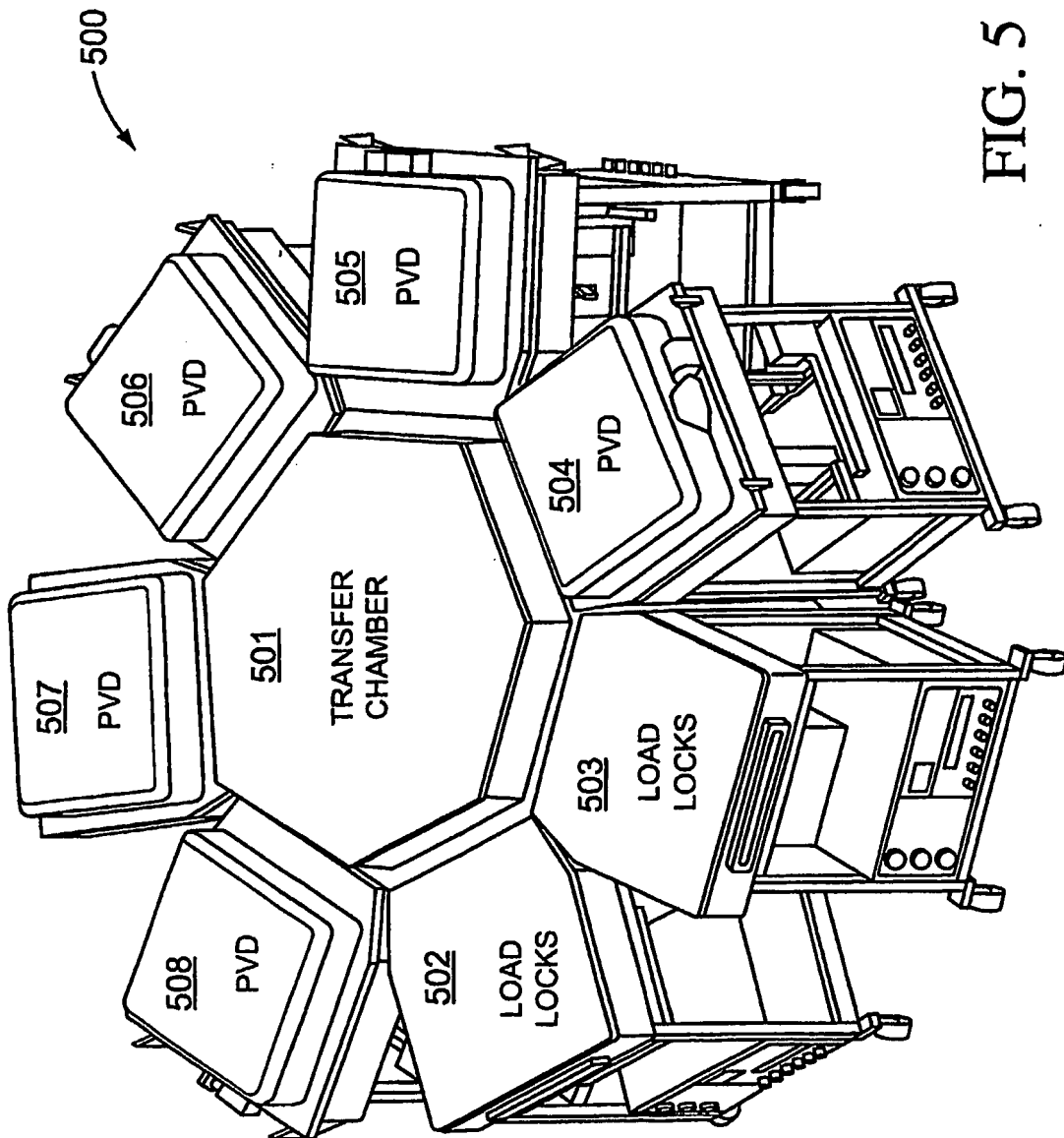


FIG. 5

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875	Application or Docket Number 10/954,182	Filing Date 10/01/2004	<input type="checkbox"/> To be Mailed
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APPLICATION AS FILED – PART I			OTHER THAN SMALL ENTITY				
	(Column 1)	(Column 2)	SMALL ENTITY <input type="checkbox"/>	OR			
FOR	NUMBER FILED	NUMBER EXTRA	RATE (\$)	FEE (\$)	OR	RATE (\$)	FEE (\$)
<input type="checkbox"/> BASIC FEE <small>(37 CFR 1.16(a), (b), or (c))</small>	N/A	N/A	N/A			N/A	
<input type="checkbox"/> SEARCH FEE <small>(37 CFR 1.16(k), (l), or (m))</small>	N/A	N/A	N/A			N/A	
<input type="checkbox"/> EXAMINATION FEE <small>(37 CFR 1.16(o), (p), or (q))</small>	N/A	N/A	N/A			N/A	
TOTAL CLAIMS <small>(37 CFR 1.16(i))</small>	minus 20 =	*	X \$ =		OR	X \$ =	
INDEPENDENT CLAIMS <small>(37 CFR 1.16(h))</small>	minus 3 =	*	X \$ =			X \$ =	
<input type="checkbox"/> APPLICATION SIZE FEE <small>(37 CFR 1.16(s))</small>	If the specification and drawings exceed 100 sheets of paper, the application size fee due is \$250 (\$125 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).						
<input type="checkbox"/> MULTIPLE DEPENDENT CLAIM PRESENT <small>(37 CFR 1.16(j))</small>							
* If the difference in column 1 is less than zero, enter "0" in column 2.			TOTAL			TOTAL	

APPLICATION AS AMENDED – PART II					OTHER THAN SMALL ENTITY				
	(Column 1)	(Column 2)	(Column 3)		SMALL ENTITY	OR			
AMENDMENT	10/24/2007	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE (\$)	ADDITIONAL FEE (\$)	OR	RATE (\$)	ADDITIONAL FEE (\$)
	Total <small>(37 CFR 1.16(i))</small>	* 25	Minus	** 61 = 0	X \$ =		OR	X \$50=	0
	Independent <small>(37 CFR 1.16(h))</small>	* 2	Minus	*** 13 = 0	X \$ =		OR	X \$210=	0
	<input type="checkbox"/> Application Size Fee <small>(37 CFR 1.16(s))</small>								
	<input type="checkbox"/> FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <small>(37 CFR 1.16(j))</small>						OR		
					TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE	0

	(Column 1)	(Column 2)	(Column 3)		SMALL ENTITY	OR			
AMENDMENT		CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE (\$)	ADDITIONAL FEE (\$)	OR	RATE (\$)	ADDITIONAL FEE (\$)
	Total <small>(37 CFR 1.16(i))</small>	*	Minus	** =	X \$ =		OR	X \$ =	
	Independent <small>(37 CFR 1.16(h))</small>	*	Minus	*** =	X \$ =		OR	X \$ =	
	<input type="checkbox"/> Application Size Fee <small>(37 CFR 1.16(s))</small>								
	<input type="checkbox"/> FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <small>(37 CFR 1.16(j))</small>						OR		
					TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE	

* If the entry in column 1 is less than the entry in column 2, write "0" in column 3.
 ** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20".
 *** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3".

Legal Instrument Examiner:
 Cassandra B. Paris

The "Highest Number Previously Paid For" (Total or Independent) is the highest number found in the appropriate box in column 1.

This collection of information is required by 37 CFR 1.16. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

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MULTIPLE DEPENDENT CLAIM FEE CALCULATION SHEET Substitute for Form PTO-1360 (For use with Form PTO/SB/06)	Application Number 10/954,182	Filing Date 01 October, 2004	<input type="checkbox"/> To be Mailed
	Applicant(s) ZHANG ET AL.		Page 1 of 1

* May be used for additional claims or amendments													
CLAIMS	AS FILED		AFTER FIRST AMENDMENT 10/24/2007		AFTER SEC. AMENDMENT		*		*		*		
	Indep	Depend	Indep	Depend	Indep	Depend	Indep	Depend	Indep	Depend	Indep	Depend	
1							51		1				
2							52		1				
3							53		1				
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46						1	96						
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48						1	98						
49						1	99						
50						1	100						
Total Indep							Total Indep	2					
Total Depend				9			Total Depend		14				
Total Claims				9			Total Claims		16				

This collection of information is required by 37 CFR 1.16. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2

Part of Paper No20071105-1.

EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	0	recalibrat\$4 with sputter\$3 with (chamber or reactor)	US-PGPUB; USPAT	OR	OFF	2007/11/06 15:10
L2	1848876	@ad>"20020316" or @rlad>"20020316"	US-PGPUB; USPAT	OR	OFF	2007/11/06 15:07
L3	3	recalibrat\$4 with sputter\$3	US-PGPUB; USPAT	OR	OFF	2007/11/06 15:09
L4	0	3 not 2	US-PGPUB; USPAT	OR	OFF	2007/11/06 15:09
L5	0	recondiction\$3 with sputter\$3 with (chamber or reactor)	US-PGPUB; USPAT	OR	OFF	2007/11/06 16:00
L6	1	recondition\$3 with sputter\$3 with (chamber or reactor)	US-PGPUB; USPAT	OR	OFF	2007/11/06 15:10
L7	17	recondition\$3 with sputter\$3	US-PGPUB; USPAT	OR	OFF	2007/11/06 15:52
L8	7	7 not 2	US-PGPUB; USPAT	OR	OFF	2007/11/06 15:11
L9	2421	((438/769) or (438/770) or (438/771) or (438/787) or (438/788)).CCLS.	US-PGPUB; USPAT	OR	OFF	2007/11/06 15:53
L10	1496	9 not 2	US-PGPUB; USPAT	OR	OFF	2007/11/06 15:53
L11	2210	((257/E23.132) or (257/E21.091) or (257/E21.169) or (257/E21.2) or (257/E21.462)).CCLS.	US-PGPUB; USPAT	OR	OFF	2007/11/06 15:58
L12	36950	target and DC and magnetic	US-PGPUB; USPAT	OR	OFF	2007/11/06 16:00
L13	153	11 AND 12	US-PGPUB; USPAT	OR	OFF	2007/11/06 16:00
L14	2631	(poison or metallic)•with mode	US-PGPUB; USPAT	OR	OFF	2007/11/06 16:00
L15	2	13 AND 14	US-PGPUB; USPAT	OR	OFF	2007/11/06 16:00
L16	2	15 not 2	US-PGPUB; USPAT	OR	OFF	2007/11/06 16:00
L17	6	11 and 14	US-PGPUB; USPAT	OR	OFF	2007/11/06 16:00
L18	0	(recondiction\$3 or recalibrat\$4) and sputter\$3 and (chamber or reactor)	FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	OFF	2007/11/06 16:01
L19	35	sputter\$3 and ((poison or metallic) with mode)	FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	OFF	2007/11/06 16:02



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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/954,182	10/01/2004	Hongmei Zhang	10655.0016-01	9873

22852 7590 11/15/2007
 FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER
 LLP
 901 NEW YORK AVENUE, NW
 WASHINGTON, DC 20001-4413

EXAMINER

ESTRADA, MICHELLE

ART UNIT	PAPER NUMBER
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2823

MAIL DATE	DELIVERY MODE
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11/15/2007

PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Office Action Summary	Application No. 10/954,182	Applicant(s) ZHANG ET AL.	
	Examiner Michelle Estrada	Art Unit 2823	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

- 1) Responsive to communication(s) filed on 24 October 2007.
- 2a) This action is **FINAL**.
- 2b) This action is non-final.
- 3) Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims

- 4) Claim(s) 41-43, 45-58, 62, 85 and 87-89 is/are pending in the application.
 - 4a) Of the above claim(s) _____ is/are withdrawn from consideration.
- 5) Claim(s) _____ is/are allowed.
- 6) Claim(s) 41-43, 45-58, 62, 85 and 87-89 is/are rejected.
- 7) Claim(s) _____ is/are objected to.
- 8) Claim(s) _____ are subject to restriction and/or election requirement.

Application Papers

- 9) The specification is objected to by the Examiner.
- 10) The drawing(s) filed on _____ is/are: a) accepted or b) objected to by the Examiner.
 - Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
 - Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

Priority under 35 U.S.C. § 119

- 12) Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
 - a) All b) Some * c) None of:
 - 1. Certified copies of the priority documents have been received.
 - 2. Certified copies of the priority documents have been received in Application No. _____.
 - 3. Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

* See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

- 1) Notice of References Cited (PTO-892)
- 2) Notice of Draftsperson's Patent Drawing Review (PTO-948)
- 3) Information Disclosure Statement(s) (PTO/SB/08)
Paper No(s)/Mail Date _____
- 4) Interview Summary (PTO-413)
Paper No(s)/Mail Date. _____
- 5) Notice of Informal Patent Application
- 6) Other: _____

DETAILED ACTION

Claim Rejections - 35 USC § 103

The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

Claims 62 and 87-89 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff et al. (6,117,279) in view of Fu et al. (6,306,265).

With respect to claims 62, Smolanoff et al. disclose providing pulsed DC power (21) between a target (16) and a substrate (Col. 5, lines 50-55); providing process gas between the target and the substrate (Col. 7, lines 25-28).

Smolanoff et al. do not clearly disclose reconditioning the target; and wherein reconditioning the target includes reactive sputtering in the metallic mode and then reactive sputtering in the poison mode.

Fu et al. disclose wherein conditioning the target includes sputtering with the target in a metallic mode to remove the surface of the target and sputtering with the target in a poisonous mode to prepare the surface (Col. 19, lines 35-40).

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff et al. and Fu et al. to enable the conditioning step of Smolanoff et al. to be performed according to the teachings of Fu et al. because one of ordinary skill in the art would have been motivated to look to alternative suitable methods of performing the disclosed conditioning step of Smolanoff et al. and art

recognized suitability for an intended purpose has been recognized to be motivation to combine. See MPEP 2144.07.

Re claim 87, providing RF bias power to a substrate (15) positioned opposite the target (Col. 5, lines 60-65). Using an specific type of filter is a matter of design choice depending on the quality of product needed, and it is obvious that the filter is going to work at certain frequencies. Furthermore, the limitation "the filter is a band rejection filter at a frequency of the bias power" is a structural limitation in a method claim, so no matter what filter is used, as long as the same result is achieved, as explained above.

Re claims 88 and 89, One of ordinary skill in the art would have been led to the recited bandwidth and frequency through routine experimentation to achieve a desired device associated characteristics and rate of sputtering.

In addition, the selection of the bandwidth and frequency, its obvious because it is a matter of determining optimum process conditions by routine experimentation with a limited number of species of result effective variables. These claims are prima facie obvious without showing that the claimed ranges achieve unexpected results relative to the prior art range. In re Woodruff, 16 USPQ2d 1935, 1937 (Fed. Cir. 1990). See also In re Huang, 40 USPQ2d 1685, 1688 (Fed. Cir. 1996)(claimed ranges or a result effective variable, which do not overlap the prior art ranges, are unpatentable unless they produce a new and unexpected result which is different in kind and not merely in degree from the results of the prior art). See also In re Boesch, 205 USPQ 215 (CCPA)

(discovery of optimum value of result effective variable in known process is ordinarily within skill or art) and *In re Aller*, 105 USPQ 233 (CCPA 1995) (selection of optimum ranges within prior art general conditions is obvious).

Note that the specification contains no disclosure of either the critical nature of the claimed bandwidth and frequency or any unexpected results arising therefrom. Where patentability is said to be based upon particular chosen bandwidth and frequency or upon another variable recited in a claim, the Applicant must show that the chosen bandwidth and frequency are critical. *In re Woodruff*, 919 F.2d 1575, 1578, 16 USPQ2d 1934, 1936 (Fed. Cir. 1990).

Claims 41, 45, 47, 49, 51, 52 and 85 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff et al. in view of Fu et al. as applied to claims 62 and 87-89 above, and further in view of Li et al. (NPL provided in this office action).

Re claim 85, The combination of Smolanoff et al. and Fu et al. does not disclose forming an oxide film by reactive sputtering in a mode between a metallic mode and a poison mode.

Li et al. disclose changing from metallic mode to poison mode (page 5, fig. 3) and while a current of oxygen is present due to RF power producing O radicals which can oxidize the target (page 6).

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff et al., Fu et al. and Li et al. to enable the oxide formation step of Li et al. to be performed in the process of Smolanoff and Fu to oxidize the target

away from the sputtering track and therefore raises the secondary electron component of current.

Re claim 41, Li et al. disclose wherein the target is a metallic target and the process gas includes oxygen (abstract)

Re claim 45, Smolanoff et al. disclose wherein the magnetic field is provided by a moving magnetron (Col. 5, lines 39-49).

Re claim 47, Smolanoff et al. disclose wherein the process gas includes a mixture of oxygen and argon (Col. 7, lines 22-27).

Re claim 49, Smolanoff et al. disclose wherein the process gas further includes nitrogen (Col. 7, lines 25-26).

Re claim 51, Smolanoff et al. disclose further including uniformly sweeping the target with a magnetic field (Col. 6, lines 1-6).

Re claim 52, Smolanoff et al. disclose wherein sweeping the target with a magnetic field includes sweeping a magnet in one direction across the target where the magnet extends beyond the target in the opposite direction (Col. 6, lines 1-6).

Claims 42, 48 and 50 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff et al. in view of Fu et al. and Li et al. as applied to claims 41, 45, 47, 49, 51, 52 and 85 above, and further in view of Chen et al. (2004/0077161).

Re claim 42, The combination of Smolanoff et al., Fu et al. and Li et al. does not disclose wherein the target is a metallic target and the process gas includes N₂.

Chen et al. disclose wherein the target is a metallic target and the process gas includes N₂ (Page 2, [0028] and [0030]).

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff, Fu, Li and Chen to enable the reactive gas step of the combination to be add nitrogen according to the teachings of Chen et al. because one of ordinary skill in the art would have been motivated to look to alternative suitable methods of performing the disclosed reactive gas step of the combination and art recognized suitability for an intended purpose has been recognized to be motivation to combine. See MPEP 2144.07.

Re claim 46, Chen et al. disclose further including holding the temperature of the substrate substantially constant (Page 3, Paragraph [0046]).

Re claim 48, Chen et al. disclose wherein the oxygen flow is adjusted by the mass flow controllers; thereby it will adjust the index refraction of the film.

Re claim 50, Chen et al. disclose wherein providing pulsed DC power to a target includes providing pulsed DC power to a target, which has an area larger than that of the substrate (See fig. 3).

Claims 43 and 53-58 are rejected under 35 U.S.C. 103(a) as being unpatentable over Smolanoff et al. in view of Fu et al. and Li et al. as applied to claims 41, 45, 47, 49, 51, 52 and 85 above, and further in view of Milonopoulou et al. (2003/0175142).

Re claim 43, the combination of Smolanoff et al., Fu et al. and Li et al. does not disclose wherein the target is a ceramic target.

Milonopoulou et al. disclose forming a coating layer on a substrate; providing a target (12), which is ceramic (Abstract).

It would have been within the scope of one of ordinary skill in the art to combine the teachings of Smolanoff et al., Fu et al., Li et al. and Milonopoulou et al. to enable the target material of the combination to be the same according to the teachings of Milonopoulou et al. because one of ordinary skill in the art would have been motivated to look to alternative suitable target materials of the disclosed target of the combination and art recognized suitability for an intended purpose has been recognized to be motivation to combine. See MPEP 2144.07.

Re claim 53, Milonopoulou et al. disclose wherein the target is an alloyed target (Abstract).

Re claim 54, Milonopoulou et al. disclose wherein the alloyed target includes one or more rare earth ions.

Re claim 55, Milonopoulou et al. disclose wherein the alloyed target includes Si and Al.

Re claim 56, Milonopoulou et al. disclose wherein the alloyed target includes one or more elements taken from a set consisting of Si, Al, Er and Yb.

Re claim 57, Milonopoulou et al. disclose wherein the alloyed target is a tiled target.

Re claim 58, Milonopoulou et al. disclose wherein each tiled target is formed by pre-alloy atomization and hot isostatic pressing of a powder (Page 2, Paragraph [0020]).

Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Michelle Estrada whose telephone number is 571-272-1858. The examiner can normally be reached on Monday through Friday.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Matthew Smith can be reached on 571-272-1907. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 571-272-2800.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).


Michelle Estrada
Primary Examiner
Art Unit 2823

ME
November 9, 2007

Notice of References Cited	Application/Control No. 10/954,182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.	
	Examiner Michelle Estrada	Art Unit 2823	Page 1 of 1

U.S. PATENT DOCUMENTS

*	Document Number Country Code-Number-Kind Code	Date MM-YYYY	Name	Classification
	A	US-		
	B	US-		
	C	US-		
	D	US-		
	E	US-		
	F	US-		
	G	US-		
	H	US-		
	I	US-		
	J	US-		
	K	US-		
	L	US-		
	M	US-		


FOREIGN PATENT DOCUMENTS

*	Document Number Country Code-Number-Kind Code	Date MM-YYYY	Country	Name	Classification
	N				
	O				
	P				
	Q				
	R				
	S				
	T				

NON-PATENT DOCUMENTS

*	Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)	
U	Li, Ning et al., "Enhancement of aluminum oxide physical vapor deposition with a secondary plasma", November 28, 2001, Scien Direct, pages, 1-11.	
V		
W		
X		

*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)
Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.

Index of Claims 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

✓	Rejected
=	Allowed


-	Cancelled
÷	Restricted

N	Non-Elected
I	Interference

A	Appeal
O	Objected

Claims renumbered in the same order as presented by applicant
 CPA
 T.D.
 R.1.47


CLAIM		DATE							
Final	Original	07/16/2007	11/09/2007						
	1	-	-						
	2	-	-						
	3	-	-						
	4	-	-						
	5	-	-						
	6	-	-						
	7	-	-						
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	11	-	-						
	12	-	-						
	13	-	-						
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	33	-	-						
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	35	-	-						
	36	-	-						

Index of Claims 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

✓	Rejected	-	Cancelled	N	Non-Elected	A	Appeal
=	Allowed	÷	Restricted	I	Interference	O	Objected

Claims renumbered in the same order as presented by applicant
 CPA
 T.D.
 R.1.47

CLAIM		DATE							
Final	Original	07/16/2007	11/09/2007						
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	39	-	-						
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	71	-	-						
	72	-	-						

Index of Claims 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

✓	Rejected
=	Allowed


-	Cancelled
÷	Restricted

N	Non-Elected
I	Interference

A	Appeal
O	Objected

Claims renumbered in the same order as presented by applicant
 CPA
 T.D.
 R.1.47


CLAIM		DATE							
Final	Original	07/16/2007	11/09/2007						
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	76	-	-						
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Index of Claims 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

✓	Rejected	-	Cancelled	N	Non-Elected	A	Appeal
=	Allowed	÷	Restricted	I	Interference	O	Objected

Claims renumbered in the same order as presented by applicant
 CPA
 T.D.
 R.1.47

CLAIM		DATE							
Final	Original	07/16/2007	11/09/2007						
	109	✓	-						

Search Notes 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

SEARCHED			
Class	Subclass	Date	Examiner
Updated as before		11/6/07	ME
257	E23.132,E21.091,E21.169,E21.2,E21.462	11/6/07	ME

SEARCH NOTES		
Search Notes	Date	Examiner
See East search attached	11/6/07	ME
google search	11/5/07	ME

INTERFERENCE SEARCH			
Class	Subclass	Date	Examiner

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)
)
Hongmei ZHANG et al.) Group Art Unit: 2823
)
Application No.: 10/954,182) Examiner: Michelle ESTRADA
)
Filed: October 1, 2004)
)
For: BIASED PULSE DC REACTIVE) Confirmation No.: 9873
SPUTTERING OF OXIDE FILMS)

MAIL STOP AMENDMENT

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

AMENDMENT AND RESPONSE TO OFFICE ACTION

In reply to the Office Action mailed November 15, 2007, please amend the above-identified application as follows:

Amendments to the Claims are reflected in the listing of claims in this paper beginning on page 2.

Remarks/Arguments follow the amendment sections of this paper beginning on page 7.

Attachments to this amendment include: Copies of referenced articles by P.F. Cheng et al., *J. Vac. Sci. Technol. B* 13 2 (1995), pp. 203-208, and S. M. Rossnagel et al., *Appl. Phys. Lett.* 63 (1993), p. 24.

AMENDMENTS TO THE CLAIMS:

This listing of claims will replace all prior versions and listings of claims in the application:

Claims 1-40 (Canceled).

Claim 41 (Previously presented): The method of claim 85 wherein the target is a metallic target and the process gas includes oxygen.

Claim 42 (Previously presented): The method of claim 85 wherein the target is a metallic target and the process gas includes one or more of a set consisting of N₂, NH₃, CO, NO, CO₂, halide containing gasses.

Claim 43 (Previously presented): The method of claim 85 wherein the target is a ceramic target.

Claim 44 (Canceled).

Claim 45 (Previously presented): The method of claim 85 wherein the magnetic field is provided by a moving magnetron.

Claim 46 (Previously presented) The method of claim 85 further including holding the temperature of the substrate substantially constant.

Claim 47 (Previously presented): The method of claim 85 wherein the process gas includes a mixture of Oxygen and Argon.

Claim 48 (Previously presented): The method of claim 85 wherein the Oxygen flow is adjusted to adjust the index of refraction of the film.

Claim 49 (Previously presented): The method of claim 85 wherein the process gas further includes nitrogen.

Claim 50 (Previously presented): The method of claim 85 wherein providing pulsed DC power to a target includes providing pulsed DC power to a target which has an area larger than that of the substrate.

Claim 51 (Previously presented): The method of claim 85, further including uniformly sweeping the target with a magnetic field.

Claim 52 (Previously presented): The method of claim 51 wherein uniformly sweeping the target with a magnetic field includes sweeping a magnet in one direction across the target where the magnet extends beyond the target in the opposite direction.

Claim 53 (Previously presented): The method of claim 85 wherein the target is an alloyed target.

Claim 54 (Previously presented): The method of claim 53 wherein the alloyed target includes one or more rare-earth ions.

Claim 55 (Previously presented): The method of claim 53 wherein the alloyed target includes Si and Al.

Claim 56 (Previously presented): The method of claim 53 wherein the alloyed target includes one or more elements taken from a set consisting of Si, Al, Er, Yb, Zn, Ga, Ge, P, As, Sn, Sb, Pb, Ag, Au, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy Ho, Tm, and Lu.

Claim 57 (Previously presented): The method of claim 53 wherein the alloyed target is a tiled target.

Claim 58 (Previously presented): The method of claim 57 wherein each tile of the tiled target is formed by prealloy atomization and hot isostatic pressing of a powder.

Claims 59-61 (Canceled).

Claim 62 (Currently amended): A method of depositing a film on [[a]] an insulating substrate, comprising:

providing a process gas between a conductive target and [[a]] the substrate;

providing pulsed DC power to the target through a narrow band rejection filter such that the target alternates between positive and negative voltages;

providing an RF bias at a frequency that corresponds to the narrow band rejection filter to the substrate;

providing a magnetic field to the target; and

reconditioning the target;

wherein reconditioning the target includes reactive sputtering in the metallic mode and then reactive sputtering in the poison mode.

Claims 63-84 (Canceled).

Claim 85 (Currently amended): A method of depositing ~~[[a]]~~ an insulating film on a substrate, comprising:

providing a process gas between a target and a substrate;

providing pulsed DC power to the target through a narrow band rejection filter such that the voltage on the target alternates between positive and negative voltages;

providing an RF bias that corresponds to the narrow band rejection filter to the substrate;

and

providing a magnetic field to the target;~~and~~

wherein ~~[[a]]~~ an oxide material is deposited on the substrate, and ~~an oxide~~ the insulating film is formed by reactive sputtering in a mode between a metallic mode and a poison mode.

Claims 86-87 (Canceled).

Claim 88 (Currently amended): The method according to claim ~~[[87]]~~ 85, wherein the narrow band-rejection filter has a bandwidth of about 100 kHz.

Claim 89 (Previously presented): The method according to claim ~~[[87]]~~ 85, wherein the RF frequency is about 2 MHz.

Claims 90-109 (Canceled).

REMARKS

Claims 41-43, 45-58, 62, 85, and 87-89 are pending in the above-identified application. The Examiner has rejected claims 41-43, 45-58, 62, 85, and 87-89. In this amendment, claims 62 and 85 have been amended as agreed during an Interview held on December 11, 2007. Claim 87 has been cancelled.

Examiner's Interview

Applicants thank the Examiner for meeting with us on December 11, 2007 (the "Interview"). In attendance at the Interview were Examiner Michelle Estrada, Inventor R. Ernest Demaray, and Applicants' representative Gary J. Edwards. During the interview, all of the claims were discussed as well as the art that has been cited against the claims. Agreement with respect to the claims was reached. In this Amendment, the claims have been amended as discussed during the interview. The Examiner indicated in the Interview Summary that the proposed language for the claims "would overcome the rejection on record."

The substance of the discussion with the Examiner with respect to the claims and the art is provided below.

Claim Rejections Under 35 U.S.C. § 103(a)

Claims 62 and 87-89

Claims 62, and 87-89 are rejected under 35 U.S.C. § 103(a) as being unpatentable over U.S. Patent No. 6,117,279 to Smolanoff et al. ("Smolanoff") in view of U.S. Patent No. 6,306,265 to Fu et al. ("Fu"). As discussed during the interview, Smolanoff teaches away from a system where the target voltage becomes positive, and therefore teaches away from "providing pulsed DC power to the target through a narrow band rejection filter such that the target

alternates between positive and negative voltages” and “providing an RF bias at a frequency that corresponds to the narrow band rejection filter to the substrate,” as is recited in claims 62.

Additionally, because Smolanoff teaches away from the elements of claim 62, there is no reason to combine Smolanoff with Fu as is suggested by the Examiner. However, even if they were combinable, the combination of Smolanoff and Fu does not teach or suggest the combination of “providing pulsed DC power to the target through a narrow band rejection filter such that the target alternates between positive and negative voltages” and “providing an RF bias at a frequency that corresponds to the narrow band rejection filter to the substrate,” as is recited in claim 62.

Smolanoff teaches a directed ion metal vapor source for deposition of conductive films. Although Smolanoff states that the DC source can be a pulsed DC source, Smolanoff also states that “[p]ower from the steady or **pulsed DC power supply 21** and/or RF generator 24 **produces a negative potential on the target 16.**” (Smolanoff, col. 5, line 66, -col. 6, line 1) (emphasis added). In every disclosure of target voltage, Smolanoff teaches that the target voltage must be negative. (*See, e.g.* col. 5, lines 39-44 (“[t]he magnet structure 20 preferably includes magnets that produce a closed magnetic tunnel over the surface of the target 16 that traps electrons given off into the chamber 12 by the cathode assembly 17 when the cathode assembly 17 is **electrically energized to a negative potential** as is familiar to one skilled in the art”); col. 6, lines 9-12 (“[t]his main plasma in the region 23 becomes a source of positive ions of gas that are accelerated toward, and collide against, **the negatively charged surface of the target 16,** thereby ejecting particles of coating material from the target 16”) (emphasis added)).

Smolanoff never teaches that the target can be positive and, in accordance with the teachings of Smolanoff, the target voltage must always be negative. Therefore, Smolanoff

teaches away from “providing pulsed DC power to the target through a narrow band rejection filter such that the target alternates between positive and negative voltages” as is recited in claim 62. Additionally, Smolanoff then teaches away from the combination “providing pulsed DC power to the target through a narrow band rejection filter such that the voltage on the target alternates between positive and negative voltages” and “providing an RF bias that corresponds to the narrow band rejection filter to the substrate,” as is recited in claim 62.

Even if Smolanoff could be combined with Fu as suggested, the combination would not teach or suggest the claimed invention. The Examiner stated that “Smolanoff et al. do not clearly disclose reconditioning the target; and wherein reconditioning the target includes reactive sputtering in the metallic mode and then reactive sputtering in the poison mode.” (Office Action, page 2). Fu is relied upon to disclose “wherein conditioning the target includes sputtering with the target in a metallic mode to remove the surface of the target and sputtering with the target in a poisonous mode to prepare the surface (Col. 19, lines 35-40).” (Office Action, page 2).

Fu teaches high density, magnetic field enhanced ionized metal vapor deposition of conducting films. (See Fu, abstract). Fu, however, teaches utilization of a DC power supply (Fu, col. 1, lines 30-32) in combination with an RF bias applied to the substrate (Fu, col. 2, lines 36-41). Therefore, Fu fails to teach the combination “providing pulsed DC power to the target through a narrow band rejection filter such that the voltage on the target alternates between positive and negative voltages” and “providing an RF bias that corresponds to the narrow band rejection filter to the substrate,” as is recited in claim 62.

Fu does teach operation in the poison mode and operation in the metallic mode as applied to TiN deposition, but does not teach “wherein an oxide material is deposited on the substrate,

and the insulating film is formed by reactive sputtering in a mode between a metallic mode and a poison mode,” as is recited in claim 62. As stated by Fu,

Reactive sputtering to produce TiN is known to operate in two modes, metallic mode and poison mode. Metallic mode produces a high-density, gold-colored film on the wafer. Poison mode, which is often associated with a high nitrogen flow, produces a purple/brown film which advantageously has low stress. However, the poison-mode film has many grain boundaries, and film defects severely reduce chip yield. Furthermore, the deposition rate in poison mode is typically only one-quarter of the rate in metallic mode. It is generally believed that in poison mode the nitrogen reacts with the target to form a TiN surface on the Ti target while in metallic mode the target surface remains clean and TiN forms only the wafer.

(Fu, col. 19, lines 28-30). Fu teaches operation in either metallic mode or poison mode, and does not teach “wherein an oxide material is deposited on the substrate, and the insulating film is formed by reactive sputtering in a mode between a metallic mode and a poison mode,” as is recited in claim 62.

Therefore, claim 62 is allowable over the combination of Smolanoff and Fu. Similar to the discussion regarding claim 62, the combination of Smolanoff and Fu does not teach “providing pulsed DC power to the target through a narrow band rejection filter such that the voltage on the target alternates between positive and negative voltages” in combination with “providing an RF bias that corresponds to the narrow band rejection filter to the substrate,” as is recited in claim 85. Further, Fu does not teach “wherein an oxide material is deposited on the substrate, and the insulating film is formed by reactive sputtering in a mode between a metallic mode and a poison mode,” as is recited in claim 85. Claim 87 has been canceled. Claims 88-89 depend from claim 85 and are therefore allowable for at least the same reasons as is claim 85.

In addition, the Examiner initially indicated, with regard to the narrow band-rejection filter, that “[u]sing an specific type of filter is a matter of design choice depending on the quality

of product needed, and it is obvious that the filter is going to work at certain frequencies.” (Office Action, page 3). However, as explained during the interview, that is not the case. The narrow band rejection filter allows the combination of pulsed-dc power to the target (where the target voltage is oscillated between positive and negative voltages) and an RF bias on the substrate. A filter that blocks too many of the constituent frequencies of the pulsed DC waveform results in the target voltage not attaining a positive voltage. A filter that does not block the RF bias voltage can result in failure of the DC power supply. Smolanoff does not teach the “narrow band rejection filtering” recited in each of claims 62 and 85.

Claims 41, 45, 47, 49, 51, 52, and 85

Claims 41, 45, 47, 49, 51, 52, and 85 are rejected under 35 U.S.C. § 103(a) as being unpatentable over Smolanoff in view of Fu, as applied to claims 62, and 87-89, and in further view of reference titled “Enhancement of Aluminum Oxide Physical Vapor Deposition with a Secondary Plasma” to Li et al. (“Li”). As discussed above, claim 85 is allowable over the combination of Smolanoff and Fu. Li also fails to teach “providing pulsed DC power to the target through a narrow band rejection filter such that the voltage on the target alternates between positive and negative voltages” in combination with “providing an RF bias that corresponds to the narrow band rejection filter to the substrate,” as is recited in claim 85.

At best, Li teaches a pulsed DC source with a positive target voltage and a DC substrate bias. With regard to substrate bias, Li states that

The angular distribution of the sputtered atoms is roughly a cosine distribution, and is further broadened by gas phase scattering, yielding insufficient bottom coverage and voids during filling of high aspect ratio features. **This problem is solved by ionizing the metal flux and applying a bias on the substrate, accelerating**

the metal ions through the plasma sheath near the substrate surface [14 and 15].

(Li, pgs 162). There is no teaching of an RF bias to the substrate. Further, the referenced articles (P.F. Cheng et al., *J. Vac. Sci. Technol. B* **13** 2 (1995), pp. 203-208, and S. M. Rosnagel et al., *Appl. Phys. Lett.* **63** (1993), p. 24) teach only a DC bias. (For the Examiner's convenience copies of these articles are attached to this response). Therefore, as discussed with the Examiner, Li does not teach or suggest "providing pulsed DC power to the target through a narrow band rejection filter such that the voltage on the target alternates between positive and negative voltages" in combination with "providing an RF bias that corresponds to the narrow band rejection filter to the substrate," as is recited in claim 85.

Therefore, at least for the reasons discussed above claim 85 is allowable over the combination of Smolanoff, Fu, and Li. Claims 41, 45, 47, 49, 51, and 52 depend from claim 85 and are allowable over Smolanoff, Fu, and Li for at least the same reasons as is claim 85.

Claims 42, 48, and 50

Claims 42, 48, and 50 are rejected under 35 U.S.C. § 103(a) as being unpatentable over Smolanoff in view of Fu and Li, as applied to claims 41, 45, 47, 49, 51, 52, and 85, and in further view of U.S. Patent Publication No. 2004/0077161 to Chen et al. ("Chen").

As discussed above, claim 85 is allowable over Smolanoff, Fu, and Li. Chen also does not teach "providing pulsed DC power to the target through a narrow band rejection filter such that the voltage on the target alternates between positive and negative voltages" in combination with "providing an RF bias that corresponds to the narrow band rejection filter to the substrate," as is recited in claim 85.

Chen teaches “[a] method of layer formation on a substrate with high aspect ratio features.” (Chen, par. 0008). As taught in Chen, “[t]he target power source 108 is used to infuse the one or more process gases with energy and may comprise a DC source, a radio frequency (RF) source, a DC-pulsed source, or a microwave source.” Chen does not teach “providing pulsed DC power to the target through a narrow band rejection filter such that the voltage on the target alternates between positive and negative voltages,” as is recited in claim 85. Chen further teaches that “the PVD chamber 36 may include a bias power source 124 for biasing the substrate 120” and further that “[t]he bias power source 124 is typically an AC source having a frequency of, for example, about 400 kHz.” (Chen, par. 0034). Because Chen does not teach “providing pulsed DC power,” Chen does not teach or suggest “providing pulsed DC power to the target through a narrow band rejection filter such that the voltage on the target alternates between positive and negative voltages” in combination with “providing an RF bias that corresponds to the narrow band rejection filter to the substrate,” as is recited in claim 85.

Therefore, claim 85 is allowable over Smolanoff, Fu, Li and Chen. Claims 42, 48, and 50 depend from claim 85 and are therefore allowable for at least the same reasons as is claim 85.

Claims 43, and 53-58

Claims 43, and 53-58 are rejected under 35 U.S.C. § 103(a) as being unpatentable over Smolanoff in view of Fu and Li, as applied to claims 41, 45, 47, 49, 51, 52, and 85, and in further view of U.S. Patent Publication No. 2003/0175142 to Milonopoulou et al. (“Milonopoulou”).

Milonopoulou was filed on the same day as was the present application, includes an overlap of inventorship with the present application (R. Ernest Demaray), is co-owned with the

present application, and was incorporated by reference in the present application (Paragraph 0048). Therefore, Milonopoulou can not be prior art to the present application.

Claims 43 and 53-58 depend from claim 85 and are allowable over the cited art for at least the same reasons as is claim 85.

Conclusion

In view of the foregoing amendments and remarks, Applicants respectfully request timely allowance of the pending claims. If the Examiner has any questions about these Amendments or Remarks, the Examiner is invited to call Applicants' representative.

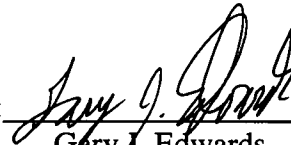
Please grant any extensions of time required to enter this response and charge any additional required fees to our deposit account 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Dated: December 18, 2007

By: _____



Gary J. Edwards
Reg. No. 41,008
(650) 849-6622

Patent Application No. 10/954,182, filed 10/13/05, pp. 203-208, and
S. M. Rossmagel et al., *J. Vac. Sci. Technol. B* 13 (1995), pp. 203-208, and
S. M. Rossmagel et al., *Appl. Phys. Lett.* 63 (1993), p. 24.

Electronic Acknowledgement Receipt

EFS ID:	2610504
Application Number:	10954182
International Application Number:	
Confirmation Number:	9873
Title of Invention:	Biased pulse DC reactive sputtering of oxide films
First Named Inventor/Applicant Name:	Hongmei Zhang
Customer Number:	22852
Filer:	Gary James Edwards/Annie Wong
Filer Authorized By:	Gary James Edwards
Attorney Docket Number:	10655.0016-01
Receipt Date:	18-DEC-2007
Filing Date:	01-OCT-2004
Time Stamp:	20:02:45
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	no
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File Listing:

Document Number	Document Description	File Name	File Size(Bytes) /Message Digest	Multi Part /.zip	Pages (if appl.)
1	Amendment - After Non-Final Rejection	Amendment_Response_to_OA_10655-0016-01.pdf	215331 <small>2ba839c98c9fff133bf287a814ec986384469d58</small>	no	14

Warnings:

The page size in the PDF is too large. The pages should be 8.5 x 11 or A4. If this PDF is submitted, the pages will be resized upon entry into the Image File Wrapper and may affect subsequent processing

Information:

2	NPL Documents	Cheng_PF_et_al.pdf	472139	no	6
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Warnings:

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3	NPL Documents	Rossnagel_SM_et_al.pdf	1551026	no	5
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Information:

Total Files Size (in bytes):			2238496		
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This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

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PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875	Application or Docket Number 10/954,182	Filing Date 10/01/2004	<input type="checkbox"/> To be Mailed
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FOR	NUMBER FILED	NUMBER EXTRA	RATE (\$)	FEE (\$)	OR	RATE (\$)	FEE (\$)
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<input type="checkbox"/> SEARCH FEE <small>(37 CFR 1.16(k), (l), or (m))</small>	N/A	N/A	N/A			N/A	
<input type="checkbox"/> EXAMINATION FEE <small>(37 CFR 1.16(o), (p), or (q))</small>	N/A	N/A	N/A			N/A	
TOTAL CLAIMS <small>(37 CFR 1.16(i))</small>	minus 20 =	*	X \$ =		OR	X \$ =	
INDEPENDENT CLAIMS <small>(37 CFR 1.16(h))</small>	minus 3 =	*	X \$ =			X \$ =	
<input type="checkbox"/> APPLICATION SIZE FEE <small>(37 CFR 1.16(s))</small>	If the specification and drawings exceed 100 sheets of paper, the application size fee due is \$250 (\$125 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).						
<input type="checkbox"/> MULTIPLE DEPENDENT CLAIM PRESENT <small>(37 CFR 1.16(j))</small>							
* If the difference in column 1 is less than zero, enter "0" in column 2.			TOTAL			TOTAL	

APPLICATION AS AMENDED – PART II					OTHER THAN SMALL ENTITY				
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					TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE	0

APPLICATION AS AMENDED – PART II					OTHER THAN SMALL ENTITY			
	(Column 1)	(Column 2)	(Column 3)		SMALL ENTITY	OR		
AMENDMENT	CLAIMS REMAINING AFTER AMENDMENT	HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE (\$)	ADDITIONAL FEE (\$)	OR	RATE (\$)	ADDITIONAL FEE (\$)
	Total <small>(37 CFR 1.16(i))</small>	*	Minus	**	X \$ =		OR	X \$ =
	Independent <small>(37 CFR 1.16(h))</small>	*	Minus	***	X \$ =		OR	X \$ =
	<input type="checkbox"/> Application Size Fee <small>(37 CFR 1.16(s))</small>							
	<input type="checkbox"/> FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM <small>(37 CFR 1.16(j))</small>						OR	
					TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE

* If the entry in column 1 is less than the entry in column 2, write "0" in column 3.
 ** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20".
 *** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3".
 The "Highest Number Previously Paid For" (Total or Independent) is the highest number found in the appropriate box in column 1.

Legal Instrument Examiner:
 /GAIL D. D. WOOTEN/

This collection of information is required by 37 CFR 1.16. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

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901 New York Avenue, N.W.
Washington, DC 20001-4413

Telephone
(202) 408-4000

Facsimile
(202) 408-4400

FACSIMILE TRANSMITTAL

To: Michelle Estrada
Firm: USPTO Centralized Facsimile Office
Fax No.: (571) 273-8300 Phone No.: (571) 272-1858
Application No. 10/954,182
Confirmation No. 9873
Subject: Atty. Doc. 10655.0016-01000 Date: January 17, 2008

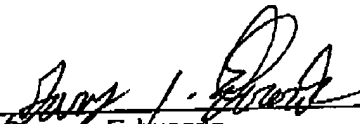
From: Gary J. Edwards Phone No.: 650-849-6622
Fax # Verified by: A. Wong; x6738 No. of Pages (incl. this page) 2

Confirmation Copy to Follow: No

Message:

CERTIFICATE OF TRANSMISSION UNDER 37 C.F.R. § 1.8

I hereby certify that the corrected copy of Form PTO/SB/08, filed with a Supplemental Information Disclosure Statement on October 24, 2007, is being transmitted by facsimile to the U.S. Patent and Trademark Office on the above-identified date.



Gary J. Edwards
Reg. No. 41,008

GJE/aw

If there is a problem with this transmission, notify fax room at (202) 408-4174 or the sender at the number above.

This facsimile is intended only for the individual to whom it is addressed and may contain information that is privileged, confidential, or exempt from disclosure under applicable law. If you have received this facsimile in error, please notify the sender immediately by telephone (collect), and return the original message by first-class mail to the above address.

IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>			Application Number	10/954,182
			Filing Date	October 1, 2004
			First Named Inventor	ZHANG, Hongmei
			Art Unit	2823
			Examiner Name	ESTRADA, Michelle
			Attorney Docket Number	10655.0016-01
Sheet	1	of	1	

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U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS				
Examiner Initials	Cite No.	Document Number	Issue or Publication Date	Name of Patentee or Applicant of Cited Document
		Number-Kind Code ² (if known)	MM-DD-YYYY	
		US-6,391,166 B1	05-21-2002	Wang
		US-7,262,131 B2	08-28-2007	Narasimhan et al.

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

FOREIGN PATENT DOCUMENTS						
Examiner Initials	Cite No. ¹	Foreign Patent Document	Publication Date	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁹
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)				
		WO 2007/027535 A2	03-08-2007	Symmorphix, Inc.		

NON PATENT LITERATURE DOCUMENTS			
Examiner Initials	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁹
		Office Action dated May 21, 2007, in U.S. Application No. 10/291,179 (Attorney Docket No. 9140.0001-00).	
		Final Office Action dated April 13, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Amendment/RCE filed August 9, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Office Action dated September 5, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Final Office Action dated September 7, 2007, in U.S. Application No. 11/100,856 (Attorney Docket No. 9140.0015-01).	
		Final Office Action dated May 2, 2007, in U.S. Application No. 10/101,863 (Attorney Docket No. 9140.0016-00).	
		Response to Final Office Action filed October 2, 2007, in U.S. Application No. 10/101,863 (Attorney Docket No. 9140.0016-00).	
		Response to Office Action filed July 9, 2007, in U.S. Application No. 10/650,461 (Attorney Docket No. 9140.0025-00).	
		Final Office Action dated October 10, 2007, in U.S. Application No. 10/650,461 (Attorney Docket No. 9140.0025-00).	
		Corrected Notice of Allowance dated June 7, 2007, in U.S. Application No. 11/228,805 (Attorney Docket No. 9140.0030-01).	
		Supplemental Notice of Allowance dated July 5, 2007, in U.S. Application No. 11/228,805 (Attorney Docket No. 9140.0030-01).	
		Second Supplemental Preliminary Amendment filed May 31, 2007, in U.S. Application No. 11/297,057 (Attorney Docket No. 9140.0042-00).	

Examiner Signature	Date Considered
--------------------	-----------------

EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

EXPRESS MAIL LABEL NO.
FM 100825779 IIS

EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	1942053	@ad>"20020316" or @rlad>"20020316"	US-PGPUB; USPAT	OR	OFF	2008/01/22 09:25
L2	2457	((438/769) or (438/770) or (438/771) or (438/787) or (438/788)).CCLS.	US-PGPUB; USPAT	OR	OFF	2008/01/22 09:25
L3	1498	L2 not L1	US-PGPUB; USPAT	OR	OFF	2008/01/22 09:25
L4	2241	((257/E23.132) or (257/E21.091) or (257/E21.169) or (257/E21.2) or (257/E21.462)).CCLS.	US-PGPUB; USPAT	OR	OFF	2008/01/22 09:25
L5	38060	target and DC and magnetic	US-PGPUB; USPAT	OR	OFF	2008/01/22 09:25
L6	158	L4 AND L5	US-PGPUB; USPAT	OR	OFF	2008/01/22 09:25
L7	35	sputter\$3 and ((poison or metallic) with mode)	FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	OFF	2008/01/22 09:25
L8	35	sputter\$3 and ((poison or metallic) with mode)	FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	OFF	2008/01/22 09:25
L9	0	(recondiction\$3 or recalibrat\$4) and sputter\$3 and (chamber or reactor)	FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	OFF	2008/01/22 09:25
L10	2676	(poison or metallic) with mode	US-PGPUB; USPAT	OR	OFF	2008/01/22 09:26
L11	2	L6 AND L10	US-PGPUB; USPAT	OR	OFF	2008/01/22 09:26
L12	156	6 not 2	US-PGPUB; USPAT	OR	OFF	2008/01/22 09:26



9

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NOTICE OF ALLOWANCE AND FEE(S) DUE

22852 7590 02/01/2008

FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER
LLP
901 NEW YORK AVENUE, NW
WASHINGTON, DC 20001-4413

EXAMINER: ESTRADA, MICHELLE
ART UNIT: 2823
PAPER NUMBER:
DATE MAILED: 02/01/2008

Table with 5 columns: APPLICATION NO., FILING DATE, FIRST NAMED INVENTOR, ATTORNEY DOCKET NO., CONFIRMATION NO.
Row 1: 10/954,182, 10/01/2004, Hongmei Zhang, 10655.0016-01, 9873
TITLE OF INVENTION: BIASED PULSE DC REACTIVE SPUTTERING OF OXIDE FILMS

Table with 7 columns: APPLN. TYPE, SMALL ENTITY, ISSUE FEE DUE, PUBLICATION FEE DUE, PREV. PAID ISSUE FEE, TOTAL FEE(S) DUE, DATE DUE
Row 1: nonprovisional, NO, \$1440, \$300, \$0, \$1740, 05/01/2008

THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT. PROSECUTION ON THE MERITS IS CLOSED. THIS NOTICE OF ALLOWANCE IS NOT A GRANT OF PATENT RIGHTS. THIS APPLICATION IS SUBJECT TO WITHDRAWAL FROM ISSUE AT THE INITIATIVE OF THE OFFICE OR UPON PETITION BY THE APPLICANT. SEE 37 CFR 1.313 AND MPEP 1308.

THE ISSUE FEE AND PUBLICATION FEE (IF REQUIRED) MUST BE PAID WITHIN THREE MONTHS FROM THE MAILING DATE OF THIS NOTICE OR THIS APPLICATION SHALL BE REGARDED AS ABANDONED. THIS STATUTORY PERIOD CANNOT BE EXTENDED. SEE 35 U.S.C. 151. THE ISSUE FEE DUE INDICATED ABOVE DOES NOT REFLECT A CREDIT FOR ANY PREVIOUSLY PAID ISSUE FEE IN THIS APPLICATION. IF AN ISSUE FEE HAS PREVIOUSLY BEEN PAID IN THIS APPLICATION (AS SHOWN ABOVE), THE RETURN OF PART B OF THIS FORM WILL BE CONSIDERED A REQUEST TO REAPPLY THE PREVIOUSLY PAID ISSUE FEE TOWARD THE ISSUE FEE NOW DUE.

HOW TO REPLY TO THIS NOTICE:

I. Review the SMALL ENTITY status shown above.

If the SMALL ENTITY is shown as YES, verify your current SMALL ENTITY status:

- A. If the status is the same, pay the TOTAL FEE(S) DUE shown above.
B. If the status above is to be removed, check box 5b on Part B - Fee(s) Transmittal and pay the PUBLICATION FEE (if required) and twice the amount of the ISSUE FEE shown above, or

If the SMALL ENTITY is shown as NO:

- A. Pay TOTAL FEE(S) DUE shown above, or
B. If applicant claimed SMALL ENTITY status before, or is now claiming SMALL ENTITY status, check box 5a on Part B - Fee(s) Transmittal and pay the PUBLICATION FEE (if required) and 1/2 the ISSUE FEE shown above.

II. PART B - FEE(S) TRANSMITTAL, or its equivalent, must be completed and returned to the United States Patent and Trademark Office (USPTO) with your ISSUE FEE and PUBLICATION FEE (if required). If you are charging the fee(s) to your deposit account, section "4b" of Part B - Fee(s) Transmittal should be completed and an extra copy of the form should be submitted. If an equivalent of Part B is filed, a request to reapply a previously paid issue fee must be clearly made, and delays in processing may occur due to the difficulty in recognizing the paper as an equivalent of Part B.

III. All communications regarding this application must give the application number. Please direct all communications prior to issuance to Mail Stop ISSUE FEE unless advised to the contrary.

IMPORTANT REMINDER: Utility patents issuing on applications filed on or after Dec. 12, 1980 may require payment of maintenance fees. It is patentee's responsibility to ensure timely payment of maintenance fees when due.

PART B - FEE(S) TRANSMITTAL

**Complete and send this form, together with applicable fee(s), to: Mail Mail Stop ISSUE FEE
 Commissioner for Patents
 P.O. Box 1450
 Alexandria, Virginia 22313-1450
 or Fax (571)-273-2885**

INSTRUCTIONS: This form should be used for transmitting the ISSUE FEE and PUBLICATION FEE (if required). Blocks 1 through 5 should be completed where appropriate. All further correspondence including the Patent, advance orders and notification of maintenance fees will be mailed to the current correspondence address as indicated unless corrected below or directed otherwise in Block 1, by (a) specifying a new correspondence address; and/or (b) indicating a separate "FEE ADDRESS" for maintenance fee notifications.

CURRENT CORRESPONDENCE ADDRESS (Note: Use Block 1 for any change of address)

Note: A certificate of mailing can only be used for domestic mailings of the Fee(s) Transmittal. This certificate cannot be used for any other accompanying papers. Each additional paper, such as an assignment or formal drawing, must have its own certificate of mailing or transmission.

22852 7590 02/01/2008

FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER
 LLP
 901 NEW YORK AVENUE, NW
 WASHINGTON, DC 20001-4413

Certificate of Mailing or Transmission
 I hereby certify that this Fee(s) Transmittal is being deposited with the United States Postal Service with sufficient postage for first class mail in an envelope addressed to the Mail Stop ISSUE FEE address above, or being facsimile transmitted to the USPTO (571) 273-2885, on the date indicated below.

(Depositor's name)
(Signature)
(Date)

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/954,182	10/01/2004	Hongmei Zhang	10655.0016-01	9873

TITLE OF INVENTION: BIASED PULSE DC REACTIVE SPUTTERING OF OXIDE FILMS

APPLN. TYPE	SMALL ENTITY	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	NO	\$1440	\$300	\$0	\$1740	05/01/2008

EXAMINER	ART UNIT	CLASS-SUBCLASS
ESTRADA, MICHELLE	2823	438-002000

<p>1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363). <input type="checkbox"/> Change of correspondence address (or Change of Correspondence Address form PTO/SB/122) attached. <input type="checkbox"/> "Fee Address" indication (or "Fee Address" Indication form PTO/SB/47; Rev 03-02 or more recent) attached. Use of a Customer Number is required.</p>	<p>2. For printing on the patent front page, list (1) the names of up to 3 registered patent attorneys or agents OR, alternatively, (2) the name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.</p> <p>1 _____ 2 _____ 3 _____</p>
---	---

3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document has been filed for recordation as set forth in 37 CFR 3.11. Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE _____ (B) RESIDENCE: (CITY and STATE OR COUNTRY) _____

Please check the appropriate assignee category or categories (will not be printed on the patent) : Individual Corporation or other private group entity Government

<p>4a. The following fee(s) are submitted:</p> <input type="checkbox"/> Issue Fee <input type="checkbox"/> Publication Fee (No small entity discount permitted) <input type="checkbox"/> Advance Order - # of Copies _____	<p>4b. Payment of Fee(s): (Please first reapply any previously paid issue fee shown above)</p> <input type="checkbox"/> A check is enclosed. <input type="checkbox"/> Payment by credit card. Form PTO-2038 is attached. <input type="checkbox"/> The Director is hereby authorized to charge the required fee(s), any deficiency, or credit any overpayment, to Deposit Account Number _____ (enclose an extra copy of this form).
--	---

5. Change in Entity Status (from status indicated above)

a. Applicant claims SMALL ENTITY status. See 37 CFR 1.27. b. Applicant is no longer claiming SMALL ENTITY status. See 37 CFR 1.27(g)(2).

NOTE: The Issue Fee and Publication Fee (if required) will not be accepted from anyone other than the applicant; a registered attorney or agent; or the assignee or other party in interest as shown by the records of the United States Patent and Trademark Office.

Authorized Signature _____ Date _____
 Typed or printed name _____ Registration No. _____

This collection of information is required by 37 CFR 1.311. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, Virginia 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, Virginia 22313-1450.

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Table with 5 columns: APPLICATION NO., FILING DATE, FIRST NAMED INVENTOR, ATTORNEY DOCKET NO., CONFIRMATION NO.
10/954,182 10/01/2004 Hongmei Zhang 10655.0016-01 9873

22852 7590 02/01/2008
FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER
LLP
901 NEW YORK AVENUE, NW
WASHINGTON, DC 20001-4413

Table with 2 columns: ART UNIT, PAPER NUMBER
2823
DATE MAILED: 02/01/2008

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b)
(application filed on or after May 29, 2000)

The Patent Term Adjustment to date is 0 day(s). If the issue fee is paid on the date that is three months after the mailing date of this notice and the patent issues on the Tuesday before the date that is 28 weeks (six and a half months) after the mailing date of this notice, the Patent Term Adjustment will be 0 day(s).

If a Continued Prosecution Application (CPA) was filed in the above-identified application, the filing date that determines Patent Term Adjustment is the filing date of the most recent CPA.

Applicant will be able to obtain more detailed information by accessing the Patent Application Information Retrieval (PAIR) WEB site (http://pair.uspto.gov).

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Customer Service Center of the Office of Patent Publication at 1-(888)-786-0101 or (571)-272-4200.

Notice of Allowability

Application No. 10/954,182	Applicant(s) ZHANG ET AL.	
Examiner Michelle Estrada	Art Unit 2823	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address--

All claims being allowable, PROSECUTION ON THE MERITS IS (OR REMAINS) CLOSED in this application. If not included herewith (or previously mailed), a Notice of Allowance (PTOL-85) or other appropriate communication will be mailed in due course. **THIS NOTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT RIGHTS.** This application is subject to withdrawal from issue at the initiative of the Office or upon petition by the applicant. See 37 CFR 1.313 and MPEP 1308.

- 1. This communication is responsive to 12/18/07.
- 2. The allowed claim(s) is/are 41-43, 45-58, 62, 85, 88 and 89.
- 3. Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
 - a) All b) Some* c) None of the:
 - 1. Certified copies of the priority documents have been received.
 - 2. Certified copies of the priority documents have been received in Application No. _____.
 - 3. Copies of the certified copies of the priority documents have been received in this national stage application from the International Bureau (PCT Rule 17.2(a)).

* Certified copies not received: _____.

Applicant has THREE MONTHS FROM THE "MAILING DATE" of this communication to file a reply complying with the requirements noted below. Failure to timely comply will result in ABANDONMENT of this application.
THIS THREE-MONTH PERIOD IS NOT EXTENDABLE.

- 4. A SUBSTITUTE OATH OR DECLARATION must be submitted. Note the attached EXAMINER'S AMENDMENT or NOTICE OF INFORMAL PATENT APPLICATION (PTO-152) which gives reason(s) why the oath or declaration is deficient.
 - 5. CORRECTED DRAWINGS (as "replacement sheets") must be submitted.
 - (a) including changes required by the Notice of Draftsperson's Patent Drawing Review (PTO-948) attached
 - 1) hereto or 2) to Paper No./Mail Date _____.
 - (b) including changes required by the attached Examiner's Amendment / Comment or in the Office action of Paper No./Mail Date _____.
- Identifying indicia such as the application number (see 37 CFR 1.84(c)) should be written on the drawings in the front (not the back) of each sheet. Replacement sheet(s) should be labeled as such in the header according to 37 CFR 1.121(d).
- 6. DEPOSIT OF and/or INFORMATION about the deposit of BIOLOGICAL MATERIAL must be submitted. Note the attached Examiner's comment regarding REQUIREMENT FOR THE DEPOSIT OF BIOLOGICAL MATERIAL.

Attachment(s)

- 1. Notice of References Cited (PTO-892)
- 2. Notice of Draftsperson's Patent Drawing Review (PTO-948)
- 3. Information Disclosure Statements (PTO/SB/08),
Paper No./Mail Date 1/17/08
- 4. Examiner's Comment Regarding Requirement for Deposit of Biological Material
- 5. Notice of Informal Patent Application
- 6. Interview Summary (PTO-413),
Paper No./Mail Date _____
- 7. Examiner's Amendment/Comment
- 8. Examiner's Statement of Reasons for Allowance
- 9. Other _____


MICHELLE ESTRADA
PRIMARY EXAMINER

IDS Form PTO/SB/08: Substitute for form 1449A/PTO INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Use as many sheets as necessary)		Complete if Known	
		Application Number Filing Date First Named Inventor Art Unit Examiner Name Attorney Docket Number	10/954,182 October 1, 2004 ZHANG, Hongmei 2823 ESTRADA, Michelle 10655.0016-01
Sheet	1	of	1

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner Initials	Cite No. ¹	Document Number	Issue or Publication Date	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)	MM-DD-YYYY		
<i>[Signature]</i>		US-6,391,166 B1	05-21-2002	Wang	
		US-7,262,131 B2	08-28-2007	Narasimhan et al.	

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

FOREIGN PATENT DOCUMENTS						
Examiner Initials	Cite No. ¹	Foreign Patent Document	Publication Date	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁴
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)	MM-DD-YYYY			
<i>[Signature]</i>		WO 2007/027535 A2	03-08-2007	Symmorphix, Inc.		

NON PATENT LITERATURE DOCUMENTS			
Examiner Initials	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁴
<i>[Signature]</i>		Office Action dated May 21, 2007, in U.S. Application No. 10/291,179 (Attorney Docket No. 9140.0001-00).	
		Final Office Action dated April 13, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Amendment/RCE filed August 9, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Office Action dated September 5, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Final Office Action dated September 7, 2007, in U.S. Application No. 11/100,856 (Attorney Docket No. 9140.0015-01).	
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		Response to Final Office Action filed October 2, 2007, in U.S. Application No. 10/101,863 (Attorney Docket No. 9140.0016-00).	
		Response to Office Action filed July 9, 2007, in U.S. Application No. 10/650,461 (Attorney Docket No. 9140.0025-00).	
		Final Office Action dated October 10, 2007, in U.S. Application No. 10/650,461 (Attorney Docket No. 9140.0025-00).	
		Corrected Notice of Allowance dated June 7, 2007, in U.S. Application No. 11/228,805 (Attorney Docket No. 9140.0030-01).	
		Supplemental Notice of Allowance dated July 5, 2007, in U.S. Application No. 11/228,805 (Attorney Docket No. 9140.0030-01).	
		Second Supplemental Preliminary Amendment filed May 31, 2007, in U.S. Application No. 11/297,057 (Attorney Docket No. 9140.0042-00).	

Examiner Signature	<i>[Signature]</i>	Date Considered	1/22/08
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

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Bib Data Sheet

CONFIRMATION NO. 9873

SERIAL NUMBER 10/954,182	FILING OR 371(c) DATE 10/01/2004 RULE	CLASS 438	GROUP ART UNIT 2823	ATTORNEY DOCKET NO. 10655.0016-01
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APPLICANTS
 Hongmei Zhang, San Jose, CA;
 Mukundan Narasimhan, San Jose, CA;
 Ravi Mullapudi, San Jose, CA;
 Richard E. Demaray, Portola Valley, CA;

**** CONTINUING DATA *******
 This application is a CON of 10/101,863 03/16/2002 *AE*

**** FOREIGN APPLICATIONS *******
none AE

IF REQUIRED, FOREIGN FILING LICENSE GRANTED
 ** 11/20/2004

Foreign Priority claimed <input type="checkbox"/> yes <input checked="" type="checkbox"/> no	STATE OR COUNTRY CA	SHEETS DRAWING 27	TOTAL CLAIMS 45 21	INDEPENDENT CLAIMS 2
35 USC 119 (a-d) conditions met <input type="checkbox"/> yes <input checked="" type="checkbox"/> no <input type="checkbox"/> Met after Nov 2003				
Verified and Acknowledged <i>Mukundan Narasimhan</i> Examiner's Signature Initials				

ADDRESS
 22852

TITLE
 Biased pulse DC reactive sputtering of oxide films

FILING FEE RECEIVED 7000	FEES: Authority has been given in Paper No. _____ to charge/credit DEPOSIT ACCOUNT No. _____ for following:	<input type="checkbox"/> All Fees
		<input type="checkbox"/> 1.16 Fees (Filing)
		<input type="checkbox"/> 1.17 Fees (Processing Ext. of time)
		<input type="checkbox"/> 1.18 Fees (Issue)
		<input type="checkbox"/> Other _____
		<input type="checkbox"/> Credit

Issue Classification



Application/Control No.

10954182

Applicant(s)/Patent Under Reexamination

ZHANG ET AL.

Examiner


Estrada, Michelle

Art Unit

2823

ORIGINAL						INTERNATIONAL CLASSIFICATION														
CLASS		SUBCLASS				CLAIMED				NON-CLAIMED										
438		769				H	0	1	L	21 / 31 (2006.01.01)										
CROSS REFERENCE(S)						H	0	1	L	21 / 469 (2006.01.01)										
						CLASS						SUBCLASS (ONE SUBCLASS PER BLOCK)								
438		770	771																	
257		E21.091	E21.169	E21.2	E21.462	E23.132														


NONE (Assistant Examiner)	(Date)	<i>Michelle Estrada</i> MICHELLE ESTRADA PRIMARY EXAMINER	Total Claims Allowed: 21	
<i>N. Steptoe</i> (Legal Instruments Examiner)	1/24/08 (Date)	Estrada, Michelle (Primary Examiner)	1/22/08 (Date)	O.G. Print Claim(s) 1
				O.G. Print Figure 1A

Search Notes 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

SEARCHED			
Class	Subclass	Date	Examiner
Updated as before		11/6/07	ME
257	E23.132,E21.091,E21.169,E21.2,E21.462	11/6/07	ME
Updated as above		1/22/08	ME

SEARCH NOTES		
Search Notes	Date	Examiner
See East search attached	11/6/07	ME
google search	11/5/07	ME


INTERFERENCE SEARCH			
Class	Subclass	Date	Examiner
PG Pub text search		1/22/08	ME

Index of Claims 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

✓	Rejected	-	Cancelled	N	Non-Elected	A	Appeal
=	Allowed	÷	Restricted	I	Interference	O	Objected

Claims renumbered in the same order as presented by applicant
 CPA
 T.D.
 R.1.47

CLAIM		DATE							
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Index of Claims 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

✓	Rejected
=	Allowed


-	Cancelled
÷	Restricted

N	Non-Elected
I	Interference

A	Appeal
O	Objected

Claims renumbered in the same order as presented by applicant
 CPA
 T.D.
 R.1.47

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Index of Claims 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

✓	Rejected
=	Allowed


-	Cancelled
÷	Restricted

N	Non-Elected
I	Interference

A	Appeal
O	Objected

Claims renumbered in the same order as presented by applicant
 CPA
 T.D.
 R.1.47

CLAIM		DATE							
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	108	✓	-	-					

Index of Claims 	Application/Control No. 10954182	Applicant(s)/Patent Under Reexamination ZHANG ET AL.
	Examiner Estrada, Michelle	Art Unit 2823

✓	Rejected
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-	Cancelled
÷	Restricted

N	Non-Elected
I	Interference

A	Appeal
O	Objected

Claims renumbered in the same order as presented by applicant
 CPA
 T.D.
 R.1.47

CLAIM		DATE							
Final	Original	07/16/2007	11/09/2007	01/22/2008					
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02-1508

IFW\$



PATENT
Customer No. 22,852
Attorney Docket No. 10655.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
)	
ZHANG, Hongmei et al.)	Group Art Unit: 2823
)	
Application No.: 10/954,182)	Examiner: ESTRADA, Michelle
)	
Filed: October 1, 2004)	
)	Confirmation No.: 9873
For: BIASED PULSE DC REACTIVE)	
SPUTTERING OF OXIDE FILMS)	

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT
UNDER 37 C.F.R. § 1.97(d)

Pursuant to 37 C.F.R. §§ 1.56 and 1.97(d), Applicants brings to the attention of the Examiner the documents on the attached listing. This Supplemental Information Disclosure Statement is being filed after a Notice of Allowance but before payment of the issue fee and the Commissioner is authorized to charge the fee of \$180.00 to Deposit Account No. 06-0916, as specified under § 1.17(p) and a statement as specified under § 1.97(e).

Based on reasonable inquiry, no document listed in this Supplemental Information Disclosure Statement was cited in a communication from a foreign patent office in a counterpart foreign application, and no document listed in this Supplemental Information Disclosure Statement was known to any individual designated in 37 C.F.R. § 1.56(c) more than three months prior to the filing date of this Supplemental Information Disclosure Statement.

02/15/2008 WASFAW1 00000036 060916 10954182

01 FC:1806 180.00 DA

Copies of the listed non-patent literature documents are attached. Applicants respectfully request that the Examiner consider the listed documents and indicate that they were considered by making appropriate notations on the attached form.

This submission does not represent that a search has been made or that no better art exists and does not constitute an admission that each or all of the listed documents are material or constitute "prior art." If the Examiner applies any of the documents as prior art against any claims in the application and Applicants determine that the cited documents do not constitute "prior art" under United States law, Applicants reserve the right to present to the office the relevant facts and law regarding the appropriate status of such documents.

Applicants further reserve the right to take appropriate action to establish the patentability of the disclosed invention over the listed documents, should one or more of the documents be applied against the claims of the present application.

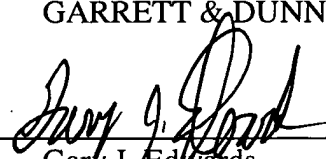
If there is any additional fee due in connection with the filing of this Statement, please charge the fee to Deposit Account 06-0916.

Respectfully submitted,

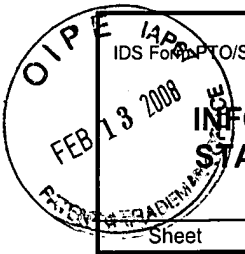
FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Dated: February 13, 2008

By: _____


Gary J. Edwards
Reg. No. 41,008
(650) 849-6622

**EXPRESS MAIL LABEL NO.
EM 074697408 US**



IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>			<i>Application Number</i>	10/954,182
			<i>Filing Date</i>	October 1, 2004
			<i>First Named Inventor</i>	Hongmei ZHANG
			<i>Art Unit</i>	2823
			<i>Examiner Name</i>	Michelle ESTRADA
			<i>Attorney Docket Number</i>	10655.0016-01
Sheet	1	of	1	

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner Initials ¹	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
		US-			

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

FOREIGN PATENT DOCUMENTS						
Examiner Initials ¹	Cite No. ¹	Foreign Patent Document	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁶
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)				

NONPATENT LITERATURE DOCUMENTS			
Examiner Initials ¹	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
		Response to Office Action dated November 21, 2007, in U.S. Appl. No. 10/291,179 (Attorney Docket No. 9140.0001-00).	
		Response to Office Action dated December 5, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Final Office Action dated January 29, 2008, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Office Action dated January 25, 2008, in U.S. Application No. 11/100,856 (Attorney Docket No. 9140.0015-01).	
		Office Action dated November 15, 2007, in U.S. Application No. 10/101,863 (Attorney Docket No. 9140.0016-00).	
		Response to Office Action dated December 18, 2007, in U.S. Application No. 10/101,863 (Attorney Docket No. 9140.0016-00).	
		Notice of Allowance dated January 25, 2008, in U.S. Application No. 10/101,863 (Attorney Docket No. 9140.0016-00).	
		Office Action dated November 16, 2007, in U.S. Application No. 10/650,461 (Attorney Docket No. 10655.0025-00).	

Examiner Signature		Date Considered	
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

EXPRESS MAIL LABEL NO.
EM 074697408 US



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/954,182	10/01/2004	Hongmei Zhang	10655.0016-01	9873
22852	7590	03/17/2008	EXAMINER	
FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER LLP 901 NEW YORK AVENUE, NW WASHINGTON, DC 20001-4413			ESTRADA, MICHELLE	
			ART UNIT	PAPER NUMBER
			2823	
			MAIL DATE	DELIVERY MODE
			03/17/2008	PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.



UNITED STATES DEPARTMENT OF COMMERCE

U.S. Patent and Trademark Office

Address : COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450

APPLICATION NO./ CONTROL NO.	FILING DATE	FIRST NAMED INVENTOR / PATENT IN REEXAMINATION	ATTORNEY DOCKET NO.
10954182	10/1/04	ZHANG ET AL.	10655.0016-01

FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER
LLP
901 NEW YORK AVENUE, NW
WASHINGTON, DC 20001-4413

EXAMINER

Michelle Estrada

ART UNIT	PAPER
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2823

20080304

DATE MAILED:

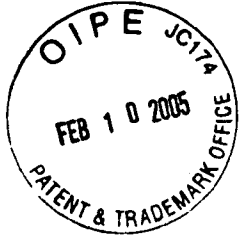
Please find below and/or attached an Office communication concerning this application or proceeding.

Commissioner for Patents

Attached are the IDSs filed 2/10/05, 3/2/06 and 2/13/08 that have been considered.

/Michelle Estrada/
Primary Examiner, Art Unit 2823

JFW



PATENT
Customer No. 22,852
Attorney Docket No. 09140-0016-01000

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
)	
ZHANG et al.)	Group Art Unit: 2882
)	
Application No.: 10/954,182)	Examiner: Not Yet Assigned
)	
Filed: October 1, 2004)	Confirmation No.: 9873
)	
For: BIASED PULSE DC REACTIVE)	
SPUTTERING OF OXIDE FILMS)	

Mail Stop AMENDMENTS
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT
UNDER 37 C.F.R. § 1.97(b)

Pursuant to 37 C.F.R. §§ 1.56 and 1.97(b), Applicants brings to the attention of the Examiner the documents listed on the attached PTO 1449. This Information Disclosure Statement is being filed within three months of the filing date of the above-referenced application.

These documents include U.S. patents and applications that are possibly related to the pending application by subject matter, as summarized in the chart below. This submission should not be construed, however, as an admission of relatedness.

U.S. Patent Application No. 10/954,182

Attorney Docket No. 09140-0016-01

Customer No. 22,852

Attorney Docket Number	U.S. Patent/ Serial No.	U.S./PCT Publication No.	Title	Examiner
09140-0001-00	10/291,179	US 2003/0134054 A1	Low temperature zirconia based thermal barrier layer by PVD	Rodney McDONALD
09140-0002-01	6,506,289	US 2002/0033330 A1	Planar optical devices and methods for their manufacture	Steven H. VERSTEEG
09140-0002-02	6,827,826	US 2003/0127319 A1	Planar optical devices and methods for their manufacture	Steven H. VERSTEEG
09140-0004-00	6,533,907	US 2002/0134671 A1	Method of Producing amorphous silicon for hard mask and waveguide applications	Steven H. VERSTEEG
09140-0014-00	09/903,081	US 2003/0063883 A1	As-deposited planar optical waveguides with low scattering loss and methods for their manufacture	John M. HOFFMANN
09140-0015-00	10/101,492	US 2003/0173208 A1	Mode size converter for a planar waveguide	Steven H. VERSTEEG
09140-0016-00	10/101,863	US 2003/0173207 A1	Biased pulse DC reactive sputtering of oxide films	Michelle ESTRADA
09140-0016-01 (present application)	10/954,182		Biased pulse DC reactive sputtering of oxide films	Not Yet Assigned
09140-0017-00	10/101,341	US 2003/0175142 A1	Rare-earth pre-alloyed PVD targets for dielectric planar applications	Daniel J. JENKINS
09140-0021-00 (abandoned)	10/101,493	US 2003/0174391 A1	Gain flattened optical amplifier	Deandra M. HUGHES
09140-0025-00	10/650,461	US 2004/0105644 A1 WO 2004/021532 A1	Optical Coupling into Highly Uniform Waveguides	Frank G. FONT
09140-0030-00	10/789,953	US 2005/0006768 A1 WO 2004/077519 A2	Dielectric Barrier Films	Not Yet Assigned

U.S. Patent Application No. 10/954,182
 Attorney Docket No. 09140-0016-01
 Customer No. 22,852

Attorney Docket Number	U.S. Patent/ Serial No.	U.S./PCT Publication No.	Title	Examiner
09140-0033-00	10/851,542	US 2004/0259305 A1	Energy Conversion and Storage Devices by Physical Vapor Deposition of Titanium and Titanium Oxides and Sub-Oxides	Not Yet Assigned
09140-0034-00	10/850,968	US 2005/0000794 A1	Transparent Conductive Oxides from a Metallic Target	Not Yet Assigned

Copies of U.S. Patents and U.S. Patent Publications are not provided. Copies of foreign patent documents and non-patent literature documents are included herewith.

Applicants submit copies of Office Actions issued by the U.S. Patent and Trademark Office in the above-listed applications and Applicants' responses to these office actions.

Applicants also submit International Search Reports and Written Opinions issued in the Patent Cooperation Treaty applications corresponding to the U.S. Patent Applications listed above.

Applicants respectfully request that the Examiner consider the listed documents and indicate that they were considered by making appropriate notations on the attached form.

This submission does not represent that a search has been made or that no better art exists and does not constitute an admission that each or all of the listed documents are material or constitute "prior art." If the Examiner applies any of the documents as prior art against any claim in the application and Applicants determine that the cited documents do not constitute "prior art" under United States law, Applicants reserve the right to present to the Office the relevant facts and law regarding the appropriate status of such documents.

U.S. Patent Application No. 10/954,182

Attorney Docket No. 09140-0016-01

Customer No. 22,852

Applicants further reserve the right to take appropriate action to establish the patentability of the disclosed invention over the listed documents, should one or more of the documents be applied against the claims of the present application.

If there is any fee due in connection with the filing of this Statement, please charge the fee to our Deposit Account No. 06-0916.

Respectfully submitted,

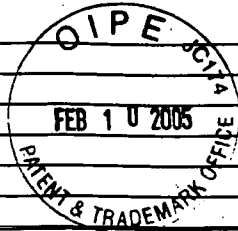
FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Dated: February 9, 2005

By:  _____

Gary J. Edwards

Reg. No. 41,008

IDS Form PTO/SB/08: Substitute for form 1449A/PTO INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Use as many sheets as necessary)			Complete if Known	
			Application Number	10/954,182
Sheet 1 of 9			Filing Date	October 1, 2004
			First Named Inventor	Zhang et al.
			Art Unit	2882
			Examiner Name	Not Yet Assigned
			Attorney Docket Number	09140-0016-01000
				

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner Initials	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
		US 2001/0027159 A1	Oct. 4, 2001	Kaneyoshi	
		US 2002/0033330 A1	Mar. 21, 2002	Demaray et al.	
		US 2002/0134671 A1	Jul. 17, 2003	Demaray et al.	
		US 2002/0170821 A1	Nov. 21, 2002	Sandlin et al.	
		US 2003/0097858 A1	May 29, 2003	Strohhofer et al.	
		US 2003/0127319 A1	Jul. 10, 2003	Demaray et al.	
		US 2003/0134054 A1	Jul. 17, 2003	Demaray et al.	
		US 2003/0173207 A1	Sep. 18, 2003	Zhang et al.	
		US 2003/0173208 A1	Sep. 18, 2003	Pan et al.	
		US 2003/0174391 A1	Sep. 18, 2003	Pan et al.	
		US 2004/0105644 A1	Jun. 3, 2004	Dawes	
		US 2004/0259305 A1	Dec. 23, 2004	Demaray et al.	
		US 2005/0000794 A1	Jan. 6, 2005	Demaray et al.	
		US 2005/0006768 A1	Jan. 13, 2005	Narasimhan et al.	
		US 5,085,904	Feb. 4, 1992	Deak et al.	
		US 5,107,538	Apr. 21, 1992	Benton et al.	
		US 5,119,460	Jun. 2, 1992	Bruce et al.	
		US 5,306,569	Apr. 26, 1994	Hiraki	
		US 5,355,089	Oct. 11, 1994	Treger	
		US 5,457,569	Oct. 10, 1995	Liou et al.	
		US 5,499,207	Mar. 12, 1996	Miki et al.	
		US 5,563,979	Oct. 8, 1996	Bruce et al.	
		US 5,591,520	Jan. 7, 1997	Migliorini et al.	
		US 5,607,789	Mar. 4, 1997	Treger et al.	
		US 5,654,984	Aug. 5, 1997	Hershbarger et al.	
		US 5,686,360	Nov. 11, 1997	Harvey, III et al.	
		US 5,689,522	Nov. 18, 1997	Beach	
		US 5,731,661	Mar. 24, 1998	So et al.	
		US 5,757,126	May 26, 1998	Harvey, III et al.	
		US 5,762,768	Jun. 9, 1998	Goy et al.	
		US 5,771,562	Jun. 30, 1998	Harvey, III et al.	

Examiner Signature	/Michelle Estrada/	Date Considered	03/04/2008
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /ME/
 Ex. 1004, Page 1010

IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known	
			<i>Application Number</i>	10/954,182
INFORMATION DISCLOSURE STATEMENT BY APPLICANT			<i>Filing Date</i>	October 1, 2004
			<i>First Named Inventor</i>	Zhang et al.
			<i>Art Unit</i>	2882
			<i>Examiner Name</i>	Not Yet Assigned
			<i>Attorney Docket Number</i>	09140-0016-01000
			<i>(Use as many sheets as necessary)</i>	
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U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner Initials	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
		US 5,811,177	Sep. 22, 1998	Shi et al.	
		US 5,831,262	Nov. 3, 1998	Greywall et al.	
		US 5,853,830	Dec. 29, 1998	McCaulley et al.	
		US 5,870,273	Feb. 9, 1999	Sogabe et al.	
		US 5,882,946	Mar. 16, 1999	Otani	
		US 5,900,057	May. 4, 1999	Buchal et al.	
		US 5,930,584	Jul. 27, 1999	Sun et al.	
		US 5,952,778	Sep. 14, 1999	Haskal et al.	
		US 5,966,491	Oct. 12, 1999	DiGiovanni	
		US 6,004,660	Dec. 21, 1999	Topolski et al.	
		US 6,046,081	Apr. 4, 2000	Kuo	
		US 6,051,296	Apr. 18, 2000	McCaulley et al.	
		US 6,052,397	Apr. 18, 2000	Jeon et al.	
		US 6,058,233	May 2, 2000	Dragone	
		US 6,071,323	Jun. 6, 2000	Kawaguchi	
		US 6,077,642	Jun. 20, 2000	Ogata et al.	
		US 6,080,643	Jun. 27, 2000	Noguchi et al.	
		US 6,106,933	Aug. 22, 2000	Nagai et al.	
		US 6,146,225	Nov. 14, 2000	Sheats et al.	
		US 6,157,765	Dec. 5, 2000	Bruce et al.	
		US 6,165,566	Dec. 26, 2000	Tropsha	
		US 6,197,167 B1	Mar. 6, 2001	Tanaka	
		US 6,198,217 B1	Mar. 6, 2001	Suzuki et al.	
		US 6,204,111 B1	Mar. 20, 2001	Uemoto et al.	
		US 6,210,544 B1	Apr. 3, 2001	Sasaki	
		US 6,214,660 B1	Apr. 10, 2001	Uemoto et al.	
		US 6,248,640 B1	Jun. 19, 2001	Nam	
		US 6,261,917 B1	Jul. 17, 2001	Quek et al.	
		US 6,300,215 B1	Oct. 9, 2001	Shin	
		US 6,302,939 B1	Oct. 16, 2001	Rabin et al.	
		US 6,365,319 B1	Apr. 2, 2002	Heath et al.	

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IDS Form PTO/SB/08: Substitute for form 1449A/PTO INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Use as many sheets as necessary)			Complete if Known				
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			Filing Date		October 1, 2004		
			First Named Inventor		Zhang et al.		
			Art Unit		2882		
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		US 6,413,645 B1	Jul. 2, 2002	Graff et al.	
		US 6,416,598 B1	Jul. 9, 2002	Sircar	
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		US 6,444,750 B1	Sep. 3, 2002	Touhsaent	
		US 6,506,289 B1	Jan. 14, 2003	Demaray et al.	
		US 6,533,907 B1	Mar. 18, 2003	Demaray et al.	
		US 6,576,546 B2	Jun. 10, 2003	Gilbert et al.	
		US 6,750,156 B2	Jun. 15, 2004	Le et al.	
		US 6,827,826 B1	Dec. 7, 2004	Demaray et al.	

Note: Copies of the U.S. Patent Documents are not Required in IDS filed after October 21, 2004

FOREIGN PATENT DOCUMENTS						
Examiner Initials	Cite No.	Foreign Patent Document	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁶
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)				
		EP 0 510 883 A2	10/28/1992	AT&T		
		EP 1 189 080 A2	03/20/2002	Agere Systems Optoelectronics Guardian Corporation		
		JP 2-054764 A2	02/23/1990	Leybold AG		Abstract
		WO 2004/021532 A1	03/11/2004	Symmorphix, Inc.		
		WO 2004/077519 A2	09/10/2004	Narasimhan et al.		

NON PATENT LITERATURE DOCUMENTS			
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		AFFINITO et al., "PML/oxide/PML Barrier Layer Performance Differences Arising from Use of UV or Electron Beam Polymerization of the PML Layers," <i>Thin Solid Films</i> Vol. 308-309, pp. 19-25 (1997)	
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		ALMEIDA, Vilson R. et al., "Nanotaper for compact mode conversion," <i>Optics Letters</i> , 28(15):1302-1304, (2003).		
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		BEACH R.J., "Theory and optimization of lens ducts," <i>Applied Optics</i> , 35:12:2005-15 (1996).		
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		BORSELLA et al., "Structural incorporation of silver insoda-lime glass by the ion-exchange process: a photoluminescence spectroscopy study", <i>Applied Physics A</i> 71, pp. 125-132 (2000).		
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		GARCIA, C. "Size Dependence of Lifetime and Absorption Cross Section of Si Nanocrystals Embedded in SiO ₂ ," <i>Appl. Phys. Lett.</i> , Vol. 82, No. 10, pp. 1595-7 (March 10, 2003).		
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			Filing Date	October 1, 2004
			First Named Inventor	Zhang et al.
			Art Unit	2882
			Examiner Name	Not Yet Assigned
			Attorney Docket Number	09140-0016-01000
Sheet	5	of	9	

NON PATENT LITERATURE DOCUMENTS			
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		HAN, Hak-Seung et al. "Optical Gain at 1.54 μ m in Erbium-Doped Silicon Nanocluster Sensitized Waveguide," <i>Appl. Phys. Lett.</i> , Vol. 79, No. 27, pp. 4568-70 (December 31, 2001).	
		HAYAKAWA et al, "Field enhancement effect of small Ag particles on the fluorescence from Eu ³⁺ -doped SiO ₂ glass", <i>Appl. Phys. Lett.</i> , Vol. 74, No 11, pp. 1513-1515 (15 March 1999).	
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Examiner Signature	/Michelle Estrada/	Date Considered	03/04/2008
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IDS Form PTO/SB/08: Substitute for form 1449A/PTO				Complete if Known	
				<i>Application Number</i>	10/954,182
INFORMATION DISCLOSURE STATEMENT BY APPLICANT				<i>Filing Date</i>	October 1, 2004
				<i>First Named Inventor</i>	Zhang et al.
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		LADOUCEUR, F. et al., "Effect of side wall roughness in buried channel waveguides," <i>IEEE Proc.</i> , vol. 141, pp. 242-248 (Aug. 1994).		
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Examiner Signature	/Michelle Estrada/		Date Considered	03/04/2008

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		OHTSUKI et al., "Gain Characteristics of a high concentration Er ³⁺ -doped phosphate glass waveguide", <i>J. Appl. Phys.</i> 78(6), pp. 3617-3621 (1995).	
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			03/04/2008

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		VON ROTTKAY et al. "Influence of stoichiometry on electrochromic cerium-titanium oxide compounds," Lawrence Berkeley National Laboratory, UC Berkeley, CA, (date unknown).	
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		YANAGAWA, H. et al., "Index-and-Dimensional Taper and Its Application to Photonic Devices," <i>Journal of Lightwave Technology</i> , 10(5):587-591 (1992).	
		Office Action issued on September 27, 2004 in U.S. Serial No. 10/291,179 (Attorney Docket No. 09140-0001-00).	
		Office Action issued on November 28, 2001 in U.S. Patent No. 6,506,289 (Attorney Docket No. 09140-0002-01).	
		Response to Office Action filed on February 20, 2002 in U.S. Patent No. 6,506,289 (Attorney Docket No. 09140-0002-01).	
		Office Action issued on April 17, 2002 in U.S. Patent No. 6,506,289 (Attorney Docket No. 09140-0002-01).	
		Response to Office Action filed on July 17, 2002 in U.S. Patent No. 6,506,289 (Attorney Docket No. 09140-0002-01).	
		Quayle Action issued on November 10, 2003 in U.S. Patent No. 6,827,826 (Attorney Docket No. 09140-0002-02).	
		Office Action issued on May 2, 2002 in U.S. Patent No. 6,533,907 (Attorney Docket No. 09140-0004-00).	
		Response to Office Action filed on September 3, 2002 in U.S. Patent No. 6,533,907 (Attorney Docket No. 09140-0004-00).	
		Office Action issued on February 12, 2004 in U.S. Serial No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
		Response to Office Action filed on August 10, 2004 in U.S. Serial No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
		Office Action issued on September 10, 2004 in U.S. Serial No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
		Office Action issued on May 14, 2003 in U.S. Serial No. 10/101,492 (Attorney Docket No. 09140-0015-00).	
		Response to Office Action filed on August 14, 2003 in U.S. Serial No. 10/101,492 (Attorney Docket No. 09140-0015-00).	
		Office Action issued on September 3, 2003 in U.S. Serial No. 10/101,492 (Attorney Docket No. 09140-0015-00).	
		Response to Office Action filed on March 3, 2004 in U.S. Serial No. 10/101,492 (Attorney Docket No. 09140-0015-00).	

Examiner Signature	/Michelle Estrada/	Date Considered	03/04/2008
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /ME/
Ex. 1004, Page 1017

IDS Form PTO/SB/08: Substitute for form 1449A/PTO INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Use as many sheets as necessary)			Complete if Known	
			Application Number	10/954,182
Sheet 9 of 9			Filing Date	October 1, 2004
			First Named Inventor	Zhang et al.
			Art Unit	2882
			Examiner Name	Not Yet Assigned
			Attorney Docket Number	09140-0016-01000

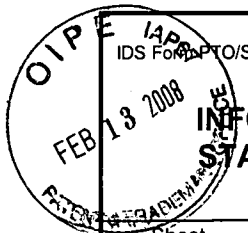
NON PATENT LITERATURE DOCUMENTS			
Examiner Initials	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
		Office Action issued on February 24, 2004 in U.S. Serial No. 10/101,863 (Attorney Docket No. 09140-0016-00).	
		Response to Office Action filed on July 23, 2004 in U.S. Serial No. 10/101,863 (Attorney Docket No. 09140-0016-00).	
		Office Action issued on October 6, 2004 in U.S. Serial No. 10/101,863 (Attorney Docket No. 09140-0016-00).	
		Office Action issued on October 22, 2003 in U.S. Serial No. 10/101,341 (Attorney Docket No. 09140-0017-00).	
		Response to Office Action filed on February 23, 2004 in U.S. Serial No. 10/101,341 (Attorney Docket No. 09140-0017-00).	
		Office Action issued on June 10, 2004 in U.S. Serial No. 10/101,341 (Attorney Docket No. 09140-0017-00).	
		Response to Office Action filed on December 08, 2004 in U.S. Serial No. 10/101,341 (Attorney Docket No. 09140-0017-00).	
		Office Action issued on May 4, 2004 in U.S. Serial No. 10/101,493 (Attorney Docket No. 09140-0021-00).	
		International Search Report issued on November 21, 2003 in PCT/US03/24809 (Attorney Docket No. 09140-0025-00).	
		International Search Report issued on October 11, 2004 in PCT/US2004/005531 (Attorney Docket No. 09140-0030-00)	
		Written Opinion issued on October 11, 2004 in PCT/US2004/005531 (Attorney Docket No. 09140-0030-00)	

Examiner Signature	/Michelle Estrada/	Date Considered	03/04/2008
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next application submission to applicant.

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Ex. 1004, Page 1018



IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>			Application Number	10/954,182
			Filing Date	October 1, 2004
			First Named Inventor	Hongmei ZHANG
			Art Unit	2823
			Examiner Name	Michelle ESTRADA
			Attorney Docket Number	10655.0016-01
Sheet	1	of	1	

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner Initials ¹	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
		US-			

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

FOREIGN PATENT DOCUMENTS						
Examiner Initials ¹	Cite No. ¹	Foreign Patent Document	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁶
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)				

NONPATENT LITERATURE DOCUMENTS			
Examiner Initials ¹	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
		Response to Office Action dated November 21, 2007, in U.S. Appl. No. 10/291,179 (Attorney Docket No. 9140.0001-00).	
		Response to Office Action dated December 5, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Final Office Action dated January 29, 2008, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).	
		Office Action dated January 25, 2008, in U.S. Application No. 11/100,856 (Attorney Docket No. 9140.0015-01).	
		Office Action dated November 15, 2007, in U.S. Application No. 10/101,863 (Attorney Docket No. 9140.0016-00).	
		Response to Office Action dated December 18, 2007, in U.S. Application No. 10/101,863 (Attorney Docket No. 9140.0016-00).	
		Notice of Allowance dated January 25, 2008, in U.S. Application No. 10/101,863 (Attorney Docket No. 9140.0016-00).	
		Office Action dated November 16, 2007, in U.S. Application No. 10/650,461 (Attorney Docket No. 10655.0025-00).	

Examiner Signature	/Michelle Estrada/	Date Considered	03/04/2008
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PATENT
Customer No. 22,852
Attorney Docket No. 9140.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
)	
ZHANG, Hongmei et al.)	Group Art Unit: 2823
)	
Application No.: 10/954,182)	Examiner: ESTRADA, Michelle
)	
Filed: October 1, 2004)	
)	Confirmation No.: 9873
For: BIASED PULSE DC REACTIVE)	
SPUTTERING OF OXIDE FILMS)	

MAIL STOP AMENDMENT
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

FOURTH SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT
UNDER 37 C.F.R. § 1.97(c)

Pursuant to 37 C.F.R. §§ 1.56 and 1.97(c), Applicants bring to the attention of the Examiner the documents on the attached listing. This Information Disclosure Statement is being filed after the events recited in Section 1.97(b) but, to the undersigned's knowledge, before the mailing date of either a Final action, Quayle action, or a Notice of Allowance. Under the provisions of 37 C.F.R. § 1.97(c), the Commissioner is hereby authorized to charge the fee of \$180.00 to Deposit Account No. 06-0916 as specified by Section 1.17(p).

Copies of the listed non-patent literature documents are attached. Copies of the U.S. patents and patent publications are not enclosed.

Applicants respectfully request that the Examiner consider the listed documents and indicate that they were considered by making appropriate notations on the attached form.

This submission does not represent that a search has been made or that no better art exists and does not constitute an admission that each or all of the listed documents are material or constitute "prior art." If the Examiner applies any of the documents as prior art against any claims in the application and Applicants determine that the cited documents do not constitute "prior art" under United States law, Applicants reserve the right to present to the office the relevant facts and law regarding the appropriate status of such documents.

Applicants further reserve the right to take appropriate action to establish the patentability of the disclosed invention over the listed documents, should one or more of the documents be applied against the claims of the present application.

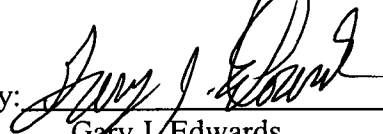
If there is any fee due in connection with the filing of this Statement, please charge the fee to our Deposit Account No. 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Dated: March 2, 2006

By:

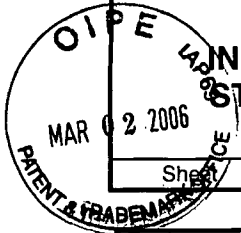


Gary L. Edwards
Reg. No. 41,008

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IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>			Application Number	10/954,182
			Filing Date	October 1, 2004
			First Named Inventor	Hongmei ZHANG
			Art Unit	2823
			Examiner Name	ESTRADA, Michelle
			Attorney Docket Number	9140.0016-01
Sheet	1	of	3	



U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS

Examiner Initials*	Cite No. ¹	Document Number	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)			
		US 3,309,302	03-14-1967	Heil	
		US 5,338,625	08-16-1994	Bates et al.	
		US 5,561,004	10-01-1996	Bates et al.	
		US 5,909,346	06-01-1999	Malhotra et al.	
		US 5,930,046	07-27-1999	Solberg et al.	
		US 6,000,603	12-14-1999	Koskenmaki et al.	
		US 6,133,670	10-17-2000	Rodgers et al.	
		US 6,242,129 B1	06-05-2001	Johnson	
		US 6,280,875 B1	08-28-2001	Kwak et al.	
		US 6,290,821 B1	09-18-2001	McLeod	
		US 6,356,694 B1	03-12-2002	Weber	
		US 6,376,027 B1	04-23-2002	Lee et al.	
		US 6,632,563 B1	10-14-2003	Krasnov et al.	
		US 6,768,855 B1	07-27-2004	Bakke et al.	
		US 6,683,244 B2	01-27-2004	Fujimori et al.	
		US 6,683,749 B2	01-27-2004	Daby et al.	
		US 2002/0001746 A1	01-03-2002	Jenson	
		US 2002/0076133 A1	06-20-2002	Li et al.	
		US 2002/0140103 A1	10-03-2002	Kloster et al.	

Note: Submission of copies of U.S. Patents and published U.S. Patent Applications is not required.

FOREIGN PATENT DOCUMENTS

Examiner Initials*	Cite No. ¹	Foreign Patent Document	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation ⁶
		Country Code ³ Number ⁴ Kind Code ⁵ (if known)				
		WO 2004/106581 A2	12-09-2004	Symmorphix, Inc.		
		WO 2004/106582 A2	12-09-2004	Symmorphix, Inc.		

NON PATENT LITERATURE DOCUMENTS

Examiner Initials*	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or	Translation ⁶

Examiner Signature	/Michelle Estrada/	Date Considered	03/04/2008
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EXPRESS MAIL NO. EV 860819695 US

IDS Form PTO/SB/08: Substitute for form 1449A/PTO INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Use as many sheets as necessary)			Complete if Known	
			Application Number	10/954,182
			Filing Date	October 1, 2004
			First Named Inventor	Hongmei ZHANG
			Art Unit	2823
			Examiner Name	ESTRADA, Michelle
			Attorney Docket Number	9140.0016-01
Sheet	2	of	3	

NON PATENT LITERATURE DOCUMENTS			
		country where published.	
		KIM, H-K. and YOON, Y., "Characteristics of rapid-thermal-annealed LiCoO ₂ cathode film for an all-solid-state thin film microbattery," <i>J. Vac. Sci. Technol. A</i> 22(4):1182-1187 (2004).	
		Response to Office Action filed on October 17, 2005 in U.S. Application No. 10/291,179 (Attorney Docket No. 09140-0001-00).	
		Final Office Action issued on December 14, 2005 in U.S. Application No. 10/291,179 (Attorney Docket No. 09140-0001-00).	
		PCT Invitation to Pay Additional Fees for PCT/US01/22750, dated March 13, 2002 (Attorney Docket No. 09140.0002-00304).	
		PCT International Search Report for PCT/US01/22750, dated July 19, 2002 (Attorney Docket No. 09140.0002-00304).	
		PCT Written Opinion for PCT/US01/22750, dated July 23, 2002 (Attorney Docket No. 09140.0002-00304).	
		PCT International Preliminary Examination Report for PCT/US01/22750, dated October 8, 2002 (Attorney Docket No. 09140.0002-00304).	
		Amendment/RCE filed on March 10, 2005 in U.S. Application No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
		Office Action issued on November 28, 2005 in U.S. Application No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
		Response to Office Action filed February 17, 2006 in U.S. Application No. 11/100,856 (Attorney Docket No. 09140.0015-01).	
		Response to Office Action filed December 5, 2005, in U.S. Application No. 10/101,863 (Attorney Docket No. 09140.0016-00).	
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		Response to Office Action filed February 24, 2006, in U.S. Application No. 10/101,863 (Attorney Docket No. 09140.0016-00).	
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		Office Action issued on February 13, 2006, in U.S. Application No. 10/101,341 (Attorney Docket No. 09140-0017-00).	
		Response to Office Action filed on January 3, 2006 in U.S. Application No. 10/650,461 (Attorney Docket No. 09140-0025-00).	
		International Preliminary Examination Report mailed on April 15, 2004 in PCT/US03/24809 (Attorney Docket No. 09140-0025-00304).	
		Office Action issued on December 2, 2005 in U.S. Application No. 10/789,953 (Attorney Docket No. 09140.0030-00).	
		Specification and Preliminary Amendment as filed for U.S. Appl. No. 11/297,057 (Attorney Docket No. 09140.0030-01).	
		Response to Office Action filed January 19, 2006 in U.S. Application No. 10/851,542 (Attorney Docket No. 09140.0033-00).	

Examiner Signature	/Michelle Estrada/	Date Considered	03/04/2008
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IDS Form PTO/SB/08: Substitute for form 1449A/PTO				Complete if Known	
				<i>Application Number</i>	10/954,182
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>				<i>Filing Date</i>	October 1, 2004
				<i>First Named Inventor</i>	Hongmei ZHANG
				<i>Art Unit</i>	2823
				<i>Examiner Name</i>	ESTRADA, Michelle
				<i>Attorney Docket Number</i>	9140.0016-01
				Sheet	3

NON PATENT LITERATURE DOCUMENTS			
		PCT International Search Report and Written Opinion for Application No. PCT/US2004/014524 dated March 2, 2005 (Attorney Docket No. 09140.0033-00304).	
		PCT International Preliminary Report on Patentability for Application No. PCT/US2004/014524, dated December 8, 2005 (Attorney Docket No. 09140.0033-00304).	
		PCT International Search Report for Application No. PCT/US2004/014523 dated January 17, 2005 (Attorney Docket No. 09140.0034-00304).	
		PCT Written Opinion for Application No. PCT/US2004/014523 dated January 17, 2005 (Attorney Docket No. 09140.0034-00304).	
		PCT International Preliminary Report on Patentability for Application No. PCT/US2004/014523, dated December 8, 2005 (Attorney Docket No. 09140.0034-00304).	
		Specification as filed for U.S. Appl. No. 11/297,057 (Attorney Docket No. 09140.0042-00).	

Examiner Signature	/Michelle Estrada/	Date Considered	03/04/2008
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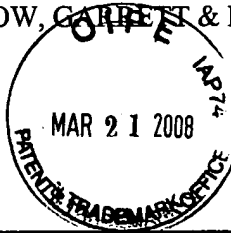
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22852 7590 02/01/2008

FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER
 LLP
 901 NEW YORK AVENUE, NW
 WASHINGTON, DC 20001-4413



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 Express Mail Label No.: EM 074697252 US

Signed: Annie Wong
 Annie Wong

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/954,182	10/01/2004	Hongmei Zhang	10655.0016-01	9873

TITLE OF INVENTION: BIASED PULSE DC REACTIVE SPUTTERING OF OXIDE FILMS

APPLN. TYPE	SMALL ENTITY	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE FEE	TOTAL FEE(S) DUE	DATE DUE
nonprovisional	NO	\$1440	\$300	\$0	\$1740	05/01/2008

EXAMINER	ART UNIT	CLASS-SUBCLASS
ESTRADA, MICHELLE	2823	438-002000

1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363).

- Change of correspondence address (or Change of Correspondence Address form PTO/SB/122) attached.
- "Fee Address" indication (or "Fee Address" Indication form PTO/SB/47; Rev 03-02 or more recent) attached. Use of a Customer Number is required.

2. For printing on the patent front page, list

- (1) the names of up to 3 registered patent attorneys or agents OR, alternatively,
- (2) the name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.

1. Finnegan, Henderson,
2. Farabow, Garrett
3. & Dunner, LLP

3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document has been filed for recordation as set forth in 37 CFR 3.11. Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE: SpringWorks, LLC (B) RESIDENCE: (CITY and STATE OR COUNTRY) Minnetonka, Minnesota

Please check the appropriate assignee category or categories (will not be printed on the patent): Individual Corporation or other private group entity Government

4a. The following fee(s) are submitted:

- Issue Fee
- Publication Fee (No small entity discount permitted)
- Advance Order - # of Copies _____

4b. Payment of Fee(s): (Please first reapply any previously paid issue fee shown above)

- A check is enclosed.
- Payment by credit card. Form PTO-2038 is attached.
- The Director is hereby authorized to charge the required fee(s), any deficiency, and credit any overpayment, to Deposit Account Number 06-0916 (enclose an extra copy of this form).

5. Change in Entity Status (from status indicated above)

- a. Applicant claims SMALL ENTITY status. See 37 CFR 1.27.
- b. Applicant is no longer claiming SMALL ENTITY status. See 37 CFR 1.27(g)(2).

NOTE: The Issue Fee and Publication Fee (if required) will not be accepted from anyone other than the applicant; a registered attorney or agent; or the assignee or other party in interest as shown by the records of the United States Patent and Trademark Office.

Authorized Signature Gary J. Edwards
 Typed or printed name Gary J. Edwards

Date March 20, 2008
 Registration No. 41,008

This collection of information is required by 37 CFR 1.311. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, Virginia 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, Virginia 22313-1450.

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IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>			<i>Application Number</i>	10/954,182
			<i>Filing Date</i>	October 1, 2004
			<i>First Named Inventor</i>	Zhang et al.
			<i>Art Unit</i>	2882
			<i>Examiner Name</i>	Not Yet Assigned
			<i>Attorney Docket Number</i>	09140-0016-01000
Sheet	8	of	9	

NON PATENT LITERATURE DOCUMENTS			
Examiner Initials	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
		VON ROTTKAY et al. "Influence of stoichiometry on electrochromic cerium-titanium oxide compounds," Lawrence Berkeley National Laboratory, UC Berkeley, CA, (date unknown).	
		WESTLINDER et al. "Simulation and Dielectric Characterization of Reactive dc Magnetron Cosputtered (Ta ₂ O ₅) _{1-x} (TiO ₂) _x Thin Films," <i>J. Vac. Sci. Technol. B</i> , Vol 20, No. 3, pp. 855-861. (May/June 2002).	
		WILKES, Kenneth T. "Gas Permeation Through Vacuum Barrier Films and its Effect on VIP Thermal Performance," Vacuum Insulation Panel Symp., Baltimore, Maryland (May 3 1999)	
		YANAGAWA, H. et al., "Index-and-Dimensional Taper and Its Application to Photonic Devices," <i>Journal of Lightwave Technology</i> , 10(5):587-591 (1992).	
		Office Action issued on September 27, 2004 in U.S. Serial No. 10/291,179 (Attorney Docket No. 09140-0001-00).	
		Office Action issued on November 28, 2001 in U.S. Patent No. 6,506,289 (Attorney Docket No. 09140-0002-01). <i>Demaray et al. 1/2003 ✓</i>	
		Response to Office Action filed on February 20, 2002 in U.S. Patent No. 6,506,289 (Attorney Docket No. 09140-0002-01). <i>1/2003 ✓</i>	
		Office Action issued on April 17, 2002 in U.S. Patent No. 6,506,289 (Attorney Docket No. 09140-0002-01). <i>1/2003 ✓</i>	
		Response to Office Action filed on July 17, 2002 in U.S. Patent No. 6,506,289 (Attorney Docket No. 09140-0002-01). <i>1/03 ✓</i>	
		Quayle Action issued on November 10, 2003 in U.S. Patent No. 6,827,826 (Attorney Docket No. 09140-0002-02). <i>12/04 ✓</i>	
		Office Action issued on May 2, 2002 in U.S. Patent No. 6,533,907 (Attorney Docket No. 09140-0004-00). <i>3/03 ✓</i>	
		Response to Office Action filed on September 3, 2002 in U.S. Patent No. 6,533,907 (Attorney Docket No. 09140-0004-00). <i>3/03 ✓</i>	
		Office Action issued on February 12, 2004 in U.S. Serial No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
		Response to Office Action filed on August 10, 2004 in U.S. Serial No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
		Office Action issued on September 10, 2004 in U.S. Serial No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
		Office Action issued on May 14, 2003 in U.S. Serial No. 10/101,492 (Attorney Docket No. 09140-0015-00).	
		Response to Office Action filed on August 14, 2003 in U.S. Serial No. 10/101,492 (Attorney Docket No. 09140-0015-00).	
		Office Action issued on September 3, 2003 in U.S. Serial No. 10/101,492 (Attorney Docket No. 09140-0015-00).	
		Response to Office Action filed on March 3, 2004 in U.S. Serial No. 10/101,492 (Attorney Docket No. 09140-0015-00).	

28
 11/17/05

Examiner Signature	/Michelle Estrada/	Date Considered	03/04/2008
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /ME/

IDS Form PTO/SB/08: Substitute for form 1449A/PTO				Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>				<i>Application Number</i>	10/854,182
				<i>Filing Date</i>	October 1, 2004
				<i>First Named Inventor</i>	ZHANG, Hongmei
				<i>Art Unit</i>	2823
				<i>Examiner Name</i>	ESTRADA, Michelle
Sheet	3	of	3	<i>Attorney Docket Number</i>	9140.0018-01

NON PATENT LITERATURE DOCUMENTS		
		Response to Final Office Action filed April 14, 2006, in U.S. Appl. No. 10/291,179 (Atty. Docket No. 9140.0001-00).
		Office Action mailed April 27, 2006, in U.S. Appl. No. 10/291,179 (Atty. Docket No. 9140.0001-00).
		Response to Office Action filed July 27, 2006, in U.S. Appl. No. 10/291,179 (Atty. Docket No. 9140.0001-00).
		Notice of Allowance mailed August 6, 2002, for US Patent No. 6,506,289 (Atty. Docket No. 09140.0002-01). <i>Demovsky et al. 11/2003</i>
		Response to Office Action filed February 28, 2006 in U.S. Application No. 09/903,081 (Atty. Docket No. 09140-0014-00).
		Final Office Action mailed May 8, 2006 in U.S. Application No. 09/903,081 (Atty. Docket No. 09140-0014-00).
		Final Office Action mailed June 9, 2006 in U.S. Appl. No. 11/100,856 (Atty. Docket No. 09140.0015-01).
		Office Action mailed March 22, 2006, in U.S. Appl. No. 10/101,863 (Atty. Docket No. 09140.0016-00).
		Response to Office Action filed June 12, 2006, in U.S. Appl. No. 10/101,863 (Atty. Docket No. 09140.0016-00).
		Response to Office Action filed on May 15, 2006, in U.S. Application No. 10/101,341 (Atty. Docket No. 09140-0017-00).
		Office Action issued on August 2, 2006, in U.S. Application No. 10/101,341 (Atty. Docket No. 09140-0017-00).
		Office Action issued on March 23, 2006, in U.S. Application No. 10/650,461 (Atty. Docket No. 09140-0025-00).
		Response to Office Action filed on July 24, 2006, in U.S. Application No. 10/650,461 (Atty. Docket No. 09140-0025-00).
		Response to Office Action filed March 2, 2006 in U.S. Application No. 10/789,953 (Atty. Docket No. 09140.0030-00).
		Final Office Action issued on May 19, 2006 in U.S. Application No. 10/789,953 (Atty. Docket No. 09140.0030-00).
		Office Action from Singapore Patent Office in Appl. No. 200505388-9, dated March 20, 2006 (Atty. Docket No. 9140.0030-00256).
		Office Action mailed April 19, 2006 in U.S. Application No. 10/851,542 (Atty. Docket No. 09140.0033-00).
		Response to Office Action filed July 26, 2006 in U.S. Application No. 10/851,542 (Atty. Docket No. 09140.0033-00).
		Specification as filed September 2, 2005, for U.S. Appl. No. 11/218,652 (Atty. Docket No. 09140.0052-00).

<i>Michelle Estrada</i>	<i>Michelle Estrada</i>	Date Considered	10/30/06
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

**Express Mail Label No.
EV 901562545 US**

IDS Form PTO/SB/08: Substitute for form 1449A/PTO			Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>			Application Number	10/954,182
			Filing Date	October 1, 2004
			First Named Inventor	ZHANG, Hongmei
			Art Unit	2823
			Examiner Name	ESTRADA, Michelle
			Attorney Docket Number	9140.0016-01
Sheet	2	of	3	

NON PATENT LITERATURE DOCUMENTS			
		No. 10,291,179 (Attorney Docket No. 9140.0001-00).	
ME		Office Action dated December 1, 2006, in U.S. Application No. 10,291,179 (Attorney Docket No. 9140.0001-00).	
		Notice of Allowance mailed March 25, 2004 for US Patent No. 6,827,826 (Atty. Docket No. 09140.0002-02). <i>Demaray et al. 12/2004</i>	<i>28</i>
		Notice of Allowance issued on October 8, 2002, in U.S. Patent No. 6,533,907 (Atty. Docket No. 09140-0004-00). <i>Demaray et al. 3/2003</i>	<i>4117108</i>
		Amendment dated October 19, 2006, in U.S. Application No. 09/903,081 (Atty. Docket No. 09140.0014-00).	
		Notice of Allowance issued on October 21, 2004, in U.S. Application No. 10/101,492 (Atty. Docket No. 09140-0015-00).	
		Response to Office Action filed September 11, 2006 in U.S. Application No. 11/100,856 (Atty. Docket No. 09140.0015-01.)	
		Office Action mailed December 1, 2006, in U.S. Application No. 11/100,856 (Attorney Docket No. 09140.0015-01).	<i>L</i>
		Office Action mailed September 6, 2006, in U.S. Appl. No. 10/101,863 (Atty. Docket No. 09140.0016-00).	
		Final Office Action mailed October 19, 2006, in U.S. Application No. 10/650,461 (Attorney Docket No. 09140.0025-00).	
		Voluntary Amendment filed July 26, 2006 in TW Appl. No. 92123625 (Atty. Docket No. 09140.0025-00270).	
		Response to Final Office Action filed August 3, 2006, in U.S. Application No. 10/789,953 (Atty. Docket No. 09140.0030-00).	
		Notice of Allowance mailed October 23, 2006, in U.S. Application No. 10/789,953 (Atty. Docket No. 09140.0030-00).	
		Office Action dated October 12, 2006, for U.S. Application No. 11/228,805 (Attorney Docket No. 09140.0030-01000).	
		Office Action dated September 22, 2006 from Korean Patent Office in Appl. No. 10-2005-7016055 (Atty. Docket No. 09140.0030-00202)	
	↓	Response to Office Action mailed November 8, 2006, to the Korean Patent Office in Application No. 10-2005-7016055 (Attorney Docket No. 09140.0030-00202).	
ME		Response to Office Action from Singapore Patent Office in Appl. No. 200505388-9, dated August 11, 2006 (Atty. Docket No. 9140.0030-00256).	

Examiner Signature	/Michelle Estrada/	Date Considered	03/02/2007
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EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

EXPRESS MAIL LABEL NO. EV 746096154 US

U.S. Patent Application No. 10/954,182

Attorney Docket No. 09140-0016-01

Customer No. 22,852

Attorney Docket Number	U.S. Patent/ Serial No.	U.S./PCT Publication No.	Title	Examiner
09140-0033-00	10/851,542	<i>Demaray et al.</i> 12/04 US 2004/0259305 A1	Energy Conversion and Storage Devices by Physical Vapor Deposition of Titanium and Titanium Oxides and Sub-Oxides	Not Yet Assigned
09140-0034-00	10/850,968	<i>Demaray et al.</i> 1/2005 US 2005/0000794 A1	Transparent Conductive Oxides from a Metallic Target	Not Yet Assigned

28
4117108

Copies of U.S. Patents and U.S. Patent Publications are not provided. Copies of foreign patent documents and non-patent literature documents are included herewith.

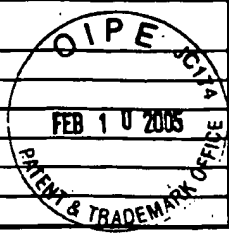
Applicants submit copies of Office Actions issued by the U.S. Patent and Trademark Office in the above-listed applications and Applicants' responses to these office actions. Applicants also submit International Search Reports and Written Opinions issued in the Patent Cooperation Treaty applications corresponding to the U.S. Patent Applications listed above.

Applicants respectfully request that the Examiner consider the listed documents and indicate that they were considered by making appropriate notations on the attached form.

This submission does not represent that a search has been made or that no better art exists and does not constitute an admission that each or all of the listed documents are material or constitute "prior art." If the Examiner applies any of the documents as prior art against any claim in the application and Applicants determine that the cited documents do not constitute "prior art" under United States law, Applicants reserve the right to present to the Office the relevant facts and law regarding the appropriate status of such documents.

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /ME/

IDS Form PTO/SB/08: Substitute for form 1449A/PTO		Complete if Known	
INFORMATION DISCLOSURE STATEMENT BY APPLICANT <i>(Use as many sheets as necessary)</i>		Application Number	10/954,182
		Filing Date	October 1, 2004
		First Named Inventor	Zhang et al.
		Art Unit	2882
		Examiner Name	Not Yet Assigned
		Attorney Docket Number	09140-0016-01000
Sheet	1	of	9



U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner Initials	Cite No.	Document Number	Issue or Publication Date	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		Number-Kind Code ² (if known)	MM-DD-YYYY		
		US 2001/0027159 A1	Oct. 4, 2001	Kaneyoshi	
		US 2002/0033330 A1	Mar. 21, 2002	Demaray et al.	
		US 2002/0134671 A1	Jul. 17, 2003	Demaray et al. 9/02	
		US 2002/0170821 A1	Nov. 21, 2002	Sandlin et al.	
		US 2003/0097858 A1	May 29, 2003	Strohhofer et al.	
		US 2003/0127319 A1	Jul. 10, 2003	Demaray et al.	
		US 2003/0134054 A1	Jul. 17, 2003	Demaray et al.	
		US 2003/0173207 A1	Sep. 18, 2003	Zhang et al.	
		US 2003/0173208 A1	Sep. 18, 2003	Pan et al.	
		US 2003/0174391 A1	Sep. 18, 2003	Pan et al.	
		US 2004/0105644 A1	Jun. 3, 2004	Dawes	
		US 2004/0259305 A1	Dec. 23, 2004	Demaray et al.	
		US 2005/0000794 A1	Jan. 6, 2005	Demaray et al.	
		US 2005/0006768 A1	Jan. 13, 2005	Narasimhan et al.	
		US 5,085,904	Feb. 4, 1992	Deak et al.	
		US 5,107,538	Apr. 21, 1992	Benton et al.	
		US 5,119,460	Jun. 2, 1992	Bruce et al.	
		US 5,306,569	Apr. 26, 1994	Hiraki	
		US 5,355,089	Oct. 11, 1994	Treger	
		US 5,457,569	Oct. 10, 1995	Liou et al.	
		US 5,499,207	Mar. 12, 1996	Miki et al.	
		US 5,563,979	Oct. 8, 1996	Bruce et al.	
		US 5,591,520	Jan. 7, 1997	Migliorini et al.	
		US 5,607,789	Mar. 4, 1997	Treger et al.	
		US 5,654,984	Aug. 5, 1997	Hershberger et al.	
		US 5,686,360	Nov. 11, 1997	Harvey, III et al.	
		US 5,689,522	Nov. 18, 1997	Beach	
		US 5,731,661	Mar. 24, 1998	So et al.	
		US 5,757,126	May 26, 1998	Harvey, III et al.	
		US 5,762,768	Jun. 9, 1998	Goy et al.	
		US 5,771,562	Jun. 30, 1998	Harvey, III et al.	
Examiner Signature	/Michelle Estrada/			Date Considered	03/04/2008

JP
4/18/08

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ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /ME/



APPLICATION NO.	ISSUE DATE	PATENT NO.	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/954,182	06/03/2008	7381657	10655.0016-01	9873

22852 7590 05/14/2008
FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER
LLP
901 NEW YORK AVENUE, NW
WASHINGTON, DC 20001-4413

ISSUE NOTIFICATION

The projected patent number and issue date are specified above.

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b) (application filed on or after May 29, 2000)

The Patent Term Adjustment is 0 day(s). Any patent to issue from the above-identified application will include an indication of the adjustment on the front page.

If a Continued Prosecution Application (CPA) was filed in the above-identified application, the filing date that determines Patent Term Adjustment is the filing date of the most recent CPA.

Applicant will be able to obtain more detailed information by accessing the Patent Application Information Retrieval (PAIR) WEB site (<http://pair.uspto.gov>).

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Customer Service Center of the Office of Patent Publication at (571)-272-4200.

APPLICANT(s) (Please see PAIR WEB site <http://pair.uspto.gov> for additional applicants):

Hongmei Zhang, San Jose, CA;
Mukundan Narasimhan, San Jose, CA;
Ravi Mullapudi, San Jose, CA;
Richard E. Demaray, Portola Valley, CA;

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re U.S. Patent No.: 7,381,657)
Inventors: Hongmei ZHANG et al.)
Issue Date.: June 3, 2008)
For: BIASED PULSE DC REACTIVE)
SPUTTERING OF OXIDE FILMS)

COMMISSIONER FOR PATENTS
OFFICE OF PATENT PUBLICATION
ATTN: Certificate of Correction Branch
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

REQUEST FOR CERTIFICATE OF CORRECTION

Pursuant to 35 U.S.C. § 254, and 37 C.F.R. §§ 1.322 and 1.323, this is a request for a Certificate of Correction in the above-identified patent. The mistake identified by a (*) below is of a clerical or typographical nature, and resulted from an error made in good faith by the patentee. The mistakes identified by a (**) below occurred through the fault of the U.S. Patent and Trademark Office, as clearly disclosed by the records of the application which matured into this patent. The Commissioner is hereby authorized to charge the fee of \$100.00 to Deposit Account No. 06-0916 as set forth in 37 C.F.R. § 1.20(a).

(*) On the title page, item (57), line 10, "inention." should read -- invention. --.

(**) In claim 17, col. 24, line 26, "P. As," should read -- P, As, --.

(**) In claim 17, col. 24, line 27, "Th," should read -- Tb, --.

The complete Certificate of Correction involves one (1) page. Issuance of a Certificate of Correction containing the correction is earnestly requested.

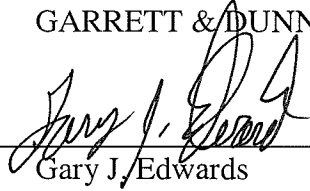
Please charge any required fees not included herewith to Deposit Account 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Dated: June 17, 2008

By: _____



Gary J. Edwards
Reg. No. 41,008
(650) 849-6622

UNITED STATES PATENT AND TRADEMARK OFFICE
CERTIFICATE OF CORRECTION

PATENT NO. 7,381,657

Page 1 of 1

APPLICATION NO.: 10/954,182

ISSUE DATE: June 3, 2008

INVENTOR(S): Hongmei ZHANG et al.

It is hereby certified that an error or errors appear in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

On the title page, item (57), line 10, "inention." should read -- invention. --.

In claim 17, col. 24, line 26, "P. As," should read -- P, As, --.

In claim 17, col. 24, line 27, "Th," should read -- Tb, --.

MAILING ADDRESS OF SENDER

Finnegan, Henderson, Farabow,
Garrett & Dunner, L.L.P.
901 New York Avenue, N.W.
Washington, D.C. 20001-4413

Electronic Patent Application Fee Transmittal

Application Number:	10954182
Filing Date:	01-Oct-2004
Title of Invention:	BIASED PULSE DC REACTIVE SPUTTERING OF OXIDE FILMS
First Named Inventor/Applicant Name:	Hongmei Zhang
Filer:	Gary James Edwards/Annie Wong
Attorney Docket Number:	10655.0016-01

Filed as Large Entity

Utility Filing Fees

Description	Fee Code	Quantity	Amount	Sub-Total in USD(\$)
Basic Filing:				
Pages:				
Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
Certificate of correction	1811	1	100	100

Extension-of-Time:

Description	Fee Code	Quantity	Amount	Sub-Total in USD(\$)
Miscellaneous:				
Total in USD (\$)				100

Electronic Acknowledgement Receipt

EFS ID:	3472815
Application Number:	10954182
International Application Number:	
Confirmation Number:	9873
Title of Invention:	BIASED PULSE DC REACTIVE SPUTTERING OF OXIDE FILMS
First Named Inventor/Applicant Name:	Hongmei Zhang
Customer Number:	22852
Filer:	Gary James Edwards/Annie Wong
Filer Authorized By:	Gary James Edwards
Attorney Docket Number:	10655.0016-01
Receipt Date:	17-JUN-2008
Filing Date:	01-OCT-2004
Time Stamp:	18:20:35
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	Deposit Account
Payment was successfully received in RAM	\$100
RAM confirmation Number	3302
Deposit Account	060916
Authorized User	

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

Charge any Additional Fees required under 37 C.F.R. Section 1.21 (Miscellaneous fees and charges)

File Listing:

Document Number	Document Description	File Name	File Size(Bytes) /Message Digest	Multi Part /.zip	Pages (if appl.)
1	Request for Certificate of Correction	Request_Certificate_of_Correction_17Jun2008_7381657.pdf	35334 e06596b4bd76d83ab6ce8502605b8ee56b68d1fe	no	3

Warnings:**Information:**

2	Fee Worksheet (PTO-06)	fee-info.pdf	8157 ceacta293a4c4e02a9650ef9c580ba9cf754cb32	no	2
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Warnings:**Information:**

Total Files Size (in bytes):	43491
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This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

UNITED STATES PATENT AND TRADEMARK OFFICE
CERTIFICATE OF CORRECTION

PATENT NO. : 7,381,657 B2
APPLICATION NO. : 10/954182
DATED : June 3, 2008
INVENTOR(S) : Hongmei Zhang et al.

Page 1 of 1

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

On the title page, item (57), Abstract line 10, "inention." should read -- invention. --.

In claim 17, col. 24, line 26, "P. As," should read -- P, As, --.

In claim 17, col. 24, line 27, "Th," should read -- Tb, --.

Signed and Sealed this

Twelfth Day of August, 2008

A handwritten signature in black ink that reads "Jon W. Dudas". The signature is stylized, with the first name "Jon" written in a cursive-like font, and "W. Dudas" following in a similar style.

JON W. DUDAS
Director of the United States Patent and Trademark Office

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)
)
Hongmei ZHANG et al.) Group Art Unit: 2823
)
Application No.: 10/954,182) Examiner: Michelle ESTRADA
)
Filed: October 1, 2004)
)
For: BIASED PULSE DC REACTIVE) Confirmation No.: 9873
SPUTTERING OF OXIDE FILMS)
)

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

**REQUEST FOR WITHDRAWAL AS ATTORNEY OR AGENT AND
CHANGE OF CORRESPONDENCE ADDRESS**

I hereby apply to withdraw myself and the practitioners associated with Customer Number 22,852 as attorney or agent for the above-identified patent application.

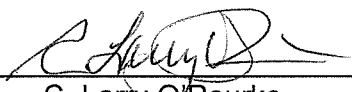
The reasons for this request are: The Assignee of Record has requested that this application be transferred to another law firm for further prosecution, therefore this request is made under the provision of 37 CFR 10.40(b)(4).

Please change the correspondence address and direct all future correspondence to:
Haynes & Boone, LLP, **USPTO Customer Number 27,683.**

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Dated: July 28, 2009

By: 

C. Larry O'Rourke
Reg. No. 26,014
(650) 849-6600

Electronic Acknowledgement Receipt

EFS ID:	5782174
Application Number:	10954182
International Application Number:	
Confirmation Number:	9873
Title of Invention:	BIASED PULSE DC REACTIVE SPUTTERING OF OXIDE FILMS
First Named Inventor/Applicant Name:	Hongmei Zhang
Customer Number:	22852
Filer:	Aaron James Capron/Drew Herndon
Filer Authorized By:	Aaron James Capron
Attorney Docket Number:	10655.0016-01
Receipt Date:	28-JUL-2009
Filing Date:	01-OCT-2004
Time Stamp:	13:46:51
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	no
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File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Petition to withdraw attorney or agent (SB83)	Request_for_Withdrawal_10655-0016-01.PDF	11787 <small>c431d494a4cc3cab23a44aa0e61de91d7665d00f</small>	no	1

Warnings:

Information:

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Via EFS-Web
COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, VA 22313-1450

POWER OF ATTORNEY TO PROSECUTE APPLIATIONS BEFORE THE USPTO

Dear Sir:

I hereby appoint practitioners associated with Haynes and Boone Customer Number **27683**, as my/our attorney(s) or agent(s) to prosecute the applications identified in Attachment A, and to transact all business in the United States Patent and Trademark Office connected therewith.

Please recognize or change the correspondence address for the applications listed in Attachment A to Customer Number **27683**.

SpringWorks, LLC, is the Assignee of record of the entire interest in the applications identified in Attachment A by virtue of the assignment recorded in the Reel and Frame numbers listed, or for which a copy therefore is attached, in Attachment A.

SPRINGWORKS, LLC

By: _____
Name: Douglas A. Kelley
Title: Receiver

Attachment A Power of Attorney to Prosecute Applications Before the USPTO

Applicant/Patent Owner: **SpringWorks LLC** hereby states that they are the assignee of the entire right, title and interest as listed below by virtue of an assignment from the inventor(s) of the patent application/patent. The assignment was recorded in the United States Patent and Trademark Office at the Reel and Frame number listed below or for which a copy therefore is attached.

Attorney Docket No.	Application Number / Patent Number	Appl. Date/ Issued Date	Inventors	Title	Current Owner	Chain of Title	Assignment Reel/Frame	Recorded Date
43668.3	11/191,643	7/27/2005	H. Zhang M. Narasimhan R. Mullapudi R. Demaray	Biased Pulse DC Reactive Sputtering Of Oxide Films	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	014766 / 0601 20134 / 0102	12/2/2003 11/19/2007
43668.4	11/100,856	4/6/2005	T. Pan R. Demaray Y. Chen Y. Xie R. Peithe	Mode Size Converter For A Planar Waveguide	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	020035 / 0110 20134 / 0102	10/30/2007 11/19/2007
43668.5	10/850,968	5/20/2004	R. Demaray M. Narasimhan	Transparent Conductive Oxides	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	014945 / 0661 20134 / 0102	8/4/2004 11/19/2007
43668.22	10/650,461	8/27/2003	D. Dawes	Optically Coupling Into Highly Uniform Waveguides	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	014897 / 0768 20134 / 0102	1/16/2007 11/19/2007
43668.39	11/726,972	3/22/2007	R. Demaray H. Zhang M. Narasimhan V. Milonopoulou	Energy Conversion And Storage Devices By Physical Vapor Deposition Of Titanium And Titanium Oxides And Sub-Oxides	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	014948 / 0097 20134 / 0102	8/5/2004 11/19/2007
43668.44	11/297,057	12/7/2005	H. Zhang R. Demaray M. Shao	Deposition Of LiCoO2	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	017196 / 0699 20134 / 0102	12/21/2006 11/19/2007
43668.53	11/218,652	9/2/2005	H. Zhang R. Demaray	Deposition Of Perovskite And Other Compound Ceramic Films From Dielectric Applications	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	016821 / 0220 20134 / 0102	11/28/2005 11/19/2007
43668.6	10/291,179 US Pat No. 7,404,877	ISSUED - 7/29/08	R. Demaray, V. Milonopoulou	Low temperature zirconia based thermal barrier layer by PVD	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	014756 / 0416 20134 / 0102	12/2/2003 11/19/2007

Attachment A Power of Attorney to Prosecute Applications Before the USPTO

Applicant/Patent Owner: **SpringWorks LLC** hereby states that they are the assignee of the entire right, title and interest as listed below by virtue of an assignment from the inventor(s) of the patent application/patent. The assignment was recorded in the United States Patent and Trademark Office at the Reel and Frame number listed below or for which a copy therefore is attached.

Attorney Docket No.	Application Number / Patent Number	Appl. Date/ Issued Date	Inventors	Title	Current Owner	Chain of Title	Assignment Reel/Frame	Recorded Date
43668.8	09/903,050 US Pat No. 6,506,289	ISSUED - 1/14/03	R. Demaray, K. Wang, R. Mullapudi, D. Stadtler, H Zhang, R. Peth	Planar optical devices and methods for their manufacture	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	012010 / 0318 20134 / 0102	07/10/2001 11/19/2007
43668.9	10/288,278 US Pat No. 6,827,826	ISSUED - 12/7/04	R. Demaray, K. Wang, R. Mullapudi, D. Stadtler, H Zhang, R. Peth	Planar optical devices and methods for their manufacture	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	012010 / 0318 20134 / 0102	07/10/2001 11/19/2007
43668.13	09/766,463 U.S. Pat No. 6,533,907	ISSUED - 3/18/03	R. Demaray, J. Shan, K. Wang, R. Mullapudi	Method of Producing amorphous silicon for hard mask and waveguide applications	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	011504 / 0738 20134 / 0102	01/19/2001 11/19/2007
43668.14	09/903,081 US Pat No. 7,469,558	ISSUED - 12/30/08	R. Demaray, K. Wang, R. Mullapudi, Q. Zhu, H. Zhang, H. Ackler, J. Egermeier, R. Peth	As-deposited planar optical waveguides with low scattering loss and methods for their manufacture	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	012010 / 0752 20134 / 0102	07/10/2001 11/19/2007
43668.15	10/101,492 US Pat No. 6,884,327	ISSUED - 4/26/05	T. Pan, R. Demaray, Y. Chen, Y. Xie, R. Peth	Mode size converter for a planar waveguide	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	020035 / 0110 20134 / 0102	10/30/2007 11/19/2007
43668.17	10/101,863 US Pat No. 7,378,356	ISSUED - 5/27/08	H. Zhang, M. Narasimhan, R. Mullapudi, R. Demaray	Biased pulse DC reactive sputtering of oxide films	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	014766 / 0601 20134 / 0102	12/2/2003 11/19/2007
43668.21	10/954,182 US Pat No. 7,381,657	ISSUED - 6/30/08	H. Zhang, M. Narasimhan, R. Mullapudi, R. Demaray	Biased pulse DC reactive sputtering of oxide films	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	014766 / 0601 20134 / 0102	12/2/2003 11/19/2007
43668.19	11/228,834 US Pat No. 7,544,276	ISSUED - 6/9/09	H. Zhang, M. Narasimhan, R. Mullapudi, R. Demaray	Biased pulse DC reactive sputtering of oxide films	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	014766 / 0601 20134 / 0102	12/2/2003 11/19/2007

Initial: 

**Attachment A
Power of Attorney to Prosecute Applications Before the USPTO**

Applicant/Patent Owner: **SpringWorks LLC** hereby states that they are the assignee of the entire right, title and interest as listed below by virtue of an assignment from the inventor(s) of the patent application/patent. The assignment was recorded in the United States Patent and Trademark Office at the Reel and Frame number listed below or for which a copy therefore is attached.

Attorney Docket No.	Application Number / Patent Number	Appl. Date/ Issued Date	Inventors	Title	Current Owner	Chain of Title	Assignment Reel/Frame	Recorded Date
43668.20	11/228,717 U.S. Pat No. 7,413,998	ISSUED - 8/19/08	H. Zhang, M. Narasimhan, R. Mullapudi, R. Demaray	Biased pulse DC reactive sputtering of oxide films	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	014766 / 0601 20134 / 0102	12/2/2003 11/19/2007
43668.25	10/789,953 US Pat No. 7,205,662	ISSUED - 4/17/07	M. Narasimhan, P. Brooks, R. Demaray	Dielectric Barrier Layer Films	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	014948 / 0111 20134 / 0102	08/05/2004 11/19/2007
43668.32	11/228,805 US Pat No. 7,262,131	ISSUED - 8/28/07	M. Narasimhan, P. Brooks, R. Demaray	Dielectric Barrier Layer Films	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	014948 / 0111 20134 / 0102	08/05/2004 11/19/2007
43668.34	10/851,542 US Pat No. 7,238,628	ISSUED - 7/3/07	R. Demaray, H. Zhang, M. Narasimhan, V. Milonopoulou	Energy Conversion and Storage Devices by Physical Vapor Deposition of Titanium and Titanium Oxides and Sub-Oxides	SpringWorks LLC	From Inventors to Symmorphix From Symmorphix to SpringWorks LLC	014948 / 0097 20134 / 0102	8/5/2004 11/19/2007

SpringWorks LLC

By: Douglas A. Kelley
Name: Douglas A. Kelley
Title: Receiver

Initial: DAK

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

CHANGE OF CORRESPONDENCE ADDRESS *Application*

Address to:
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Application Number	10/954,182
Filing Date	10-1-2004
First Named Inventor	H. Zhang
Art Unit	2823
Examiner Name	Michelle Estrada
Attorney Docket Number	43668.21

Please change the Correspondence Address for the above-identified patent application to:

 The address associated with
Customer Number:

27683

OR
 Firm or
Individual Name

Address

City

State

Zip

Country

Telephone

Email

This form cannot be used to change the data associated with a Customer Number. To change the data associated with an existing Customer Number use "Request for Customer Number Data Change" (PTO/SB/124).

I am the:

- Applicant/Inventor
- Assignee of record of the entire interest.
Statement under 37 CFR 3.73(b) is enclosed. (Form PTO/SB/96).
- Attorney or agent of record. Registration Number 41008.
- Registered practitioner named in the application transmittal letter in an application without an executed oath or declaration. See 37 CFR 1.33(a)(1). Registration Number _____.

Signature /gary j. edwards/

Typed or Printed
Name Gary J. Edwards

Date December 2, 2009

Telephone 408-660-4120

NOTE: Signatures of all the inventors or assignees of record of the entire interest or their representative(s) are required. Submit multiple forms if more than one signature is required, see below*.

 *Total of _____ forms are submitted.

This collection of information is required by 37 CFR 1.33. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 3 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: **Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

Privacy Act Statement

The **Privacy Act of 1974 (P.L. 93-579)** requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether disclosure of these records is required by the Freedom of Information Act.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (*i.e.*, GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspection or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

Electronic Acknowledgement Receipt

EFS ID:	6555066
Application Number:	10954182
International Application Number:	
Confirmation Number:	9873
Title of Invention:	BIASED PULSE DC REACTIVE SPUTTERING OF OXIDE FILMS
First Named Inventor/Applicant Name:	Hongmei Zhang
Customer Number:	22852
Filer:	Gary James Edwards/Sheila Badon
Filer Authorized By:	Gary James Edwards
Attorney Docket Number:	43668.21
Receipt Date:	02-DEC-2009
Filing Date:	01-OCT-2004
Time Stamp:	12:29:52
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	no
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File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1		43668_POA.pdf	482094 <small>50be7508dee6f2cb790d6ae139f6af2655b53fda</small>	yes	4

Multipart Description/PDF files in .zip description			
Document Description	Start	End	
Power of Attorney	1	1	
Assignee showing of ownership per 37 CFR 3.73(b).	2	4	

Warnings:

Information:

2	Change of Address	43668_21_COA.pdf	298817	no	2
			d99d50b20ef1f621e331386a1b218c7bf1aa07ba		

Warnings:

Information:

Total Files Size (in bytes):			780911		
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This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
10/954,182	10/01/2004	Hongmei Zhang	43668.21

CONFIRMATION NO. 9873

POA ACCEPTANCE LETTER



27683
HAYNES AND BOONE, LLP
IP Section
2323 Victory Avenue
Suite 700
Dallas, TX 75219

Date Mailed: 12/11/2009

NOTICE OF ACCEPTANCE OF POWER OF ATTORNEY

This is in response to the Power of Attorney filed 12/02/2009.

The Power of Attorney in this application is accepted. Correspondence in this application will be mailed to the above address as provided by 37 CFR 1.33.

/mnguyen/

Office of Data Management, Application Assistance Unit (571) 272-4000, or (571) 272-4200, or 1-888-786-0101



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
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P.O. Box 1450
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APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
10/954,182	10/01/2004	Hongmei Zhang	43668.21

CONFIRMATION NO. 9873

POWER OF ATTORNEY NOTICE



22852
FINNEGAN, HENDERSON, FARABOW, GARRETT & DUNNER
LLP
901 NEW YORK AVENUE, NW
WASHINGTON, DC 20001-4413

Date Mailed: 12/11/2009

NOTICE REGARDING CHANGE OF POWER OF ATTORNEY

This is in response to the Power of Attorney filed 12/02/2009.

- The Power of Attorney to you in this application has been revoked by the assignee who has intervened as provided by 37 CFR 3.71. Future correspondence will be mailed to the new address of record(37 CFR 1.33).

/mnguyen/

Office of Data Management, Application Assistance Unit (571) 272-4000, or (571) 272-4200, or 1-888-786-0101



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
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Alexandria, Virginia 22313-1450
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Table with 5 columns: APPLICATION NUMBER, FILING OR 371(C) DATE, FIRST NAMED APPLICANT, ATTY.DOCKET NO./TITLE, REQUEST ID. Values: 10/954,182, 10/01/2004, Hongmei Zhang, 48604.22, 93174

Acknowledgement of Change to Small Entity Status

The entity status change request below filed through Private PAIR on 08/06/2019 has been accepted.

CERTIFICATIONS:

Change of Entity Status:

X Applicant asserting small entity status. See 37 CFR 1.27.

NOTE: If the application was previously under micro entity status, checking this box will be taken to be a notification of loss of entitlement to micro entity status.

This portion must be completed by the signatory or signatories making the entity status change in accordance with 37 CFR 1.4(d)(4).

Table with 2 columns: Label (Signature, Name, Registration Number) and Value (/Gary Edwards/, Gary Edwards, 41008)

AO 120 (Rev. 08/10)

TO: Mail Stop 8 Director of the U.S. Patent and Trademark Office P.O. Box 1450 Alexandria, VA 22313-1450	REPORT ON THE FILING OR DETERMINATION OF AN ACTION REGARDING A PATENT OR TRADEMARK
--	---

In Compliance with 35 U.S.C. § 290 and/or 15 U.S.C. § 1116 you are hereby advised that a court action has been filed in the U.S. District Court _____ for the Western District of Texas _____ on the following

Trademarks or Patents. (the patent action involves 35 U.S.C. § 292.):

DOCKET NO. 6:20-cv-00634	DATE FILED 7/14/2020	U.S. DISTRICT COURT for the Western District of Texas
PLAINTIFF DEMARAY LLC		DEFENDANT INTEL CORPORATION
PATENT OR TRADEMARK NO.	DATE OF PATENT OR TRADEMARK	HOLDER OF PATENT OR TRADEMARK
1 7,544,276	6/9/2009	Demaray LLC
2 7,381,657	6/3/2008	Demaray LLC
3		
4		
5		

In the above—entitled case, the following patent(s)/ trademark(s) have been included:

DATE INCLUDED	INCLUDED BY <input type="checkbox"/> Amendment <input type="checkbox"/> Answer <input type="checkbox"/> Cross Bill <input type="checkbox"/> Other Pleading	
PATENT OR TRADEMARK NO.	DATE OF PATENT OR TRADEMARK	HOLDER OF PATENT OR TRADEMARK
1		
2		
3		
4		
5		

In the above—entitled case, the following decision has been rendered or judgement issued:

DECISION/JUDGEMENT

CLERK	(BY) DEPUTY CLERK	DATE
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Copy 1—Upon initiation of action, mail this copy to Director Copy 3—Upon termination of action, mail this copy to Director
 Copy 2—Upon filing document adding patent(s), mail this copy to Director Copy 4—Case file copy

AO 120 (Rev. 08/10)

TO: Mail Stop 8 Director of the U.S. Patent and Trademark Office P.O. Box 1450 Alexandria, VA 22313-1450	REPORT ON THE FILING OR DETERMINATION OF AN ACTION REGARDING A PATENT OR TRADEMARK
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In Compliance with 35 U.S.C. § 290 and/or 15 U.S.C. § 1116 you are hereby advised that a court action has been filed in the U.S. District Court _____ for the Western District of Texas _____ on the following

Trademarks or Patents. (the patent action involves 35 U.S.C. § 292.):

DOCKET NO. 6:20-cv-00636	DATE FILED	U.S. DISTRICT COURT for the Western District of Texas
PLAINTIFF Demaray LLC		DEFENDANT Samsung Electronics Co., LTD, Samsung Electronics America, Inc., Samsung Semiconductor, Inc., and Samsung Austin Semiconductor, LLC
PATENT OR TRADEMARK NO.	DATE OF PATENT OR TRADEMARK	HOLDER OF PATENT OR TRADEMARK
1 7,544,276	6/9/2009	Demaray LLC
2 7,381,657	6/3/2008	Demaray LLC
3		
4		
5		

In the above—entitled case, the following patent(s)/ trademark(s) have been included:

DATE INCLUDED	INCLUDED BY <input type="checkbox"/> Amendment <input type="checkbox"/> Answer <input type="checkbox"/> Cross Bill <input type="checkbox"/> Other Pleading	
PATENT OR TRADEMARK NO.	DATE OF PATENT OR TRADEMARK	HOLDER OF PATENT OR TRADEMARK
1		
2		
3		
4		
5		

In the above—entitled case, the following decision has been rendered or judgement issued:

DECISION/JUDGEMENT

CLERK	(BY) DEPUTY CLERK	DATE
-------	-------------------	------

Copy 1—Upon initiation of action, mail this copy to Director Copy 3—Upon termination of action, mail this copy to Director
 Copy 2—Upon filing document adding patent(s), mail this copy to Director Copy 4—Case file copy

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

APPLIED MATERIALS, INC.,
Petitioner,

v.

DEMARAY LLC,
Patent Owner.

IPR2021-00106
Patent 7,381,657 B2

Before CHRISTOPHER L. CRUMBLEY, KRISTINA M. KALAN, and
KIMBERLY McGRAW, *Administrative Patent Judges*.

Opinion for the Board filed by *Administrative Patent Judge* KALAN.

Opinion Concurring filed by *Administrative Patent Judge* CRUMBLEY.

McGRAW, *Administrative Patent Judge*.

DECISION
Denying Institution of *Inter Partes* Review
35 U.S.C. § 314

I. INTRODUCTION

Applied Materials, Inc. (“Petitioner”) filed a Petition (Paper 1, “Pet.”) requesting an *inter partes* review of claims 1–8 and 10–17 of U.S. Patent No. 7,381,657 B2 (Ex. 1001, “the ’657 patent”). Demaray LLC (“Patent Owner”) filed a Preliminary Response to the Petition (Paper 7, “Prelim. Resp.”). Pursuant to our authorization, Petitioner filed a Reply (Paper 10, “Pet. Reply”), and Patent Owner filed a Sur-reply (Paper 12, “Sur-reply”). Petitioner also filed a Petitioner’s Notice Regarding Multiple Petitions (Paper 2 (“Notice”)) to which Patent Owner filed a Response (Paper 8 (“Notice Response”)).

To institute an *inter partes* review, we must determine that the information presented in the Petition shows “there is a reasonable likelihood that the petitioner would prevail with respect to at least 1 of the claims challenged in the petition.” 35 U.S.C. § 314(a). The Board, however, has discretion to deny a petition even when a petitioner meets that threshold. *Id.*; *see, e.g., Cuozzo Speed Techs., LLC v. Lee*, 136 S. Ct. 2131, 2140 (2016) (“[T]he agency’s decision to deny a petition is a matter committed to the Patent Office’s discretion.”); *Gen. Plastic Indus. Co. v. Canon Kabushiki Kaisha*, IPR2016-01357, Paper 19 (PTAB Sept. 6, 2017) (precedential as to § II.B.4.i) (recognizing the same); *see also* Patent Trial and Appeal Board Consolidated Trial Practice Guide (Nov. 2019) (“Consolidated TPG”), 55–63, available at <https://www.uspto.gov/TrialPracticeGuideConsolidated>.

For the reasons discussed below, after considering the parties’ submissions and the evidence of record, we determine it is appropriate to exercise our discretion and decline to institute an *inter partes* review.

A. Related Proceedings

The parties identify IPR2021-00104 as another proceeding filed by Petitioner against the '657 patent. Pet. 1–2; Paper 5, 3. Patent Owner also identifies IPR2021-00103 and IPR2021-00105, which challenge U.S. Patent No. 7,544,276 B2, as related matters. Paper 5, 3. The parties also identify *Demaray LLC v. Samsung Electronics Co., Ltd.* et al., Case No. 6-20-cv-00636 (W.D. Tex.) (“Samsung Litigation”); *Demaray LLC v. Intel Corporation*, Case No. 6-20-cv-00634 (W.D. Tex.) (“Intel Litigation”) (collectively the “Texas Litigations”); and *Applied Materials, Inc. v. Demaray LLC*, Case No. 5-20-cv-05676 (N.D. Cal.) as related matters. Pet. 1; Paper 5, 2. Each of these proceedings involves the '657 patent. *Id.*

B. Real Parties-In-Interest

Petitioner identifies Intel Corporation; Samsung Electronics Co., Ltd.; Samsung Electronics America, Inc.; Samsung Semiconductor, Inc.; Samsung Austin Semiconductor, LLC, and itself as real parties-in-interest. Pet. 1. Patent Owner identifies itself as a real party-in-interest. Paper 5, 2.

C. The '657 Patent

The '657 patent, titled “Biased Pulse DC Reactive Sputtering of Oxide Films,” “relates to deposition of oxide and oxynitride films and, in particular, to deposition of oxide and oxynitride films by pulsed DC reactive sputtering.” Ex. 1001, code (54), 1:11–13. The '657 patent discloses that RF sputtering has been used for the deposition of oxide dielectric films, but arcing can occur between sputtering target tiles used to make such films, which causes contamination of the deposited films. *Id.* at 2:25–30.

The '657 patent further states that reactors for RF sputtering, particularly their power systems, are complicated. *Id.* at 2:30–38. The '657 patent discloses that reactive DC magnetron sputtering of nonconductive oxides “is done rarely” because insulating surfaces accumulate charge during deposition that results in arcing, which “can damage the power supply, produce particles, and degrade the properties of deposited oxide films.” *Id.* at 4:48–57.

Figure 1A of the '657 patent is reproduced below.

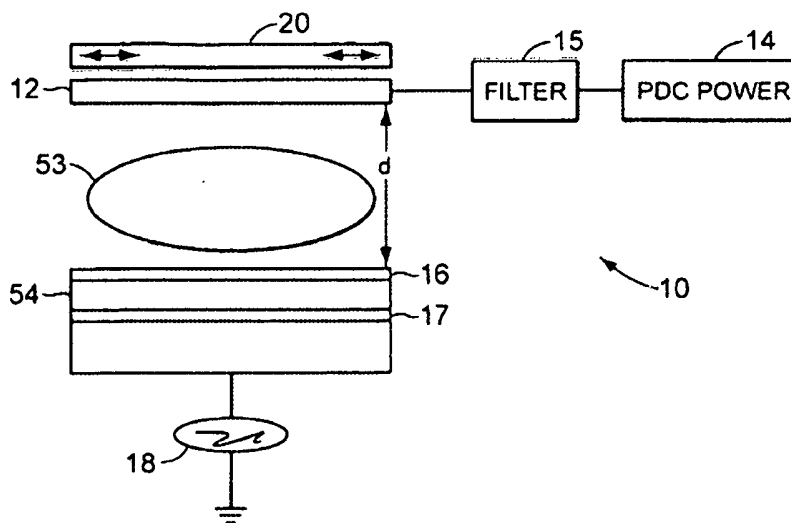


FIG. 1A

Figure 1A of the '657 patent, above, depicts a pulsed DC sputtering reactor. The '657 patent describes reactor apparatus 10 for sputtering material from target 12. *Id.* at 5:13–15. Magnet 20 is used to scan across the top of target 12, which reduces local erosion of target 12 during sputtering. *Id.* at 5:35, 8:57–66. Substrate 16 is opposite and parallel to target 12. *Id.* at 5:29–30. Substrate 16 is capacitively coupled to electrode 17 via insulator 54. *Id.* at 5:32–36. Electrode 17 can be coupled to RF power supply 18. *Id.* at 5:34–35. The '657 patent explains that RF power supply 18 is used to avoid columnar structures in a deposited film, which can be detrimental for optical

wave guide applications. *Id.* at 5:66–6:6. The '657 patent discloses that target 12 functions as a cathode when power is applied to target 12, which creates plasma 53. *Id.* at 5:30–32.

Target 12 is electrically coupled through filter 15 to pulsed DC power supply 14. *Id.* at 5:25–26. The '657 patent discloses that the polarity of the power supplied to target 12 by pulsed DC power supply 14 oscillates between negative and positive potentials. *Id.* at 5:36–39. According to the '657 patent, the insulating layer on the surface of target 12 discharges during the positive period, which prevents arcing. *Id.* at 5:39–41. The '657 patent discloses that the pulsing frequency must exceed a critical frequency, which depends on a target material, cathode current, and reverse time. *Id.* at 5:41–43.

Reactor apparatus 10 further includes filter 15, which prevents the RF power supply from coupling to into pulsed DC power supply 14. *Id.* at 5:56–57. According to the '657 patent, filter 15 can be a rejection filter, such as a 2 MHz band rejection filter when a 2 MHz power supply is used for RF power supply 18. *Id.* at 5:57–61. The '657 patent discloses that the band width of the filter 15 can be approximately 100 kHz. *Id.* at 5:61–63.

D. Illustrative Claim

Claims 1 and 2 of the '657 patent are independent. Claim 1 is representative and is reproduced below:

1. A method of depositing a film on an insulating substrate, comprising:
 - providing a process gas between a conductive target and the substrate;
 - providing pulsed DC power to the target through a narrow band rejection filter such that the target alternates between positive and negative voltages;

providing an RF bias at a frequency that corresponds to the narrow band rejection filter to the substrate;
providing a magnetic field to the target; and
reconditioning the target;
wherein reconditioning the target includes reactive sputtering in the metallic mode and then reactive sputtering in the poison mode.

Ex. 1001, 23:1–15.

E. The Asserted Grounds of Unpatentability

Petitioner contends claims 1–8 and 10–17 of the '657 patent are unpatentable on the following grounds. Pet. 7–8.

Reference(s)	Basis	Claim(s) Challenged
Licata, ¹ Kelly, ² Collins ³	§ 103	1–4, 6, 8, 10–12
Licata, Kelly, Collins, Aokura ⁴	§ 103	12, 13
Licata, Kelly, Collins, Dogheche ⁵	§ 103	5, 7
Licata, Kelly, Collins, Doessel ⁶	§ 103	14–17

In support of its unpatentability arguments, Petitioner relies on the declaration of Dr. Vivek Subramanian. Ex. 1002 (“Subramanian Declaration”). Patent Owner relies on the declaration of Dr. Alexander Glew. Ex. 2002 (“Glew Declaration”).

¹ US 6,132,564 B1, issued Oct. 17, 2000 (Ex. 1010).

² P. J. Kelly et al., *Reactive pulsed magnetron sputtering process for alumina films*, J. of Vacuum Sci. & Tech., A 18, 2890 (2000) (Ex. 1059).

³ US 6,077,384, issued June 20, 2000 (Ex. 1071).

⁴ JP H10102247 A, published Apr. 21, 1998 (Ex. 1068).

⁵ E. Dogheche, *Growth and optical characterization of aluminum nitride thin films deposited on silicon by radio-frequency sputtering*, Applied Physics Letters, 74, 1209 (1999) (Ex. 1029).

⁶ US 5,527,605, issued June 18, 1996 (Ex. 1070).

II. ANALYSIS

A. Discretion under 35 U.S.C. § 314(a) - Multiple Petitions

When evaluating whether to institute trial on more than one concurrently-filed petition addressing the same patent, we are guided by our Consolidated Trial Practice Guide, which states:

Based on the Board's prior experience, one petition should be sufficient to challenge the claims of a patent in most situations. Two or more petitions filed against the same patent at or about the same time . . . may place a substantial and unnecessary burden on the Board and the patent owner and could raise fairness, timing, and efficiency concerns. *See* U.S.C. § 316(b).

Nonetheless, the Board recognizes that there may be circumstances in which more than one petition may be necessary, including, for example, when the patent owner has asserted a large number of claims in litigation or when there is a dispute about priority date requiring arguments under multiple prior art references. In such cases two petitions by a petitioner may be needed, although this should be rare.

Consolidated TPG, 59.

Here, Petitioner filed two petitions on the same day, both challenging certain claims of the '657 patent. In the present petition, Petitioner challenges claims 1–8 and 10–17 on grounds based on Licata, Kelly, and Collins in combination with a number of other references. Notice, 1; Pet. 7–8. In IPR2021-00104, Petitioner challenges claims 1–21 of the '657 patent on grounds based on Barber and Hirose in combination with other references. IPR2021-00104, Paper 1, 3–5.

Petitioner argues that the Board should institute both petitions because Patent Owner has asserted claims of the '657 patent against real parties-in-interest Intel and Samsung in separate district court litigations. Notice 1–2. Petitioner asserts that by coordinating efforts with its real parties-in-interest,

including Intel and Samsung, and presenting only two petitions challenging the '657 patent, this “minimiz[es] issues arising from these multiple parties for the Board, despite the wealth of prior art against the '657 patent’s broad claims.” *Id.* at 2–3. Petitioner also argues that Petitioner also argues that there are material differences between the petitions that warrant institution of both petitions, and that the Board has routinely instituted two parallel petitions under similar circumstances. *Id.* at 3–5.

We are not persuaded that Petitioner’s arguments justify a second petition challenging the '657 patent.

First, we disagree with Petitioner’s argument that its coordinated efforts with Samsung and Intel to present two petitions against the '657 patent result in “minimizing issues from these multiple parties for the Board.” Notice 2–3. Significantly, neither Samsung nor Intel are petitioners in the present proceeding and neither Samsung nor Intel have made any representation that they will not file a petition challenging claims of the '657 patent. At most, Petitioner asserts that “as of their filing, [the two petitions filed by Petitioner] are the only challenges to the '657 patent before the Office.” *Id.* at 5. We decline to infer from Petitioner’s representations whether Samsung or Intel will file, or will not file, other petitions challenging the '657 patent. As such, Petitioner has not shown that its efforts minimize potential issues from multiple parties for the Board. We further note that if another party chooses to file a petition challenging claims of the '657 patent, we would determine at that time whether to institute any such petition. *See Valve Corp. v. Electronic Scripting Prods, Inc.*, IPR2019-00062, -00063, -00084, Paper 11, (PTAB Apr. 2, 2019), 2 (stating that “when different petitioners challenge

the same patent, [the Board] will consider any relationship between those petitioners when weighing the *General Plastic* factors”); *see also* Consolidated TPG, 23–26 (stating that the Board will consider the *General Plastic* factors when determining whether to institute a trial).

Second, we are not persuaded that material differences exist between the analysis presented in this Petition and the petition filed in IPR2021-00104. Petitioner argues that the IPR2021-00104 petition relies on references not of record during prosecution, whereas the present Petition relies on Kelly, which was of record during prosecution, although not applied by the Examiner. Notice 3. Petitioner does not sufficiently explain why Petitioner’s assertion of art (i.e., Kelly) that was before the Examiner in its second-ranked petition is material or justifies the institution of a second petition. *See* Consolidated TPG, 60 (stating Petitioner should provide “a succinct explanation of the differences between the petitions, why the issues addressed by the differences are material, and why the Board should exercise its discretion to institute additional petitions if it identifies one petition that satisfies petitioner’s burden under 35 U.S.C. § 314(a)”). For example, Petitioner does not explain why, if the Board institutes the first ranked petition that asserts art that was not before the Examiner, the Board should also institute a second petition that relies on art that was before the Examiner.

We also disagree with Petitioner’s contention that its two petitions present different approaches to the claim limitations. Notice 3–4. Petitioner asserts that the IPR2021-00104 petition presents grounds based on Barber, which does not expressly disclose the filter features added to the claims of the ’657 patent during prosecution. *Id.* at 3. In contrast, Petitioner asserts, the Petition in the present proceeding relies on Licata, which describes an

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RF filter between an RF power source and a pulse DC power source but does not expressly disclose bipolar aspects for the DC power source. *Id.* Petitioner also asserts that the IPR2021-00104 petition relies on Hirose to disclose the claimed filter, whereas this proceeding relies on Collins. *Id.* at 3–4. In addition, Petitioner argues that “due to the nature of the challenged claims, and the relevant prior art available, Petitioner had to separate the grounds into separate petitions in order to ensure the different grounds contained the necessary specificity.” *Id.* at 4.

Patent Owner responds that the two Petitions use one or two base references (i.e., Barber, or Barber and Belkind, in the IPR2021-00104 petition, and Licata and Kelly in this proceeding) for the claimed reactor and either Hirose or Collins for the claimed filter. Notice Response 2–4. Patent Owner argues that the Petitions start with the same base reactor system without a filter, and then make the same design-choice argument that it would have been obvious to add the claimed filter. *Id.* at 5.

Patent Owner’s arguments illustrate that there are more similarities than material differences between the challenges set forth in the petitions. Petitioner’s arguments highlight these similarities by stating that “Petition 1 relies on Hirose to disclose the claimed filter aspects, whereas Petition 2 relies on Collins.” Notice 3. Although the references relied on by the petitions to disclose the claimed reactor system differ, the petitions present similar scenarios in which base references are relied on for the reactor and another reference is relied on to disclose the claimed filter. As such, we disagree that Petitioner’s reliance on different art in each proceeding is a reason to institute multiple proceedings.

Petitioner also asserts that there are material differences between its two petitions because the petition in IPR2021-00104 challenges claims 1–21 whereas the present petition challenges claims 1–8 and 10–17. *Id.* at 4. We disagree. Because every claim challenged in the present Petition is also challenged in IPR2021-00104, Petitioner has not shown why the differences in the challenged claims is a material difference warranting institution of the present petition.

Nor has Petitioner has identified any other circumstances, such as the examples provided in the Consolidated Trial Practice Guide, that would justify a second *inter partes* review of the same patent. The '657 patent contains a reasonable number of claims (21 claims), the claims themselves are not particularly long, and evaluating the claims does not require analysis of a large number of limitations. Nor is there an alternative potential claim construction that requires Petitioner to set forth an alternative set of grounds; nor does there appear to be any dispute, at this time, as to the priority date of the '657 patent. *See generally* Pet.; Prelim. Resp.

Finally, we disagree with Petitioner that the Board has routinely instituted two parallel petitions under similar circumstances. Notice 5. None of the proceedings identified by Petitioner present similar circumstances to those presented here. The petitions before us do not involve, for example, a possible antedating issue for a reference cited in the challenges, as in IPR2019-00290 to -00293⁷ or in IPR2019-00237 and

⁷ The petitions in IPR2019-00291 and IPR2019-00293 were denied under Section 314 as improper multiple petitions. *See* IPR2019-00291, Paper 16, 8.

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IPR2019-00239.⁸ The petitions before us also do not involve, for example, challenges under 35 U.S.C. § 112 in the first petition and prior art challenges in the other petitions, as in PGR2019-00037, PGR2019-00040, and PGR2019-00042.⁹ We have determined that there are no material differences between the analyses presented in the first and the second petitions, and that the first and second petitions do not address claim limitations in different ways that could cause different outcomes, distinguishing these cases from IPR2019-01223 to -01227 and IPR2019-01464 and IPR2019-01465. Finally, IPR2019-00165 and IPR2019-00171 did not involve multiple petitions against the same patent and *inter partes* review was instituted in only one of those proceedings.

In view of the above, Petitioner has not adequately explained why the Petition in this proceeding contains sufficient material differences in its analysis to support instituting an additional *inter partes* review of the challenged claims. We acknowledge Petitioner’s novel arguments, including its “collaboration” argument and its “wealth of prior art” argument, but are not persuaded that these are sufficient reasons to justify instituting *inter partes* review on two parallel petitions, nor are we directed to any persuasive authority indicating that the Board has adopted a practice of doing so. Accordingly, because we institute an *inter partes* review of claims 1–21 of the ’657 patent on the grounds presented in the IPR2021-00104 Petition, we exercise our discretion under § 314(a) to deny institution in this proceeding.

⁸ The petition in IPR2019-00238 was denied under Section 314 as an improper multiple petition. See IPR2019-00238, Paper 15, 8.

⁹ The petition in PGR2019-00040 was denied under Section 324 as an improper multiple petition. PGR2019-00040, Paper 13, 13.

III. CONCLUSION

For the reasons set forth above, we determine that the circumstances weigh in favor of discretionary denial of this Petition. Accordingly, we exercise our discretion to deny institution under 35 U.S.C. § 314(a).

IV. ORDER

In consideration of the foregoing, it is hereby:

ORDERED that the Petition is *denied* and no trial is instituted.

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UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

APPLIED MATERIALS, INC.,
Petitioner,

v.

DEMARAY LLC,
Patent Owner.

IPR2021-00106
Patent 7,381,657 B2

Before CHRISTOPHER L. CRUMBLEY, KRISTINA M. KALAN, and
KIMBERLY McGRAW, *Administrative Patent Judges*.

CRUMBLEY, *Administrative Patent Judge*, concurring in the result.

I concur in result only.

IPR2021-00106
Patent 7,381,657 B2

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UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

APPLIED MATERIALS, INC.,
Petitioner,

v.

DEMARAY LLC,
Patent Owner.

IPR2021-00104
Patent 7,381,657 B2

Before CHRISTOPHER L. CRUMBLEY, KRISTINA M. KALAN, and
KIMBERLY McGRAW, *Administrative Patent Judges*.

McGRAW, *Administrative Patent Judge*.

DECISION
Granting Institution of *Inter Partes* Review
35 U.S.C. § 314

I. INTRODUCTION

Applied Materials, Inc. (“Petitioner”) filed a Petition (Paper 1, “Pet.”) requesting an *inter partes* review of claims 1–21 of U.S. Patent No. 7,381,657 B2 (Ex. 1001, “the ’657 patent”). Demaray LLC (“Patent Owner”) filed a Preliminary Response to the Petition (Paper 7, “Prelim. Resp.”). Pursuant to our authorization (Paper 9), Petitioner filed a Reply (Paper 10, “Pet. Reply”), and Patent Owner filed a Sur-reply (Paper 12, “PO Sur-reply”).

To institute an *inter partes* review, we must determine that the information presented in the Petition shows “there is a reasonable likelihood that the petitioner would prevail with respect to at least 1 of the claims challenged in the petition.” 35 U.S.C. § 314(a). For the reasons discussed below, after considering the parties’ submissions and the evidence of record, we determine that Petitioner has demonstrated a reasonable likelihood of prevailing with respect to at least one claim of the ’657 patent. Patent Owner has not provided us with a persuasive reason to exercise our discretion to deny institution. Thus, we institute an *inter partes* review.

A. Related Proceedings

The parties identify *Demaray LLC v. Samsung Electronics Co., Ltd.* et al., No. 6-20-cv-00636 (W.D. Tex.); *Demaray LLC v. Intel Corp.* No. 6-20-cv-00634 (W.D. Tex.) (collectively, the “Texas Litigations”); and *Applied Materials, Inc. v. Demaray LLC*, No. 5-20-cv-05676 (N.D. Cal.) as related matters. Pet. 1; Paper 5, 2. Each of these proceedings involves the ’657 patent. *Id.*

The parties further identify IPR2021-00106 as being filed by Petitioner against the ’657 patent. Pet. 2; Paper 5, 3. Patent Owner also

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identifies IPR2021-00103 and IPR2021-00105, which challenge U.S. Patent No. 7,544,276 B2, as related matters. Paper 5, 3.

B. Real Parties-In-Interest

Petitioner identifies Intel Corporation, Samsung Electronics Co., Ltd., Samsung Electronics America, Inc., Samsung Semiconductor, Inc., Samsung Austin Semiconductor, LLC, and itself as real parties-in-interest. Pet. 1. Patent Owner identifies itself as a real party-in-interest. Paper 5, 2.

C. The '657 Patent

The '657 patent, titled “Biased Pulse DC Reactive Sputtering of Oxide Films,” “relates to deposition of oxide and oxynitride films and, in particular, to deposition of oxide and oxynitride films by pulsed DC reactive sputtering.” Ex. 1001, code (54), 1:11–13. The '657 patent discloses that RF sputtering has been typically used to deposit oxide dielectric films but arcing can occur between sputtering target tiles used to make such films, which causes contamination in the deposited films. *Id.* at 2:25–30. The '657 patent further states that reactors for RF sputtering, particularly their power systems, are complicated. *Id.* at 2:30–38. The '657 patent discloses that reactive dc magnetron sputtering of nonconductive oxides is done rarely because insulating surfaces accumulate charge during deposition that results in arcing, damage to the sputtering power supply, and the production of particles that degrade the properties of deposited oxide films. *Id.* at 4:48–57.

Figure 1A of the '657 patent is reproduced below.

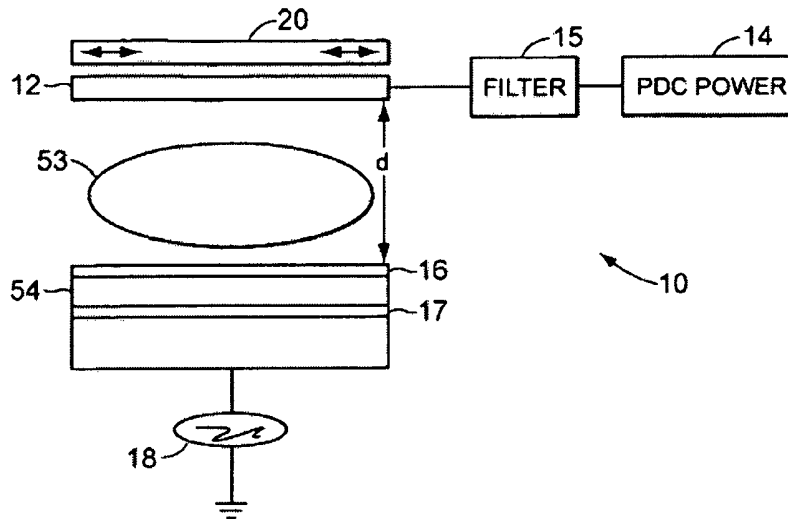


FIG. 1A

Figure 1A depicts a pulsed DC sputtering reactor

The '657 patent describes reactor apparatus 10 for sputtering material from target 12. *Id.* at 5:13–15. Magnet 20 is used to scan across the top of the target 12, which reduces local erosion of target 12 during sputtering. *Id.* at 5:35, 8:57–66. Substrate 16 is opposite and parallel to target 12. *Id.* at 5:29–30. Substrate 16 is capacitively coupled to electrode 17 via insulator 54. *Id.* at 5:32–36. Electrode 17 can be coupled to RF power supply 18. *Id.* at 5:34–35. The '657 patent explains that RF power supply 18 is used to avoid columnar structures in a deposited film, which can be detrimental for optical wave guide applications. *Id.* at 5:66–6:6. The '657 patent discloses that target 12 functions as a cathode when power is applied to the target 12, which creates plasma 53. *Id.* at 5:30–32.

Target 12 is electrically coupled through filter 15 to pulsed DC power supply 14. *Id.* at 5:25–26. The '657 patent discloses that the polarity of the power supplied to target 12 by pulsed DC power supply 14 oscillates between negative and positive potentials. *Id.* at 5:36–39. According to the

'657 patent, the insulating layer on the surface of target 12 discharges during the positive period, which prevents arcing. *Id.* at 5:39–41. The '657 patent discloses that the pulsing frequency must exceed a critical frequency, which depends on a target material, cathode current, and reverse time. *Id.* at 5:41–43.

Reactor apparatus 10 further includes filter 15, which prevents the RF power supply from coupling to into pulsed DC power supply 14. *Id.* at 5:56–57. According to the '657 patent, filter 15 can be a rejection filter, such as a 2 MHz band rejection filter when a 2 MHz power supply is used for RF power supply 18. *Id.* at 5:57–61. The '657 patent discloses that the bandwidth of filter 15 can be approximately 100 kHz. *Id.* at 5:61–63.

D. Illustrative Claim

Of the challenged claims, claims 1 and 2 are independent. Claim 2 is representative and is reproduced below with bracketed material and formatting added.

2. [a] A method of depositing an insulating film on a substrate, comprising:

[b] providing a process gas between a target and a substrate;

[c] [c1] providing pulsed DC power to the target [c2] through a narrow band rejection filter [c1] such that the voltage on the target alternates between positive and negative voltages;

[d] providing an RF bias that corresponds to the narrow band rejection filter to the substrate; and

[e] providing a magnetic field to the target;

[f] wherein an oxide material is deposited on the substrate, and the insulating film is formed by reactive sputtering in a mode between a metallic mode and a poison mode.

Ex. 1001, 23:16–27.

E. The Asserted Grounds of Unpatentability

Petitioner contends that claims 1–21 of the '657 patent are unpatentable on the following grounds. Pet. 3–5.

Reference(s)	Basis	Claim(s) Challenged
Barber, ¹ Hirose ²	§ 103 ³	2–4, 6, 8, 10–12, 21
Barber, Hirose, Dogheche ⁴	§ 103	5, 7
Barber, Hirose, Safi ⁵	§ 103	9
Barber, Hirose, Aokura ⁶	§ 103	12, 13
Barber, Hirose, Segal ⁷	§ 103	14–18
Barber, Hirose, Segal, Sakawaki ⁸	§ 103	19

¹ US 6,342,134 B1, issued Jan. 29, 2002 (Ex. 1005).

² US 6,485,602 B2, issued Nov. 26, 2002 (Ex. 1006).

³ The Leahy-Smith America Invents Act, Pub. L. No. 112-29, 125 Stat. 284 (2011) (“AIA”), amended 35 U.S.C. § 103. Because the '657 patent has an effective filing date prior to the effective date of the applicable AIA amendments, we refer to the pre-AIA versions of § 103.

⁴ E. Dogheche, *Growth and Optical Characterization of Aluminum Nitride Thin Films Deposited on Silicon by Radio-Frequency Sputtering*, Appl. Phys. Lett. 74, 1209 (1999) (Ex. 1029).

⁵ I. Safi, *A Novel Reactive Magnetron Sputtering Technique for Producing Insulating Oxides of Metal Alloys and Other Compound Thin Films*, Surface and Coatings Tech. 135, 48 (2000) (Ex. 1039).

⁶ JP H10102247 A, published Apr. 21, 1998 (Ex. 1068) ((English translation (pp. 1–12); Verified Statement of Translation (p. 13); Japanese language document (pp. 14–24).

⁷ US 2001/0047838 A1, published Dec. 6, 2001 (Ex. 1069).

⁸ US 2001/0031383 A1, published Oct. 18, 2001 (Ex. 1043).

Reference(s)	Basis	Claim(s) Challenged
Barber, Hirose, Sill ⁹	§ 103	20
Barber, Hirose, Sellers ¹⁰	§ 103	1
Barber, Hirose, Belkind ¹¹	§ 103	2–4, 6, 8, 10, 11, 12, 21
Barber, Hirose, Belkind, Dogheche	§ 103	5, 7
Barber, Hirose, Belkind, Safi	§ 103	9
Barber, Hirose, Belkind, Aokura	§ 103	12, 13
Barber, Hirose, Belkind, Segal	§ 103	14–18
Barber, Hirose, Belkind, Segal, Sakawaki	§ 103	19
Barber, Hirose, Belkind, Sill	§ 103	20
Barber, Hirose, Belkind, Sellers	§ 103	1

In support of its unpatentability arguments, Petitioner relies on the declaration of Dr. Vivek Subramanian. Ex. 1002 (“Subramanian Declaration”). Patent Owner relies on the declaration of Dr. Alexander Glew. Ex. 2002 (“Glew Declaration”).

II. ANALYSIS

A. Claim Construction

We apply the same claim construction standard that would be used to construe the claim in a civil action under 35 U.S.C. § 282(b), including construing the claim accordance with the ordinary and customary meaning

⁹ US 6,284,110 B1, issued Sept. 4, 2001 (Ex. 1057).

¹⁰ US 5,651,865, issued July 29, 1997 (Ex. 1007).

¹¹ A. Belkind et al., *Pulsed-DC Reactive Sputtering of Dielectrics: Pulsing Parameter Effects*, Society of Vacuum Coaters, 43rd Annual Technical Conference Proceedings, 86 (2000) (Ex. 1008).

of such claim as understood by one of ordinary skill in the art and the prosecution history. 37 C.F.R. § 42.100(b) (2020). Under this standard, claim terms are given their ordinary and customary meaning as would have been understood by a person of ordinary skill in the art at the time of the invention and in the context of the entire patent disclosure. *Philips v. AWH Corp.*, 415 F.3d 1303, 1312–14 (Fed. Cir. 2005) (en banc). Only terms that are in controversy need to be construed, and then only to the extent necessary to resolve the controversy. *Vivid Techs., Inc. v. Am. Sci. & Eng'g, Inc.*, 200 F.3d 795, 803 (Fed. Cir. 1999); *see also Nidec Motor Corp. v. Zhongshan Board Ocean Motor Co.*, 868 F.3d 1013, 1017 (Fed. Cir. 2017) (applying *Vivid Techs.* in the context of an *inter partes* review).

Neither party asserts a claim construction for the challenged claims. Pet. 11; *see generally* Prelim. Resp. On this record, we determine that no claim terms require express construction.

B. Principles of Law

“In an [*inter partes* review], the petitioner has the burden from the onset to show with particularity why the patent it challenges is unpatentable.” *Harmonic Inc. v. Avid Tech., Inc.*, 815 F.3d 1356, 1363 (Fed. Cir. 2016) (citing 35 U.S.C. § 312(a)(3) (requiring *inter partes* review petitions to identify “with particularity . . . the evidence that supports the grounds for the challenge to each claim”)). This burden of persuasion never shifts to Patent Owner. *See Dynamic Drinkware, LLC v. Nat'l Graphics, Inc.*, 800 F.3d 1375, 1378 (Fed. Cir. 2015) (discussing the burden of proof in *inter partes* review).

A claim is unpatentable under § 103(a) if the differences between the claimed subject matter and the prior art are such that the subject matter, as a whole, would have been obvious at the time the invention was made to a

person having ordinary skill in the art to which said subject matter pertains. *KSR Int'l Co. v. Teleflex Inc.*, 550 U.S. 398, 406 (2007). The question of obviousness is resolved on the basis of underlying factual determinations, including (1) the scope and content of the prior art; (2) any differences between the claimed subject matter and the prior art; (3) the level of skill in the art; and (4) when in evidence, objective indicia of non-obviousness (i.e., secondary considerations). *Graham v. John Deere Co.*, 383 U.S. 1, 17–18 (1966). “To satisfy its burden of proving obviousness, a petitioner cannot employ mere conclusory statements. The petitioner must instead articulate specific reasoning, based on evidence of record, to support the legal conclusion of obviousness.” *In re Magnum Oil Tools Int'l, Ltd.*, 829 F.3d 1364, 1380 (Fed. Cir. 2016).

We analyze the asserted grounds of unpatentability in accordance with these principles to determine whether Petitioner has met their burden to establish a reasonable likelihood of success at trial.

C. Level of Ordinary Skill in the Art

Petitioner argues:

A person of ordinary skill in the art (“POSITA”) would have had, at the time of the ’657 patent (March 2002): a Master’s degree in Electrical Engineering or Material Science (or an equivalent subject) plus at least two years of relevant experience (e.g., sputtering deposition of films on substrates (Ex. 1001, 1:10–14, 2:45–47)), or a Bachelor’s degree in Electrical Engineering or Material Science (or an equivalent subject) plus at least four years of relevant experience. More education can substitute for practical experience, and vice versa.

Pet. 9 (citing Ex. 1002 ¶¶ 19–20). At this stage of the proceeding, Patent Owner does not articulate a level of skill for a POSITA. *See generally* Prelim. Resp. Neither party argues that the decision whether to institute trial

depends on our adoption of any particular definition of one of ordinary skill in the art.

In light of the record before us, we adopt Petitioner’s proposal regarding the level of one of ordinary skill in the art, except that we delete the qualifier “at least” to eliminate vagueness as to the appropriate amount of relevant experience. The qualifier expands the range without an upper bound, i.e., encompassing an unlimited amount of years of experience, and thus, does not meaningfully indicate the level of ordinary skill in the art. The level of ordinary skill in the art is also reflected by the prior art of record. *See Okajima v. Bourdeau*, 261 F.3d 1350, 1355 (Fed. Cir. 2001).

D. Discretion under 35 U.S.C. § 314(a) Due to Parallel District Court Proceedings

Patent Owner argues that the Board should exercise its discretion to deny institution under 35 U.S.C. § 314(a) because the trial in the Texas Litigations, which involve real parties-in-interest Samsung and Intel, is scheduled for December 27, 2021, which is approximately 4.5 months before our projected final written decision and that litigation is in advanced stage. Prelim. Resp. 4–8 (citing Ex. 2005, 4).

For the reasons stated below, we are not persuaded to exercise our discretion to deny institution for this reason.

Institution of an *inter partes* review is discretionary. *See* 35 U.S.C. § 314(a) (authorizing institution of an *inter partes* review under particular circumstances, but not requiring institution under any circumstances); 37 C.F.R. § 42.108(a) (“[T]he Board may authorize the review to proceed.”). *Cuozzo Speed Techs., LLC v. Lee*, 136 S. Ct. 2131, 2140 (2016) (“[T]he agency’s decision to deny a petition is a matter committed to the Patent Office’s discretion.”); *SAS Inst. Inc. v. Iancu*, 138 S. Ct. 1348, 1356 (2018)

(“[Section] 314(a) invests the Director with discretion on the question whether to institute review” (emphasis omitted)); *Harmonic Inc.*, 815 F.3d at 1367 (“[T]he PTO is permitted, but never compelled, to institute an IPR proceeding.”).

When determining whether to exercise discretion to deny institution due to an earlier trial date, we consider the following: (1) whether the district court granted a stay or evidence exists that one may be granted if this proceeding is instituted; (2) proximity of the district court's trial date to the Board's projected statutory deadline for a final written decision; (3) investment in the parallel proceeding by the court and the parties; (4) overlap between issues raised in the petition and in the parallel proceeding; (5) whether the petitioner and the defendant in the parallel proceeding are the same party; and (6) other circumstances that impact the Board's exercise of discretion, including the merits. *Apple Inc. v. Fintiv, Inc.*, IPR2020-00019, Paper 11, 5–6 (Mar. 20, 2020) (precedential). In evaluating the factors, we take “a holistic view of whether efficiency and integrity of the [patent] system are best served by denying or instituting review.” *Id.* at 6.

1. Whether a Stay Exists or Is Likely to Be Granted if a Proceeding Is Instituted

As to factor 1, Petitioner asserts that it filed a motion in the Northern District of California for a preliminary injunction to enjoin the Texas Litigations from proceeding. Pet. 74 (citing Ex. 1078). This motion, however, has since been denied. Ex. 2004. Petitioner provides no other evidence indicating that a motion to stay the Texas Litigations has been filed by the parties to those actions or would be granted, if filed. In the absence of a motion for stay, we cannot assess Petitioner's arguments to the District

Court that a stay is appropriate, nor Patent Owner's opposition, if any. As a result, we have no basis to evaluate the likelihood a stay would be granted. Accordingly, we determine this factor to be neutral.

2. *Proximity of the Court's Trial Date to the Board's Projected Statutory Deadline*

Jury selection for the Texas Litigations is scheduled to begin on December 27, 2021, which predates our projected final written decision deadline by approximately four and a half months. *See* Ex. 2005 (Scheduling Order), 4 (stating the court "expects to set this date [i.e., December 27, 2021] at the conclusion of the *Markman* hearing" but that "if the actual trial date materially differs from the Court's default schedule, the Court will consider reasonable amendments to the case schedule post-*Markman* that are consistent with the Court's default deadlines"). Petitioner asserts that it is unlikely trial will start in December 2021, because the *Markman* hearing has been postponed pending resolution of a transfer motion, and the Court's order governing patent proceedings sets a default trial date "52 weeks after *Markman* hearing (or as soon as practicable)." Pet. Reply 2 (citing, *inter alia*, Judge Albright's "Order Governing Proceedings - Patent Cases 022321.pdf" available at <https://www.txwd.uscourts.gov/judges-information/standing-orders/>). Petitioner further asserts that multiple trials have been scheduled for December 27, 2021, thus providing further evidence that the trial date will likely be rescheduled. *Id.*

Although Patent Owner acknowledges that the *Markman* hearing has been cancelled, Patent Owner asserts that it expects the *Markman* hearing to be completed by mid-May 2021, and that as of yet, trial is still scheduled for December 27, 2021. PO Sur-reply 2-3.

Weighing all of this together, there is substantial uncertainty whether the Texas Litigations will proceed to trial on December 27, 2021 in view of a number of factors: the delay of the *Markman* hearing; the district court's general schedule in its Order Governing Patent Proceedings; the possible delay due to the Transfer Motion; and the scheduling of two trials on the same day. The parties cite no evidence to suggest otherwise. While we cannot know at this date how long a potential delay might be, we note that Patent Owner's suggestion of a mid-May 2021 *Markman* ruling, combined with the District Court's default trial date of 52 weeks later, could result in the trial date occurring at the same time as, if not slightly after, our final written decision in this matter. Given the wide range of potential trial dates and the uncertainty surrounding them given the District Court's cancellation of the *Markman* hearing, we cannot evaluate the proximity of the trial date to our statutory deadline, as required by the second *Fintiv* factor. As a result, we determine this factor to be neutral.

3. *Investment in the Parallel Proceeding by the Court and Parties*

Factor 3 does not weigh in favor of exercising discretion to deny institution. The *Markman* hearing has been delayed until the district court resolves the pending transfer motion. The deadlines for fact discovery and service of final infringement and invalidity contentions have likewise been delayed. *See, e.g.*, PO Sur-reply 3 (stating that fact discovery will likely begin mid-May and that additional investment will continue until the expected final written decision deadline). Thus, as of this Decision, there has been a relatively small investment in the Texas Litigations by the parties to those actions, and significant effort remains to be expended. Patent Owner's arguments regarding the accrual of investment after the

time of this Decision are unpersuasive, because we consider the investment “at the time of the institution decision.” *Fintiv*, Paper 11, 9–10.

Moreover, Petitioner was diligent in filing its petition on October 22, 2020, approximately three months after the complaints for the Texas Litigation were filed. *See* Paper 4 (Notice of Filing Date), 1; Ex. 1075 (Complaint), 25 and Ex. 1076 (Complaint), 27 (both indicating that the complaints in the Texas Litigations were filed July 14, 2020).

Considering Petitioner’s expeditious filing of the Petition and the relatively small amount of investment in the Texas Litigations, this factor weighs against exercising our discretion to deny institution.

4. Overlap between Issues Raised in the Petition and in the Parallel Proceeding

Petitioner asserts that there is no evidence that the same invalidity grounds sought here will be at issue in the Texas litigations (Pet. 76) and that the defendants in the Texas Litigations provided a stipulation that “ensures no overlap will exist with any asserted grounds upon institution” (Pet. Reply 5 (citing Ex. 2003, 27–30)). Patent Owner responds that there is substantial overlap between issues raised in the petition and the Texas Litigations because the real parties-in-interest “have included all the asserted combinations in the district court action and against all challenged claims” at issue in the present proceeding (Prelim. Resp. 7–8) and the stipulation does not obviate concerns relating to overlapping issues (PO Sur-reply 4). Patent Owner states the stipulation only applies to the “specific invalidity ground instituted by the PTAB” and “any invalidity ground based” on Barber if trial is instituted and that the defendants in the Texas Litigations can still raise any of the other cumulative prior art disclosed in their invalidity contentions. PO Sur-reply 4–5 (citing Ex.

2003, 27–40 & n.4); *Sotera Wireless, Inc. v. Masimo Corp.*, IPR 2020-01019, Paper 12, 13–14 (PTAB Dec. 1, 2020) (precedential)).

Because all of the art asserted in the present proceeding, including the primary references Barber and Hirose, are also asserted in the Texas Litigations, there is overlap between the issues raised in the petition and in the Texas Litigations. Ex. 2003, 9–10, 19, 22. We recognize that the Texas Litigation defendants have stipulated that, if *inter partes* review is instituted on a specific invalidity ground for an asserted patent (e.g., the '657 patent), they agree “to not pursue invalidity contentions for that Asserted Patent in this litigation on that specific invalidity ground instituted by the PTAB.” *Id.* at 29. The defendants also stipulate that, if *inter partes* review is instituted on a specific invalidity ground that includes Barber for an asserted patent, they agree “to not pursue invalidity contentions in this litigation for any invalidity ground based on *Barber*.” *Id.* at 29–30. The defendants’ stipulation, however, does not preclude them from relying on Barber “or any other reference as a state of the art reference to demonstrate the knowledge of state of the art by one of ordinary skill in the art at the relevant time.” *Id.* at 30 n.5.

We recognize that the defendants’ stipulation does not state that they will not pursue any ground raised or that could have been reasonably raised in the Petition, similar to the “broad stipulation” discussed in *Sotera*. The narrower stipulation made by the Texas defendants, however, “mitigates to some degree the concerns of duplicative efforts between the district court and the Board, as well as concerns of potentially conflicting decisions.” *See Sand Revolution II, LLC v. Cont’l Intermodal Group – Trucking LLC*, IPR2019-01393, Paper 24 (PTAB June 16, 2020) (informative).

Accordingly, we find this factor weighs slightly against exercising our discretion to deny.

5. *Whether the Petitioner and the Defendant in the Parallel Proceeding Are the Same Party*

Petitioner argues that “Petitioner is not the same party in the Texas Litigations.” Pet. 76. Patent Owner contends that the defendants of the Texas Litigations are real parties-in-interest for this proceeding and have coordinated with Petitioner in filing this Petition. Prelim. Resp. 8. Patent Owner notes that Petitioner does not dispute Patent Owner’s argument for factor 5. PO Sur-reply 5.

This factor considers the relationship between a Petitioner and a defendant to a court proceeding. *Fintiv*, Paper 11, 13–14 (“If a petitioner is unrelated to a defendant in an earlier court proceeding, the Board has weighed this fact against exercising discretion to deny institution.”). Here, Petitioner identifies the defendants of the Texas Litigations as real parties-in-interest. Pet. 1; Ex. 2003, 1. Petitioner also acknowledges that they have coordinated efforts with the defendants when preparing the petitions filed against the ’276 patent. Paper 2, 2. Thus, Petitioner and the Texas Litigations defendants are not “unrelated.” *See Fintiv*, Paper 11, 13–14. At the same time, Petitioner and the Texas defendants are not “the same party.”

Although Petitioner and the Texas defendants are not “the same party,” because Petitioner and the Texas defendants are related, and because of the degree of involvement by the Texas defendants in the preparation of the instant Petition, we find this factor weighs slightly in favor of exercising discretion to deny institution.

6. *Other Circumstances that Impact the Board's Exercise of Discretion, Including the Merits*

Petitioner contends the strength of its asserted grounds challenging the claims of the '657 favor institution (Pet. 77; Pet. Reply 4), while Patent Owner contends Petitioner's failure to show a reasonable likelihood that it will prevail on its challenges supports denying institution (Prelim. Reply 8; PO Sur-reply 5). Patent Owner also urges the Board to strike Petitioner's Reply arguments regarding *Fintiv* factor #6, because the Reply improperly reargues the merits of the challenges set forth in the Petition or seeks to bolster its proposed grounds of unpatentability. PO Sur-reply 5.

As the Board explained in *Fintiv*, we considered this factor as “part of a balanced assessment of all the relevant circumstances in the case.” *Fintiv*, Paper 11, 14. The goal of that assessment is to determine “whether efficiency and integrity of the system are best served by denying or instituting review.” *Id.* at 6. The assessment requires consideration of the “strengths or weaknesses regarding the merits,” but “[t]his is not to suggest that a full merits analysis is necessary to evaluate this factor.” *Id.* at 15–16.

For the reasons discussed below regarding Petitioner's obviousness challenges, Petitioner has demonstrated a reasonable likelihood that it would prevail on its challenge, and thus this factor weighs against denying institution. We do not view statements made in Petitioner's Reply addressing this factor as rearguing the merits of its challenges or bolstering its proposed grounds to a point that would require striking portions of Petitioner's Reply regarding *Fintiv* factor #6. Reply. 5. Accordingly, this factor is neutral.

7. *Evaluating the Fintiv Factors*

We have considered the circumstances and facts before us in view of the *Fintiv* factors. Because our analysis is fact driven, no single factor is determinative of whether we exercise our discretion to deny institution under § 314(a). We take “a holistic view of whether efficiency and integrity of the system are best served by denying or instituting review” when evaluating these factors. *Fintiv*, Paper 11, 6.

Based on the facts of this case, the potential overlap of issues and the potential for an earlier trial date for the Texas Litigations is outweighed by the uncertainty in that trial date due to the Transfer Motion, the relatively small investment by the parties and the district court, Petitioner’s diligence in filing the Petition, the stipulation by the defendants in the Texas Litigations, and the relationship of Petitioner to the Texas defendants. Accordingly, evaluating all of the factors on this record, we determine that the circumstances presented here do not support exercising our discretion under § 314(a) to deny institution of *inter partes* review.

E. Discretion under 35 U.S.C. § 314(a) Due to Parallel Petitions

Petitioner filed two petitions challenging the patentability of claims of the ’657 patent. The first petition, filed in the present proceeding, challenges claims 1–21 on grounds based on Barber and Hirose and other references, while the second petition, filed in IPR2021-00106, challenges claims 1–8 and 10–17 based on Licata, Kelly, and Collins and other references. Pet. 3–5; IPR2021-00106, Paper 1, 7–8. Pursuant to the Board’s Consolidated Trial Practice Guide, Petitioner filed a *Notice Regarding Multiple Petitions*. Paper 2 (“Notice”); PTAB Consolidated Trial Practice Guide, 59–60 (Nov. 2019) (available at <https://www.uspto.gov/TrialPracticeGuideConsolidated>).

Petitioner requests that we institute in both IPRs, but that we consider the Petition in this proceeding first. Notice, 1. Patent Owner filed a Response to Petitioner's Notice stating that Petitioner has not shown justification for two petitions. Paper 8, 1–5.

Because Petitioner ranked this Petition first and Patent Owner does not object, we decline to exercise our discretion to deny institution under 35 U.S.C. § 314(a) in this proceeding based on the filing of an additional petition challenging the same patent. In a separate decision, we address the petition filed in IPR2021-00106.

F. Discretion under 35 U.S.C. § 325(d)

Patent Owner argues that the Petition should be denied under 35 U.S.C. § 325(d) because Petitioner relies on art in substantially the same manner as art previously before the Office and presents arguments that are the same or substantially the same as arguments previously before the Office. Prelim. Resp. 61–67.

To evaluate whether to exercise discretion under 35 U.S.C. § 325(d), the Board uses the following two-part framework: (1) whether the same or substantially the same art previously was presented to the Office or whether the same or substantially the same arguments previously were presented to the Office; and (2) if either condition of first part of the framework is satisfied, whether the petitioner has demonstrated that the Office erred in a manner material to the patentability of challenged claims

Advanced Bionics LLC v. MED-EL Elektromedizinische Geräte GmbH, IPR2019-01469, Paper 6 at 8 (PTAB Feb. 13, 2020) (precedential).

1. Advanced Bionics Framework Part (1)

For part (1) of the *Advanced Bionics* framework, Patent Owner contends that Petitioner relies on Barber and Hirose in substantially the same manner as the Examiner relied on Smolanoff during prosecution of the '657

patent. Prelim. Resp. 62–64. For example, Patent Owner states that “during the prosecution, the examiner initially found that Smolanoff taught all limitations but the claimed filter type, which the examiner viewed as merely a design choice.” *Id.* at 62–63 (citing Ex. 1004, 956–57; Ex. 2001). Patent Owner also argues that “Petitioner’s use of Hirose is no different than how the examiners originally regarded the selection of a filter: that it was merely a modular design choice” and that “Petitioner uses Hirose in the same manner as the examiner did with Smonaoloff [sic], which is to show that it was common knowledge that a filter could be used to block interfering RF energy.” *Id.* at 65–66 (citing Pet. 23–28; Ex. 1004, 957, 978–79, 992).

Patent Owner’s arguments are unpersuasive. During prosecution, the Examiner found that Smolanoff disclosed a system that used pulsed DC power and RF bias power. Ex. 1004, 956. The Examiner concluded that “[u]sing a specific type of filter is a matter of design choice.” *Id.* at 957. In contrast, relying on the declaration testimony of Dr. Subramanian, Petitioner argues that Hirose discloses or suggests the claimed type of filter and articulates reasons POSITA would have modified the system of Barber to add a narrow band rejection filter as taught by Hirose. *See, e.g.*, Pet. 19–23 (citing Ex. 1002). Therefore, we do not agree that Petitioner relies on Barber and Hirose in substantially the same manner as the Examiner relied on Smolanoff during prosecution of the ’657 patent.

Patent Owner also contends that Petitioner makes arguments the same as, or substantially similar to, arguments previously made before the Office. Prelim. Resp. 64–65. According to Patent Owner,

Petitioner argues that a POSITA would have added a filter to Barber’s process because “a POSITA would have understood that choosing to reject a specific frequency (or narrow band of frequencies) depending on the bandwidth of RF supply 235

selected in *Barber*'s system would have been a known way to achieve the benefits . . . known at the time.”

Id. at 64 (citing Pet. 24). Patent Owner argues that Petitioner's reason to combine Barber and Hirose is the same as or substantially the same as the Examiner's use of design choice to select a specific type of filter. *Id.* at 65 (citing Pet. 24; Ex. 1004, 957, 978--79).

These arguments are also unpersuasive. During prosecution, the Examiner addressed Smolanoff's lack of disclosure of a specific type of filter by concluding that using a specific type of filter is a matter of design choice. Ex. 1004, 957. Here, Petitioner asserts that Hirose discloses the claimed type of filter and provides reasons why a POSITA would have added a narrow band rejection filter to the system of Barber. *See, e.g.*, Pet. 19--23. Patent Owner's arguments do not sufficiently explain how Petitioner's reasons for combining Barber and Hirose are the same as, or substantially the same as, the Examiner's reasons to modify Smolanoff. Furthermore, the Examiner did not have the benefit of considering Barber and Hirose in the combination asserted by Petitioner, as supported by the testimony by Dr. Subramanian. Pet. 73 (citing the benefit of expert testimony when arguing against discretionary denial over Belkind, which was identified in an Information Disclosure Statement during prosecution).

Therefore, we also do not agree with Patent Owner that the Petition presents the same or substantially the same arguments that were previously presented to the Office.

2. *Advanced Bionics Framework Part (2)*

Because part (1) of the *Advanced Bionics* framework has not been satisfied, we do not address part (2) of the framework. For these reasons, we

do not we exercise our discretion under § 325(d) to deny institution in this proceeding.

G. Asserted Obviousness of Claims 2–4, 6, 8, 10–12, and 21 over Barber and Hirose

Petitioner argues that claims 2–4, 6, 8, 10–12, and 21 are unpatentable over Barber and Hirose. Pet. 12–41.

1. Barber

Barber is a patent titled “Method for Producing Piezoelectric Films with Rotating Magnetron Sputtering System.” Ex. 1005, code (54).

Barber’s Figure 2 is reproduced below.

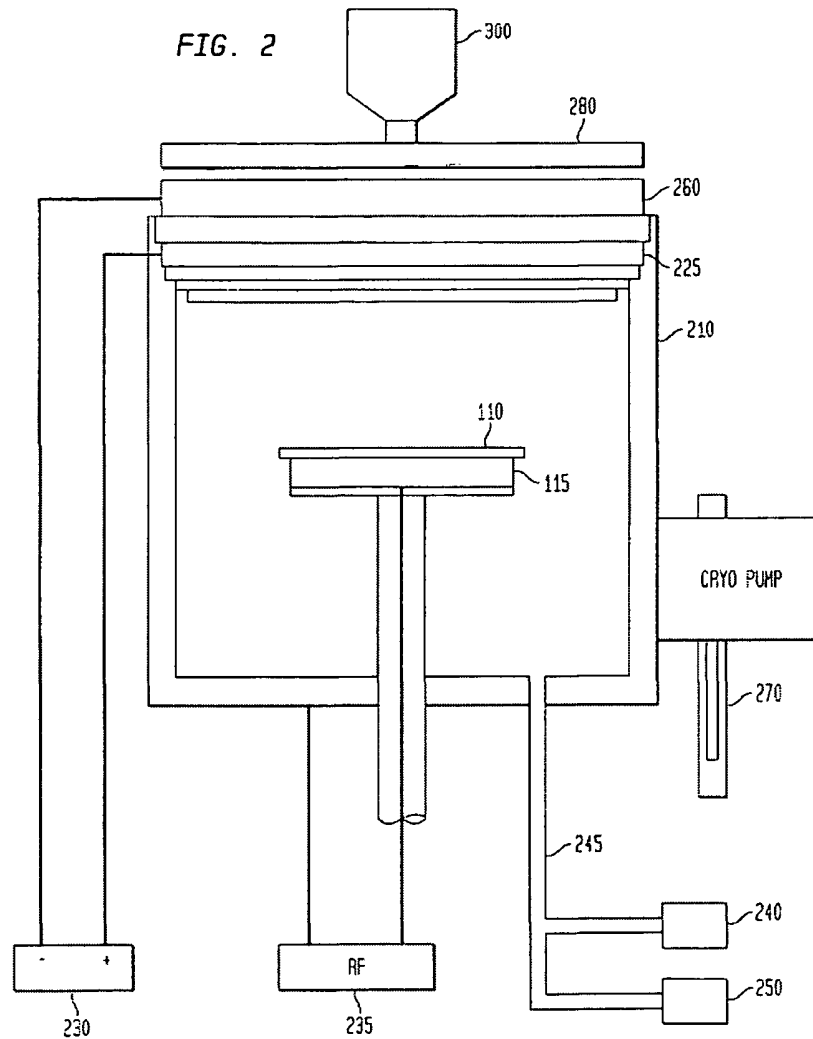


Figure 2 is a schematic diagram of a reactive sputtering arrangement. *Id.* at 3:22–24. The arrangement includes chamber 210 and a pair of electrodes (target 260 and anode ring 225) within the chamber. *Id.* at 6:4–6. Barber discloses that an “electric potential applied to the electrodes may be controlled by pulsed DC power source 230 or other suitable source.” *Id.* at 6:6–8. First flow control source 240 provides noble gases to the chamber 210 and second flow control source 250 provides a reactive gas to the chamber 210. *Id.* at 6:9–12.

Substrate 110 is also positioned within chamber 210 adjacent to rotating magnet assembly 280. *Id.* at 6:14–18. Barber discloses RF power supply 235 to apply a bias voltage to substrate platen 115 to control tensile film stress. *Id.* at 6:29–31. Barber further describes using a cross-over curve to optimize its reactive sputtering deposition process. *Id.* at 7:2–5. Specifically, Barber discloses introducing noble gas into its chamber and then introducing reactive gas incrementally so the flow rate of the reactive gas is a rate corresponding substantially to, but greater than, the flow rate for a cross-over point between producing a metallic film and an insulator. *Id.* at 7:17–29.

2. *Hirose*

Hirose is a patent titled “Plasma Processing Apparatus.” Ex. 1006, code (54). *Hirose* “relates to a plasma processing apparatus capable of suppressing a damage due to sputtering to a wall surface of a processing chamber during plasma occurrence.” *Id.* at 1:14–17. *Hirose* discloses a conventional plasma processing apparatus that applies high-frequency power with two types of frequency. *Id.* at 1:19–23. *Hirose* teaches that simultaneously applying two types of high-frequency power decreases an effective ground area to one electrode in comparison to when a single

frequency is used and this causes a sputter rate to increase for the ground area and more damage for the wall of a processing chamber. *Id.* at 2:1–9.

Hirose's Figure 1 is reproduced below.

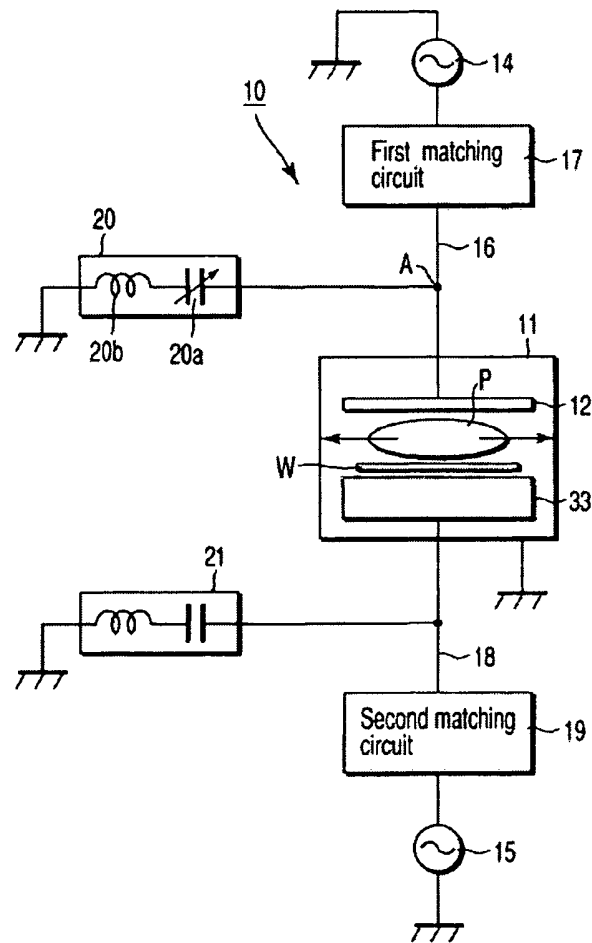


FIG. 1

Figure 1 is a configuration chart showing an embodiment of a plasma processing apparatus. *Id.* at 3:1–3. Hirose describes plasma processing apparatus 10 that includes processing chamber 11, upper electrode 12, and lower electrode 13.¹² *Id.* at 3:45–51. Hirose discloses that lower electrode

¹² Hirose states lower electrode 13 is depicted in Figure 1. Ex. 1006, 3:50. However, Figure 1 does not include reference numeral 13. We assume that

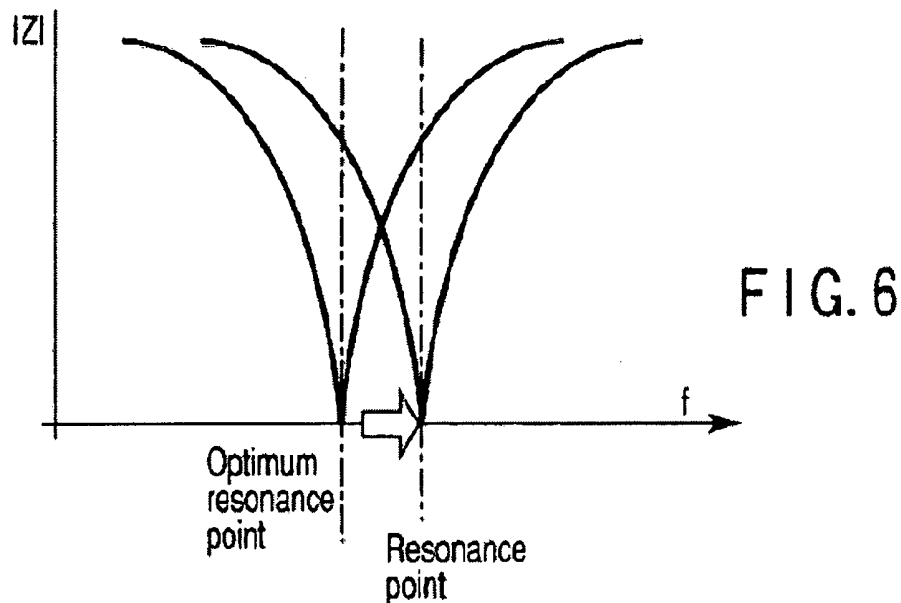
13 functions as a mount for a processing object, such as a semiconductor wafer. *Id.* at 3:56–58. Upper electrode 12 is connected to first high-frequency power supply 14 and lower electrode 13 is connected to second high-frequency power supply 15. *Id.* at 3:51–56. Hirose discloses that first high-frequency power supply 14 may apply, for example, 60 MHz power to upper electrode 12 and the second high-frequency power supply 15 may apply, for example, 2 MHz power to lower electrode 13. *Id.* at 3:59–60, 64–65.

Hirose describes first filter circuit 20 and second filter circuit 21. *Id.* at 4:9–11. First filter circuit 20 includes an inductor-capacitor (LC) resonant circuit with a varying circuit constant. *Id.* at 4:11–13. Hirose states that “[t]he LC series resonant circuit selectively filters a high-frequency current output from the second high-frequency power supply 15 for preventing the current from reaching the first high-frequency power supply 14.” *Id.* at 4:13–16. Hirose also discloses that second filter circuit 21 prevents high-frequency current from first high-frequency power supply 14 from reaching second high-frequency power supply 15. *Id.* at 4:18–21.

Hirose describes how to set the circuit constant of the first filter circuit 20. *Id.* at 4:39–40. First and second high-frequency power supplies 14, 15 apply power to upper electrode 12 and lower electrode 13, respectively, and a capacitor capacity of first filter circuit 20 is varied by measuring a voltage waveform. *Id.* at 4:42–47. Hirose teaches that when first and second high-frequency power supplies 14, 15 provide power at respective frequencies of 60 MHz and 2 MHz, a capacitor capacity of 2500 pF for first filter circuit 20

Figure 1’s reference numeral 33 may be a typographical error and represent the lower electrode 13. *See id.* at 6:29 (describing the embodiment depicted in Figure 7 as including lower electrode 33).

provides an optimum resonance point. *Id.* at 4:52–57, 5:1–3. This effect is depicted in Hirose’s Figure 6, which is reproduced below.



Hirose is concerned with sputtering the chamber wall of its device and examines the relationship between the sputter rate and the capacitor capacity. *Id.* at 2:1–10, 5:3–6, 5:11–12. Hirose states the filter circuit set at 2.5 μH and a capacitor capacity of 2000 pF greatly decreases the sputter rate as compared to the filter circuit set at 2.5 μH and a capacitor capacity of 2500 pF and that a filter with 2.5 μH and 2000 pF “is superior to the optimally resonant filter circuit” with 2.5 μH and 2500 pF, which decreases the sputter rate, but is “slightly inferior to the latter with respect to the resonance phenomenon.” *Id.* at 5:49–62.

3. Analysis for Claim 2

a) Preamble

The preamble of claim 2 recites “A method of depositing an insulating film on a substrate, comprising.” Ex. 1001, 23:16–17. Petitioner contends that Barber discloses depositing an insulating film (e.g., an aluminum nitride

film) on a substrate. Pet. 12–15 (citing, *inter alia*, Ex. 1005, code (57), 1:15–19, 2:1–5, 2:50–52, 4:31–34, 5:62–6:13, 6:15–17, 6:27–31, 6:42–46, 6:58–62, 7:1–8:12, 8:43–9:22, 9:23–10:11, Figs. 1, 2, 5; Ex. 1002 ¶¶ 56–60). Patent Owner does not dispute Petitioner’s assertions at this stage of the proceeding. *See generally* Prelim. Resp. We have reviewed Petitioner’s arguments and evidence and determine that Petitioner sufficiently shows that Barber teaches a method of depositing an insulating film on a substrate as recited in the preamble.¹³

b) Claim element 2[b]- Providing a Process Gas

Claim element 2[b] recite “providing a process gas between a target and a substrate.” Ex. 1001, 23:18. Petitioner asserts that Barber discloses the use of a noble gas and a reactive gas into chamber 210 between target 260 and substrate 110. Pet. 15 (citing Ex. 1005, 2:1–9, 6:6–17, 7:30–36, 8:49–52, Fig. 2; Ex. 1002 ¶ 61). Patent Owner does not dispute Petitioner’s contentions at this stage of the proceeding. *See generally* Prelim. Resp.

We have reviewed Petitioner’s arguments and evidence and determine that Petitioner sufficiently shows Barber discloses the subject matter recited in claim element 2[b].

c) Claim element 2[c] - Providing Pulsed DC Power through a Narrow Band Rejection Filter

Claim elements 2[c] recites “providing pulsed DC power to the target *through a narrow band rejection filter* such that the voltage on the target alternates between positive and negative voltages.” Ex. 1001, 23:19–21 (emphasis added). Petitioner addresses this claim element in two parts:

¹³ Because Petitioner sufficiently shows Barber teaches the subject matter recited in the preamble, we do not determine whether the preamble is limiting.

claim element 2[c1] – “providing pulsed DC power to the target . . . such that the voltage on the target alternates between positive and negative voltages” and claim element 2[c2] – “through a narrow band rejection filter.” We address the arguments for these two parts separately below.

(1) claim element 2[c1] – “providing pulsed DC power to the target . . . such that the voltage on the target alternates between positive and negative voltages”

Petitioner asserts that Barber’s power source 230 provides pulsed DC power to a target as recited in claim element 2[c1]. Pet. 15–17. Petitioner contends Barber discloses applying positive DC pulses to target 260 (an electrode) at a pulse frequency and a “reverse-bias pulse width,” which refers to a positive bias pulse. Pet. 15–16 (citing Ex. 1005, 2:21–26, 5:39–43, 5:50–51; 7:14–17, 8:45–48, 8:66–9:3, 9:6–11, 9:17–22, 9:48–53; Ex. 1002 ¶¶ 63–72; Ex. 1008, 1–3). Petitioner also asserts that the voltage on the target alternates between positive and negative voltages because Barber’s pulsed DC power supply 230 necessarily provides a negative bias to target 260 at times during its reactive-sputtering process in order to attract positively charged gas ions to facilitate target material bombardment for deposition onto the substrate. Pet. 16–17 (citing Ex. 1005, 2:1–5, 7:30–34; Ex. 1002 ¶¶ 66–68). Petitioner contends that in pulsed DC reactive sputtering, such as that disclosed in Barber, negative cathode/target bias is reversed periodically to positive for depositing insulating films. Pet. 17 (citing Ex. 1005, 2:14–26, 6:4–8, 8:44–9:22; Ex. 1060, 4:24–5:5, 6:2–11, 6:26–9, 7:6–11, 8:14–10:22, 11:14–12:30, Figs. 4a-b; Ex. 1002 ¶¶ 69–72). Patent Owner does not dispute these contentions.

(2) *claim element 2[c2] - “through a narrow band rejection filter”*

Petitioner admits that Barber does not expressly disclose providing pulsed DC power to the target “through a narrow band rejection filter,” as recited in claim element 2[c2] (Pet. 18), but contends that Barber in view of Hirose teaches this claim element (Pet. 17–28 (citing Ex. 1002 ¶¶ 73–100)).

For example, Petitioner states Barber uses two power supplies (DC supply 230 and RF supply 235) that operate at different frequencies and that one skilled in the art would have known the benefits of using filters in plasma systems to block interference/current of one power supply from another in such systems. Pet. 18–19 (citing, *inter alia*, Ex. 1005, 6:29–31, 8:45–48, 9:12; Ex. 1002 ¶¶ 75–77).

Petitioner asserts Hirose discloses, or makes obvious the use of, narrow band rejection filters, such as Hirose’s first filter circuit 20 that is placed between power supply 14 and upper electrode 12 to “selectively filter[] a high-frequency current output from the second high-frequency power supply 15 for preventing the current from reaching the first high-frequency power supply 14.” Pet. 20 (quoting Ex. 1006, 4:9–16); *see also* Pet. 20–21 (stating second filter 21 is likewise used to isolate supply 15 from high-frequency current from supply 14 and citing Ex. 1006, 4:16–21, 5:63–6:4; Ex. 1002 ¶ 79).

Petitioner contends that it would have been obvious to modify Barber’s system to incorporate a narrow band rejection filter, such as that taught by Hirose, to prevent the RF power from RF supply 235 from affecting DC supply 230 and to protect DC power supply 230 and allow it to provide un-interfered pulse DC power having a stable waveform to optimize film deposition. *See, e.g.*, Pet. 23 (citing Ex. 1002 ¶ 83); Ex. 1002 ¶ 75.

Petitioner asserts that a skilled artisan would have understood that “choosing to reject a specific frequency (or a narrow band of frequencies) depending on the bandwidth of [Barber’s RF] supply 235” was a known way to achieve the benefits taught by Hirose. Pet. 24 (citing, *inter alia*, Ex. 1002 ¶ 89; Ex. 1006, Figs. 1, 6). In particular, Petitioner asserts that it would have been obvious to implement a narrow band rejection filter in Barber to minimize the filter’s impact on the pulsed-DC waveform (i.e. not significantly degrade the waveform signal through filtering) while still protecting DC power supply 230 from the specific frequency signals of RF power supply 235. Pet. 25 (citing Ex. 1002 ¶ 89; Ex. 1016, 4:20–27 (“notch filter ‘intended to attenuate the 13.56 MHz signal’)). Petitioner further asserts that a skilled artisan, looking at Barber, would have known the benefits of filters in plasma systems for blocking interference/current of one power supply from another. Pet. 25–27 (citing Ex. 1002 ¶¶ 90–91; Ex. 1014, 3, Fig. 1; Ex. 1016, 3:53–63; Ex. 1017, 16:20–29; Ex. 1018, 24:29–32; Ex. 1019, 6:60–62, 7:40–42; Ex. 1020, 2:61–3:6, 4:26–31; Ex. 1021, 7:66–8:2; Ex. 1010, 6:40–41; Ex. 1001, 5:46–48; Ex. 1025, 23–24; Ex. 1026, 116; Ex. 1062, 134; Ex. 1067, 141; Ex. 1024, 151; Ex. 1065, 2; Ex. 1066, 7). Dr. Subramanian testifies that a person of ordinary skill in the art would have been motivated to modify Barber’s system, *inter alia*, to incorporate a narrow band rejection filter such that the filter prevents the RF signal from source 235 from affecting DC power supply 230 and to configure the filter to ensure that the appropriate frequencies associated with RF power supply 235 are isolated from DC power supply 230. Ex. 1002 ¶ 83.

Patent Owner raises several arguments in response. For example, Patent Owner contends that Barber does not use any RF filter with a DC power supply and does not suggest a need or desirability to include a filter to remove the coupled frequencies. Prelim. Resp. 14–18 (citing Ex. 1005, 2:13–21, 3:1–13, 6:5–6, 6:37–42, 7:14–17, 7:19–23, 7:36–40, 7:47–48, 8:45–63, 8:67–9:3, 9:29–32, Fig. 2; Ex. 1060, 4:26–30, 7:1–5, Figs. 2, 4a, 4b; Ex. 1036, 5, Fig. 7; Ex. 2002 ¶¶ 54, 59). Patent Owner also argues that Hirose’ filter is not coupled to a bipolar pulsed DC power supply to a target, as required by claim element 2[c]. Prelim. Resp. 25–29. Patent Owner contends that Hirose only involves RF power supplies, not DC power supplies, and that Hirose involves reactive ion etching (“RIE”) and that RIE does not even have a target. Prelim. Resp. 26 (citing Ex. 2002 (Glew Declaration) ¶ 72). Patent Owner also contends that neither Barber, nor the other references cited by Petitioner as supposedly using bipolar pulsed DC power supply, suggest using a filter between the pulsed DC power and the target, even when RF bias was applied to the substrate, or that a filter was desirable to achieve the required voltage attenuation. *Id.* (citing Exs. 1005, 1008, 1048, 1059).

Patent Owner’s arguments, however, attack the teachings of each reference individually, whereas Petitioner’s unpatentability argument relies on the combined teachings of these references. *Cf. In re Merck*, 800 F.2d 1091, 1097 (Fed. Cir. 1986) (“Non-obviousness cannot be established by attacking references individually where the rejection is based upon the teachings of a combination of references.”). For example, Patent Owner contends Barber does not depict an RF filter between the DC supply and the target. Prelim. Resp. 15 (citing Ex. 1005, Fig. 2).

However, Petitioner is relying upon the combined teachings of Hirose and Barber to teach providing pulsed DC power to the target through the narrow band rejection filter as recited in claim 2[c]. Similarly, Patent Owner contends Hirose's filter is not coupled to a bipolar pulsed DC power supply to a target as required by claim element 2[c]. Prelim. Resp. 24–26. Again, this argument focuses on the teaching of Hirose alone and not on Petitioner's arguments directed to the combination of Hirose and Barber.

Patent Owner also contends that RF power supplies, like those in Hirose, are configured in a materially different way from a pulsed DC power supply, and that Petitioner has provided no evidence “that the operating mechanism of a reactor process involving a pulsed DC power supply and an RF bias would have been the same as a reactor process with only RF power.” Prelim. Resp. 27–28 (citing Ex. 2002 ¶¶ 10–11, 73–75 as explaining that RF power supplies are configured in a materially different way than DC power supplies and that RF match circuits would not work with a DC power supply). The test for obviousness, however, “is not whether the features of a secondary reference may be bodily incorporated into the structure of the primary reference Rather, the test is what the combine teachings of the references would have suggested to those of ordinary skill in the art.” *In re Keller*, 642 F.2d 413, 425 (CCPA 1981). Petitioner does not propose that Hirose's filter be physically incorporated into the apparatus of Barber. Instead, Petitioner asserts that a skilled artisan would have found it obvious to modify Barber's system to incorporate a narrow band rejection filter in between the power supply and the target electrode to prevent the RF power from

RF supply 235 from affecting DC supply during Barber's process. *See, e.g.,* Pet. 23.

Based upon our review of the present record, we determine that Petitioner has sufficiently shown, at this stage of the proceeding, that the combination of Barber and Hirose teach or suggest the limitations of claim element 2[c]. For example, Petitioner sufficiently shows that Hirose teaches or suggests a narrow band rejection filter (*e.g.,* Pet. 22 (asserting Figure 6 of Hirose suggests that Hirose's filter has a very narrow frequency response) and that a POSITA would have understood that Hirose's filter operates like a narrow-band rejection filter (Pet. 22–23 (citing Ex. 1002 ¶ 81; Ex. 1006, 4:62–5:10; Ex. 1013, 5, Fig. 2–9(G))). Petitioner also sufficiently shows that Hirose teaches use of its filter, which is placed between the local power supply and an electrode of the plasma system, to block electrical current of a remote power supply (*e.g.,* power supply 15) from reaching a local power supply (*e.g.,* power supply 14). *See, e.g.,* Pet. 19–23; Ex. 1002 ¶ 82; *see also* Ex. 1006, 4:13–16 (stating that the “LC series resonant circuit selectively filters a high-frequency current output from the second high-frequency power supply 15 for preventing the current from reaching the first high-frequency power supply 14”). *Id.* at 4:13–16. Petitioner also sufficiently shows that a POSITA would have understood that incorporating a narrow band rejection filter into Barber's system in between Barber's power supply and the target electrode would prevent the RF power from RF supply from affecting DC supply 230 during Barber's process. *See, e.g.,* Pet. 23 (citing Ex. 1002 ¶ 83).

Finally, Patent Owner contends that Petitioner fails to articulate a competent reason why a skilled artisan would have included the claimed

filter in Barber's process, and that Petitioner's obviousness theory is based on hindsight. *See, e.g.*, Prelim. Resp. 48–61. For example, Patent Owner contends that Petitioner has not shown that Barber needs an RF filter to block the RF bias frequency on the substrate, let alone a need for a narrow-band rejection filter. *Id.* at 60. Patent Owner also contends that Petitioner has not provided competent evidence that a skilled artisan would have believed that the selective blocking of the specific frequencies of the substrate RF bias power would have stabilized Barber's DC waveform, or that the filter would have changed the optimal process point or improved the piezoelectric film deposition. Prelim. Resp. 52 (citing Pet. 17–18, 23–28). Additionally, Patent Owner asserts that because Barber expressly states that the “separation rate is unaffected by the power supply frequency” and that the “frequency does not affect the film quality and may be freely adjusted,” a skilled artisan would have understood that the power supply pulse frequency would have no effect on deposition rate or film quality. Prelim. Resp. 53 (quoting Ex. 1002 ¶ 55; Ex. 1005, 7:47–48, 9:2–3).

The Petition and Dr. Subramanian's declaration, however, do articulate reasons why a person skilled in the art would have combined the teachings of Barber and Hirose. For example, Dr. Subramanian asserts that Hirose teaches benefits from blocking electrical current of a remote power supply in plasma processes systems, which would have been beneficial in the sputtering system of Barber. *See e.g.*, Ex. 1002 ¶¶ 52, 85 (citing, *inter alia*, Ex. 1006, code (57); *see also* Pet. 18–19 (citing Ex. 1002 ¶¶ 75–77). Dr. Subramanian further testifies that Barber himself states a desire for a stable waveform (Ex. 1002 ¶ 74 (citing

Ex. 1005, 7:59–60, 8:66–9:2)) and that the electrical characteristics of the pulsed DC power supply are important parameters to stabilize in order to obtain high quality films (Ex. 1002 ¶ 74 (citing Ex. 1005, 7:36–40, 7:14–17, 7:41–47, 7:60–67). Dr. Subramanian further testifies that a POSITA would have looked to other apparatuses using power supplies in similar systems, such as Hirose, which also uses two power supplies of different frequencies in a plasma processing apparatus (Ex. 1002 ¶ 77) and would have been motivated to modify Barber’s system to incorporate a narrow band filter to prevent the RF signal from source 235 from affecting DC power supply 230 during the process and to ensure the appropriate frequencies associated with RF power supply 235 are isolated from DC power supply 230 (Ex. 1002 ¶ 83). Dr. Subramanian further testifies that a POSITA would have understood that using a narrow band rejection filter in Barber’s system would have helped Barber’s DC power supply provide a stable waveform to the target so as to optimize film depositions by reducing or eliminating electrical interference from the RF power supply. Ex. 1002 ¶ 86.

Additionally, relying on the deposition testimony of Dr. Subramanian, Petitioner provides evidence and argument that a skilled artisan, looking at Barber, would have known the benefits of filters in plasma systems for blocking interference/current of one power supply from another. Pet. 25–27 (citing Ex. 1002 ¶¶ 90–91; Ex. 1014, 3, Fig. 1; Ex. 1016, 3:53–63; Ex. 1017, 16:20–29; Ex. 1018, 24:29–32; Ex. 1019, 6:60–62, 7:40–42; Ex. 1020, 2:61–3:6, 4:26–31; Ex. 1021, 7:66–8:2; Ex. 1010, 6:40–41; Ex. 1001, 5:46–48; Ex. 1025, 23–24; Ex. 1026, 116; Ex. 1062, 134; Ex. 1067, 141; Ex. 1024, 151; Ex. 1065, 2; Ex. 1066, 7).

Whether such disclosures, and the additional arguments and evidence provided in the Petition, would have provided one of ordinary skill in the art with the requisite reason to modify the asserted art, and how much weight to accord Dr. Subramanian's testimony, are issues that are best resolved upon a full record.

For these reasons, Petitioner has sufficiently shown that the combination of Barber and Hirose teaches or suggests the claimed narrow band rejection filter of claim element 2[c], and that one of ordinary skill in the art would have had a reason to modify Barber in view of Hirose to use the claimed filter in Barber's system.

d) Claim element 2[d] - Providing an RF Bias Corresponding to the Narrow Band Rejection Filter to the Substrate

Claim element 2[d] recites "providing an RF bias that corresponds to the narrow band rejection filter to the substrate." Ex. 1001, 23:22–23. Petitioner contends Barber in view of Hirose teaches the subject matter of claim element 2[d]. Pet. 28 (citing Ex. 1002 ¶101; Pet. 12–28). Petitioner asserts that Barber discloses an RF power supply 235 that applies a bias voltage to a substrate platen 115 and that "bias is applied to substrate to control [deposited film's] tensile strength." Pet. 28 (citing Ex. 1005, 6:29–31, 9:12). Petitioner also asserts that Hirose's plasma processing apparatus has power supplies 14 and 15, connected to respective filters 20 and 21, and that the "filter's optimum resonance point can be modified, e.g., to adjust plasma process conditions." Pet. 19 (citing Ex. 1006, code (57), 3:45–4:38; Ex. 1002 ¶¶ 77–78). Dr. Subramanian testifies that a person of ordinary skill in the art would have configured Barber's system such that RF power supply 235 would provide an RF bias to the substrate, where the RF bias that corresponds to the narrow band rejection filter to protect DC power supply