of 57 ohms/sq. The roughness illustrated in Figure 9C can be characterized by an Ra=0.88 nm and a Rmax of 19.8 nm.

[0052] Figure 9D was deposited using 1.5 kW RF power, 300 W bias, 0 sccm O₂, 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 Ra=0.45 nm and an Rmax 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.

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Slot #	_	Target Vo	ltage (V)	Target Current (Amps)		
	Process	Min	Max	Mix	Max	
14	1.5kw/100w/200khz/2.2µs/300s/20Ar/80O2	244	252	5.94	6.14	
15	1.5kw/100w/200khz/2.2µs/300s/20Ar/40O2	254	263	5.7	5.9	
17	1.5kw/100w/200khz/2.2µs/300s/20Ar/40O2	252	260	5.76	5.96	
19	1.5kw/100w/200khz/2.2µs/300s/20Ar/36O2	254	263	5.72	5.92	
21	1.5kw/100w/200khz/2.2µs/300s/20Ar/30O2	255	268	5.76	5.9	
1	1kw/100w/200khz/2.2µs/300s/20Ar/ 80O2	224	233	4.32	4,5	
2	1kw/100w/200khz/2.2µs/300s/20Ar/ 36O2	231	243	4.12	4.3	
3	1kw/100w/200khz/2.2µs/300s/20Ar/ 32O2	232	242	4.12	4.28	
4	1kw/100w/200khz/2.2µs/300s/20Ar/ 28O2	237	243	4.1	4.22	
5	1kw/100w/200khz/2.2µs/300s/20Ar/ 24O2	233	243	4.1	4.34	
6	1kw/100w/200khz/2.2µs/300s/20Ar/ 28O2	231	245	4.12	4.3	

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· Table I

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

Table II

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Table III

1		Target			. 1					Thic		DepRa		·
	Run	Power	<u>Bias/</u>			<u>T</u>	<u>Rs</u>	Rs (non-	Bulk Rho	<u>kness</u>		te	Target	
<u>Trial</u>	(sec)	(kW)	W	<u>02</u>	Ar	<u>(oC)</u>	(Ohms/Sq)	unif)	(uOhmcm)	(Å)	n	(A/sec)	\underline{N}	Target/I
14	100	3	300	3	60	280	38.69	4.07%	4.64E-04	1200	1.864	12		
1									х.				288-	9.86-
16	100	3	300	3	30	280	56.90	7.94%	6.98E-04	1227	1.888	12.27	308	10.42
													265-	10.92-
10	100	3	100	3	60	280	50.98	11.89%	6.25E-04	1225	1.933	12.25	275	11.36
1										543.			238-	
4	100	1.5	100	3	30	280	383.62	21.72%	2.09E-03	9	2.016	5.439	251	5.98-6.32
										483.			239-	
8	100	1.5	300	3	30	280	504.02	7.23%	2.44E-03	5	2.082	4.835	250	5.98-6.33
										520.			225-	-
2	100	1.5	100	3	30	280	402.52	26.80%	2.10E-03	7	2.056	5.207	239	6.46-6.68
										580.			237-	
6	100	1.5	300	0	30	280	106.21	6.12%	6.17E-04	5	1.945	5.805	250	5.98-6.38
													285-	9.98-
12	100	3	100	4	30	280	374.34	19.43%	4.18E-03	1116	1.917	11.16	300	10.52
													282-	10.00-
15	100	3	300	4	30	100	6264.69	58.18%	6.81E-02	1087	1.897	10.87	304	10.62
										392.			237-	
7	100	1.5	200	4	30	100	7509.45	44.14%	2.95E-02	3	2.149	3.923	250	6.02-632
	•									389.		. •	238-	
1	100	1.5	100	4	30	100	12284.82	112.55%	4.78E-02	1	2.236	3.891	250	6.04-632

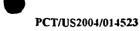
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Table III (Cont.)

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
								12.1070		1100	1.750		288-	9.78-
9	_100	3	100	0	30	100	43.78	7.47%	5.55E-04	1268	1.945	12.68	307	10.42
5	100	1.5	200	2	60	100	1202 52	14 020/	6 997 03	454.9	2.140	4 5 4 9	225-	()(())
	100	<u> </u>	200	3	60	100	1293.53	14.82%	5.88E-03	454.8	2.149	4.548	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
													264-	10.96-
13	100	3	200	0	60	100	49.05	7.24%	6.16E-04	1256	1.913	12.56	275	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

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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 indiuim-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_a less than about 10 nm with Rms 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.

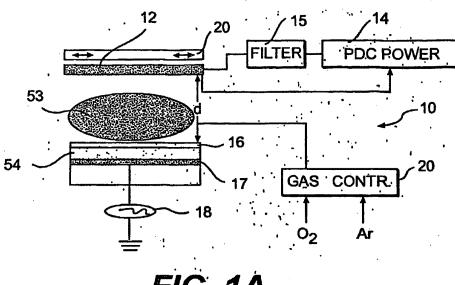
24. The method of claim 18, wherein the target can include at least one rare-earth ions.

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 cerbium.

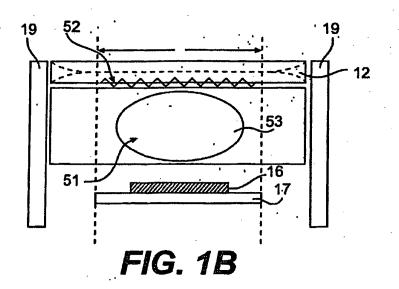
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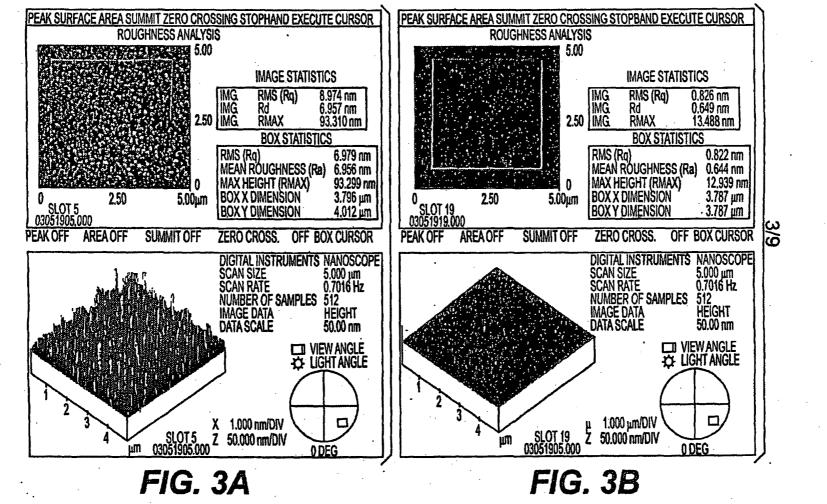
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52 52 FIG. 2

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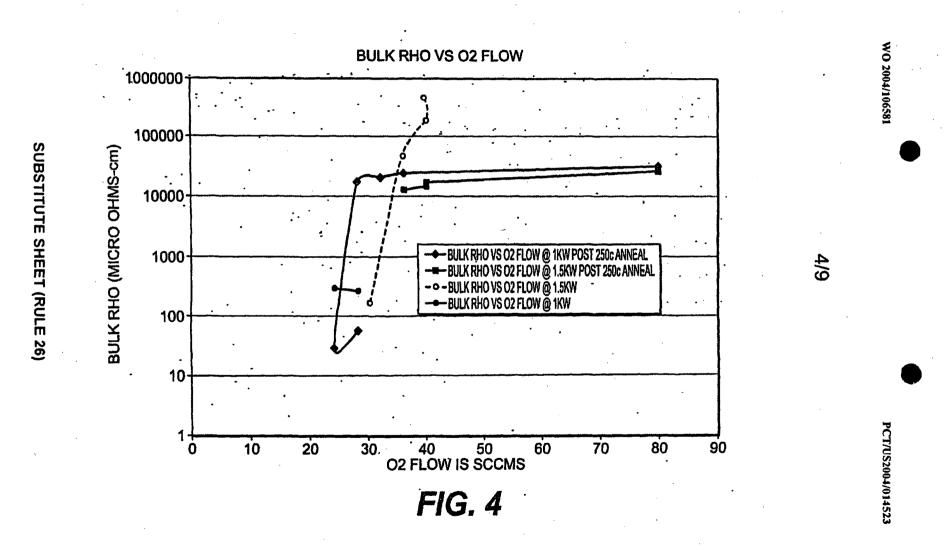


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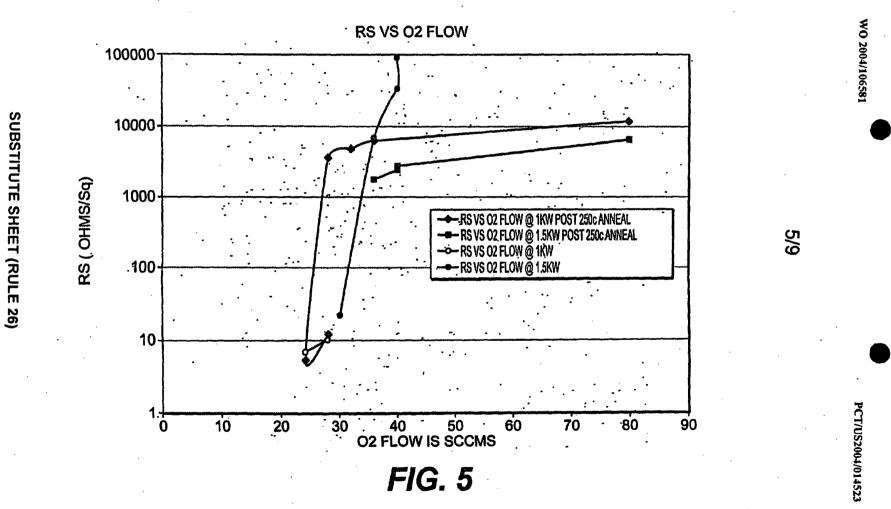
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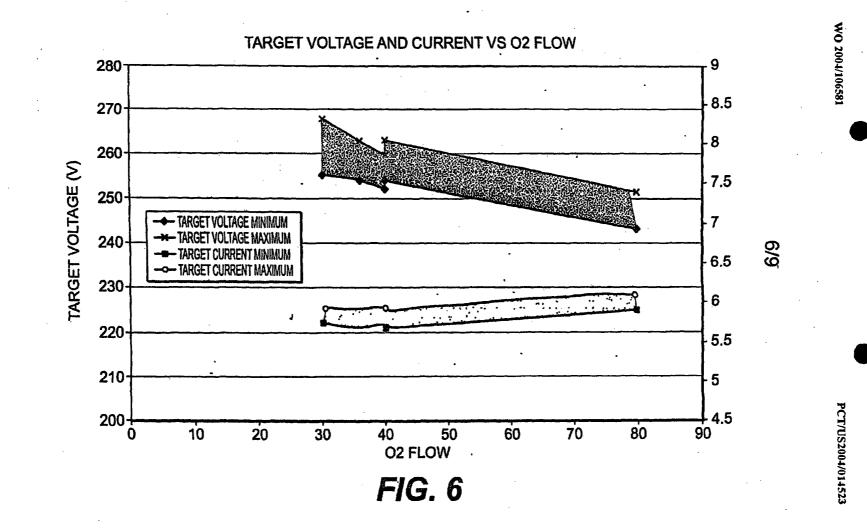


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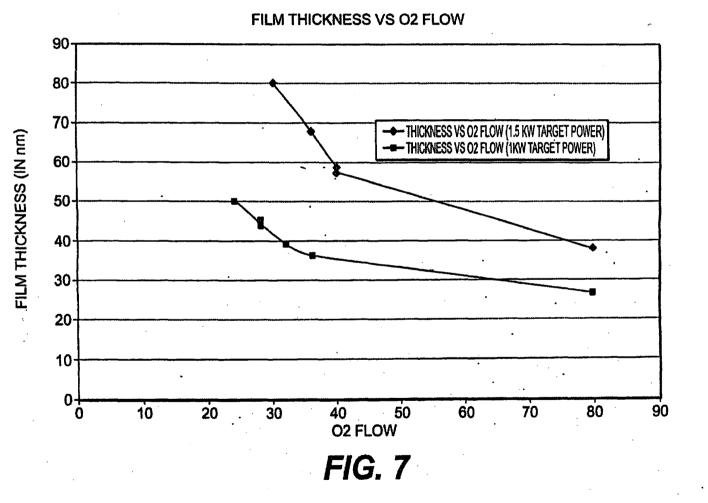
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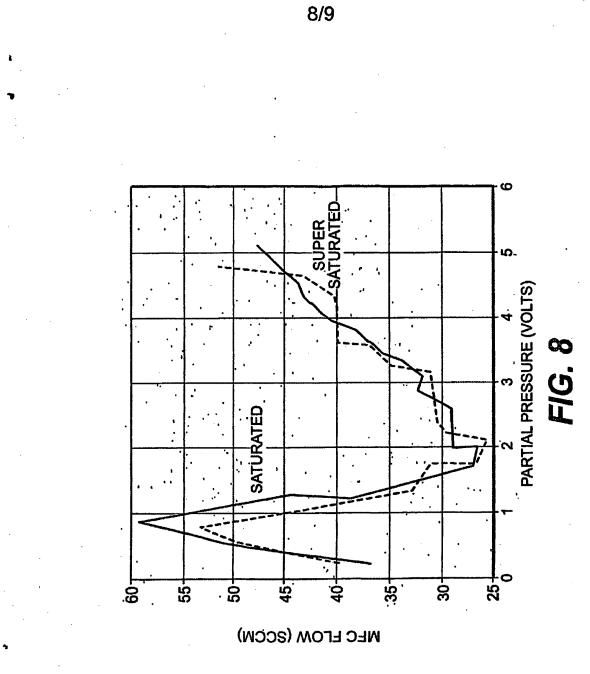


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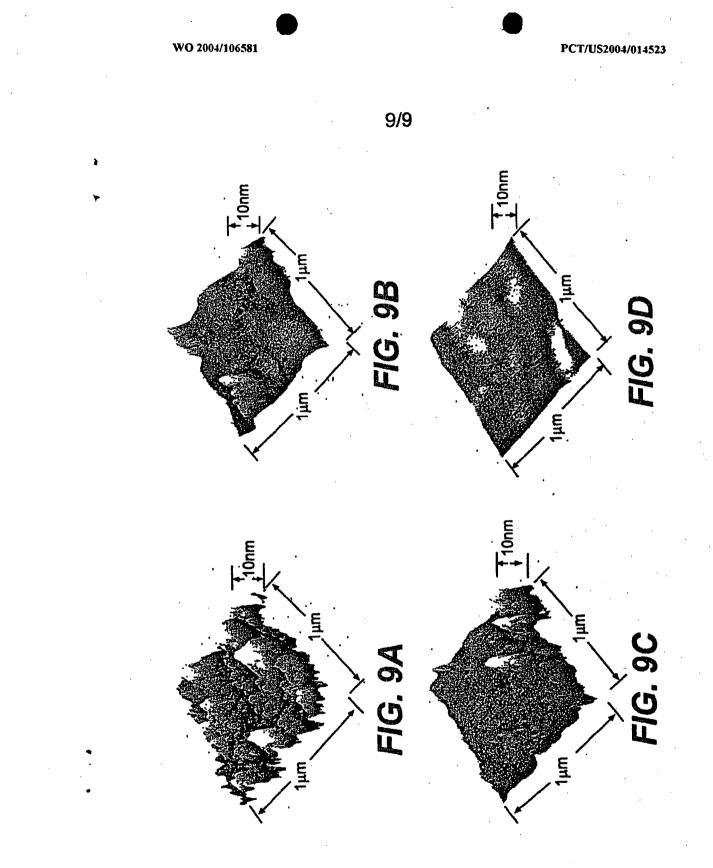
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54) Title: ENERGY CONVERSION AND STORAGE FILMS AND DEVICES BY PHYSICAL VAPOR DEPOSITION OF TI-TANIUM AND TITANIUM OXIDES AND SUB-OXIDES

(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 TiO_2 . In some embodiments, the layer is a sub-oxide of Titanium. In some embodiments, the layer is TiO_2 . In some embodiments, the layer is a sub-oxide of Titanium. 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.

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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 recrystalization 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-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, TiO₂ 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 TiO₂ 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 suboxide 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 deposited on a light-emitting device. In some embodiments of the invention, the invention, the an optically active layer deposited on a light-emitting device. In some embodiments of the invention, the 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 TiOy 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 EbonexTM and an SEM cross section of the $Ti_4O_{6.8}$ film deposited from the EbonexTM 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 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.

[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

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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 TiO₂, 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 Ti₄O₇ between a conducting layer of titanium, which is substrate 101, and the dielectric TiO₂ 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 increases 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 it's 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.

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[0052] TiO_2 layers, for example, can photocatylitically 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 photalysis 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, posses unusual levels of durability to mineral acid solutions and other corrosive gassious or liquid environments. Hayfield, P.C.S., "Development of a New Material- Monolithic Ti₄O₇ 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 stoichometry TiO. Of the several compositions, Ti_4O_7 in particular is known to posses both useful conductivity and also chemical resistance to both anodization, which would decrease it's conductivity, as well as reduction, which would decrease it's 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 stoicheometry 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 TiO₂ is known to display useful levels of

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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 erebium-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 TiO₂ dielectric protective layer is deposited over the protective conducting layer 102. Another protective conducting layer 102 can be deposited over the TiO₂ 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, Ti_4O_7 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 EbonexTM which compounded of bulk Ti_4O_7 was bonded onto a backing plate. EbonexTM 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 $1.68\mu m$. 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, TiO_2 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 $600x720mm^2$. The target was a ~7mm thick, ~ $630x750 mm^2$ 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 $2x10^{-7}$ 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 O_2 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 Ti_4O_7 layer, and layer 104 is TiO_2 . 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 TiO_2 film thickness decreases, so does the dielectric constant of the TiO_2 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 TiO_2 layers for layer 104 with $Ti-Ti_4O_7$ deposited as layer 102 of Figure 3B.

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

[0072] A layer of TiO_2 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, TiO_2 was deposited in the same chamber without oxide burn in. This process resulted in a $Ti-TiO_y -TiO_2$ (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 Ti_4O_7 EbonexTM target (Figure 9A) and an SEM cross section of the $Ti_4O_{6.8}$ layer (Figure 9B) deposited from the EbonexTM target according to the present invention. The deposited film shows smooth deposition of the layer. The EbonexTM target shown in Figure 9A shows an open porousity 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.

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



[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 TiO_2 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 TiO_2 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 crystalling 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.

Film	k	V _{bd}	FM	С	Breakdown	Film
Thickness		(MV/cm)		(pF/mm2)	Voltage	Morphology
(nm)					(V)	
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

Table II

[0080] Therefore, it is clear that amorphous TiO_2 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 TiO_2 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 TiO_2 amorphous films is to apply active cooling to the substrate in an amount sufficient to provide continuously amorphous TiO_2 films.

[0081] 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. 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 O_2 , 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, discreet 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 a 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 $1.53 \mu m$, 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.

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

Table III

[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 nonradiative. In radiative relaxation, the excited ion emits light. In non-radiative relaxation, the excited ion undergoes a cooperative process with vibrational modes of it's 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 TiO_2 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 CaF_2 or other organic material, layer 102 is

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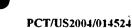
the rare-earth doped TiO_2 layer, and layer 104 is absent, then upon lift-off or upon transfer of layer 102, a free standing or applied layer having electroluminescent or photoluminescent applications can be provided over a selected device.

[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.

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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 TiO_2 .

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 rareearth 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 rareearth 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;

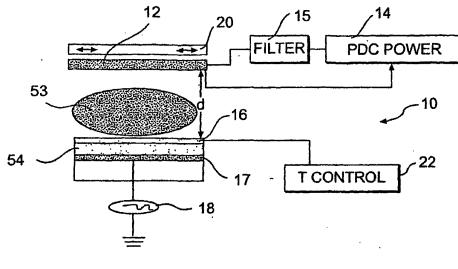
mixing the TiO powder to form a mix;

hipping the mix under a controlled atmosphere to form tiles; and forming a target from the tiles.

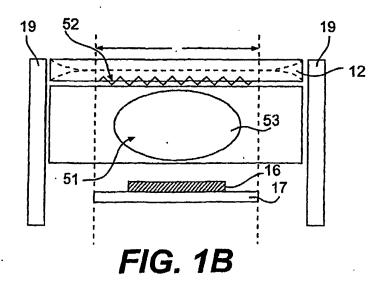
86. The method of claim 85, further including mixing at least one rare-earth oxide powder with the mix.

87. The method of claim 86, wherein the at least one rare-earth oxide includes erbium oxide.





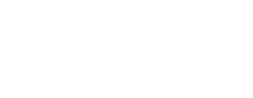


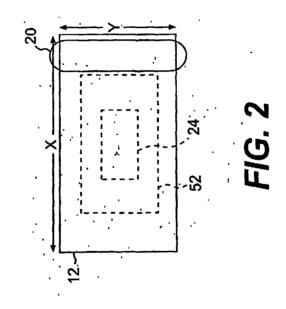


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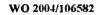
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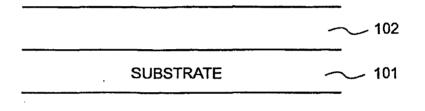


FIG. 3A

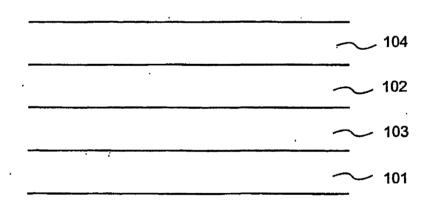


FIG. 3B

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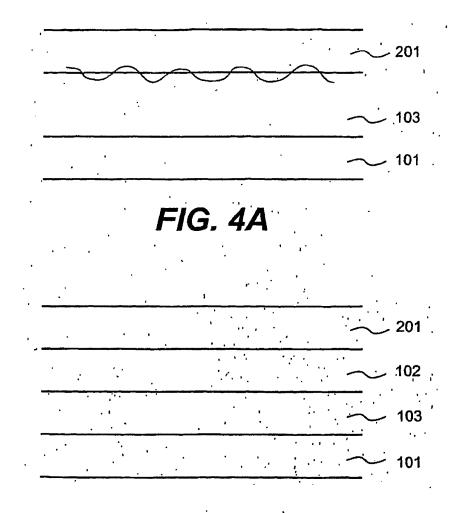


FIG. 4B

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<u>103</u>
 102
201
<u>102</u>
<u>103</u>
101

FIG. 5

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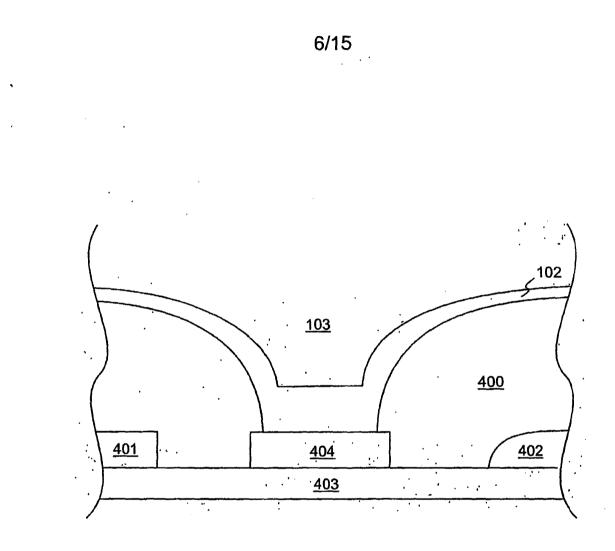
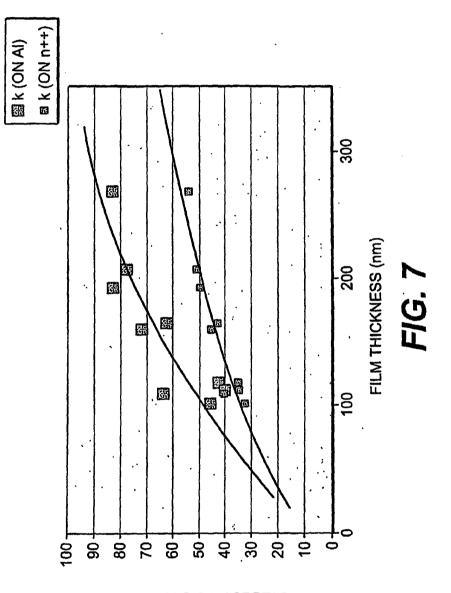


FIG. 6

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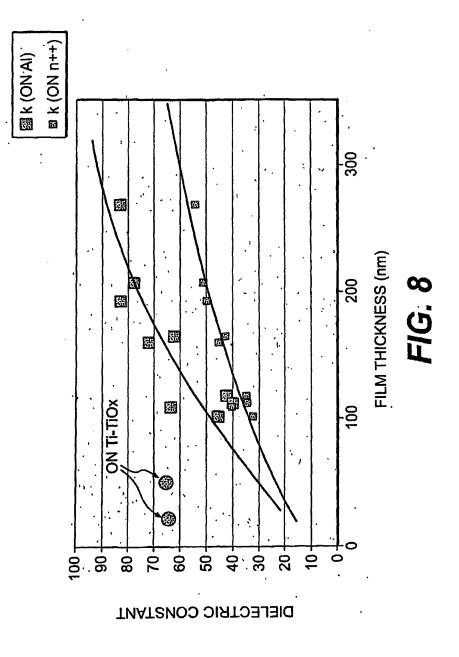


DIELECTRIC CONSTANT

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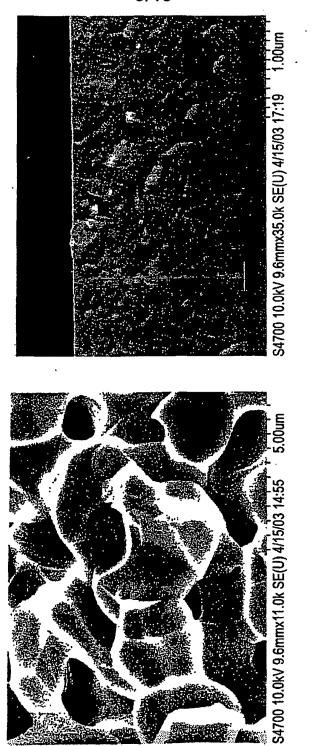
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FIG. 9B

FIG. 9A

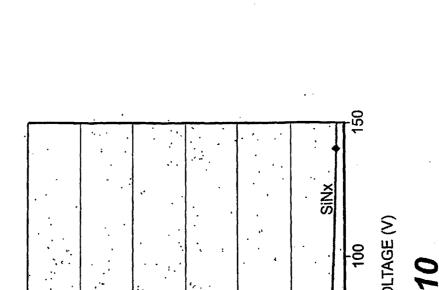
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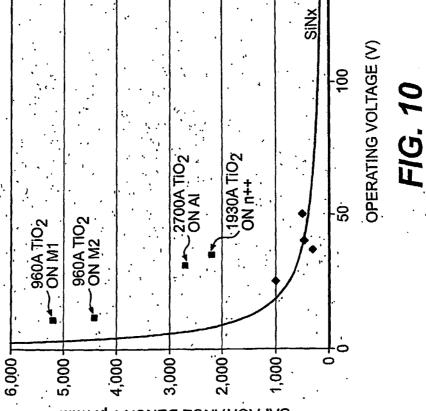


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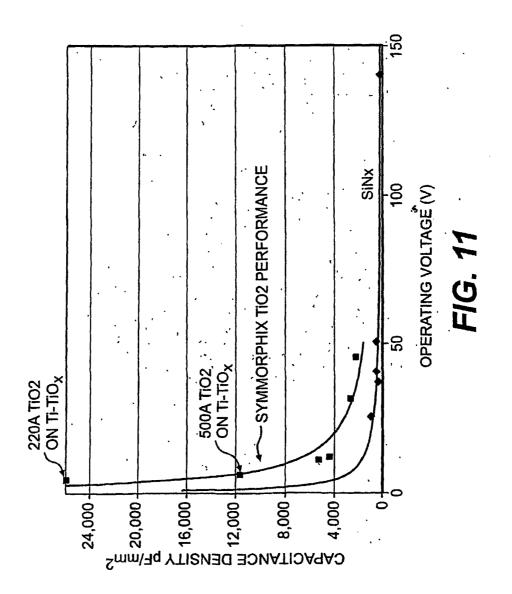




CAPACITANCE DENSITY PF/mm²

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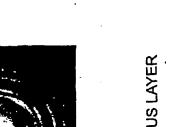




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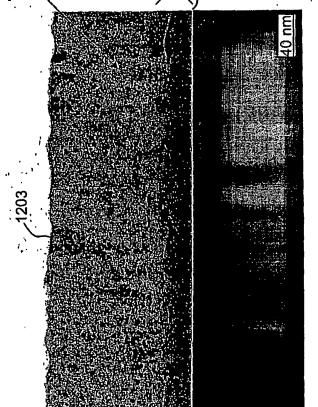
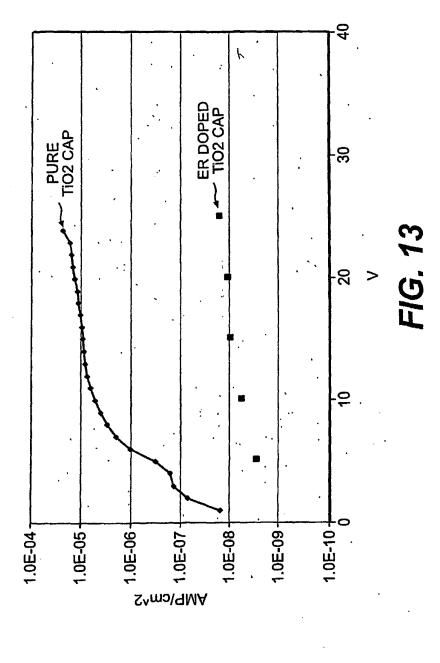


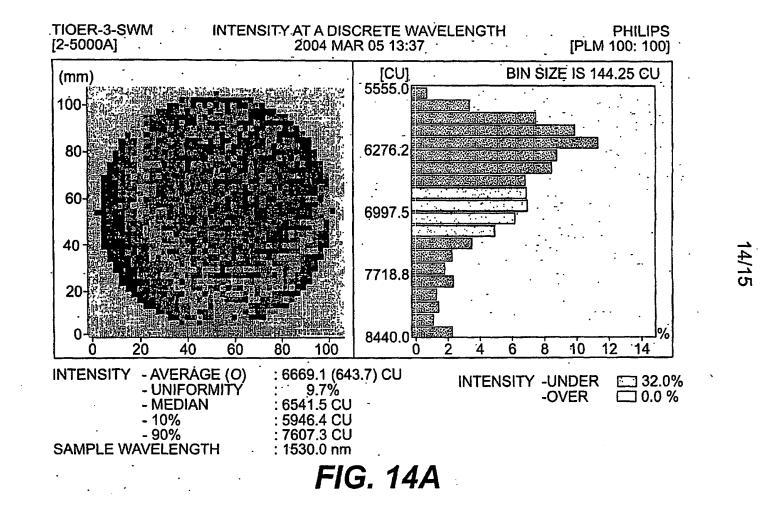
FIG. 12

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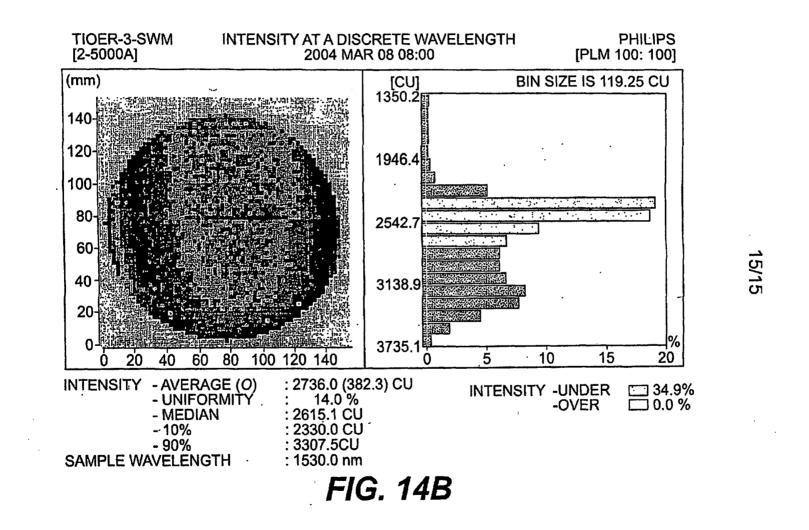


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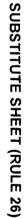
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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/954,182	10/01/2004	Hongmei Zhang	09140-0016-01000	9873
22852 75	90 03/09/2006		EXAM	INER
FINNEGAN, I LLP	HENDERSON, FAF	ABOW, GARRETT & DUNNER	ESTRADA,	MICHELLE
	K AVENUE, NW		ART UNIT	PAPER NUMBER
	N, DC 20001-4413		2823	
			DATE MAILED: 03/09/200	6

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

PTO-90C (Rev. 10/03)

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	Application No.	Applicant(s)
	10/954,182	ZHANG ET AL.
Office Action Summary	Examiner	Art Unit
	Michelle Estrada	2823
The MAILING DATE of this communication a Period for Reply	appears on the cover sheet w	vith the correspondence address
A SHORTENED STATUTORY PERIOD FOR REF WHICHEVER IS LONGER, FROM THE MAILING - Extensions of time may be available under the provisions of 37 CFR after SIX (6) MONTHS from the mailing date of this communication. - If NO period for reply is specified above, the maximum statutory peri - Failure to reply within the set or extended period for reply will, by star Any reply received by the Office later than three months after the ma earned patent term adjustment. See 37 CFR 1.704(b).	DATE OF THIS COMMUNI 1.136(a). In no event, however, may a dod will apply and will expire SIX (6) MOI tute, cause the application to become A	CATION. reply be timely filed NTHS from the mailing date of this communication. BANDONED (35 U.S.C. § 133).
Status		
1) Responsive to communication(s) filed on <u>21</u>	December 2005.	
	his action is non-final.	
3) Since this application is in condition for allow	wance except for formal mat	ters, prosecution as to the merits is
closed in accordance with the practice unde	er Ex parte Quayle, 1935 C.[D. 11, 453 O.G. 213.
Disposition of Claims		
4) Claim(s) <u>41-43,45-62,85 and 87-89</u> is/are pe	ending in the application.	
4a) Of the above claim(s) is/are withd		
5)⊠ Claim(s) <u>61,62 and 85</u> is/are allowed.		
6) Claim(s) <u>41-43 and 45-60</u> is/are rejected.		
7)⊠ Claim(s) <u>87-89</u> is/are objected to.		
8) Claim(s) are subject to restriction and	d/or election requirement.	
Application Papers		
9) The specification is objected to by the Exami	iner	
10) The drawing(s) filed on <u>21 December 2005</u> is		objected to by the Examiner
Applicant may not request that any objection to the		
Replacement drawing sheet(s) including the com		
11) The oath or declaration is objected to by the		
Priority under 35 U.S.C. § 119		
 12) Acknowledgment is made of a claim for forei a) All b) Some * c) None of: 	gn priority under 35 U.S.C.	§ 119(a)-(d) or (f).
1. Certified copies of the priority docume	ents have been received.	
2. Certified copies of the priority docume		Application No.
3. Copies of the certified copies of the pr		
application from the International Bure	-	
* See the attached detailed Office action for a li		received.
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1) Notice of References Cited (PTO-892)	4) Interview S	Summary (PTO-413) s)/Mail Date
2) D Notice of Draftsperson's Patent Drawing Review (PTO-948)		nformal Patent Application (PTO-152)
3) X Information Disclosure Statement(s) (PTO-1449 or PTO/SB/0		
B) Information Disclosure Statement(s) (PTO-1449 or PTO/SB/0 Paper No(s)/Mail Date <u>7/25/05,12/21/05</u> .	6) 🗌 Other:	

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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 negatived 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).

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.

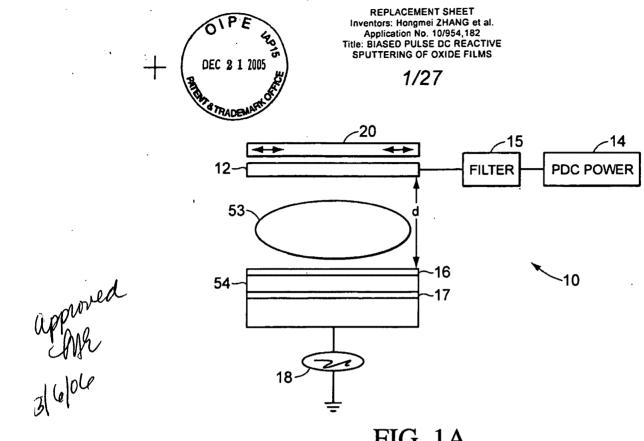
Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 571-272-2800.

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).

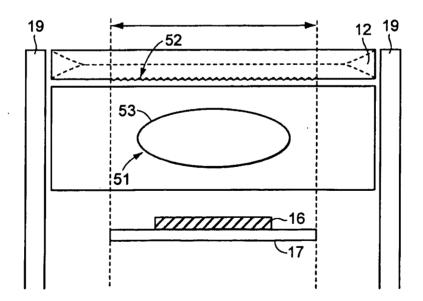
Michelle Estrada Primary Examiner Art Unit 2823

ME March 6, 2006

Page 628 of 1053



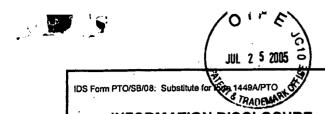






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INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Use as many sheets as necessary)

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of

EXPRESS MAIL NO. EV 727732357 US

C	omplete 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 A	AND PUBLISHE	D U.S. PATENT APPLICAT	IONS
	Examiner	Cite	Document Number	Issue or	Name of Patentee or	Pages, Columns, Lines, Where
	Initials	No.1	Number-Kind Code ² (it known)	Publication Date MM-DD-YYYY	Applicant of Cited Document	Relevant Passages or Relevant Figures Appear
	age		US 2003/0141186 A1	07-31- 2003	Wang et al.	
-	Mar.		US 2001/041460 A1	11-15-2001	Wiggins	
	108		US 2001/0027159 A1	10-04-2001	Kaneyoshi	
	hh		US 6,232,242	05-15-2001	Hata et al.	
n bar			US 6,117,279	09-12-2000	Smolanoff et al.	
N 84 L	ANG		US 5,738,731 (09-00)	04-04-1998	Shindo	
iled 105	ME		US 5,538,796	07-23-1996	Schaffer	
1-	hok		US 5,309,302	03-14-1961	Hoit-Vollmann	
	ant.		US 5,296,089	03-22-1994	Chen et al.	
	MR		US 5,173,271	12-22-1992	Chen et al.	
•	brok		US 4,587,225	05-06-1986	Tsukuma et al.	
	AR		RE 32,449	06-30-1987	Claussen	

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

			FOREI	GN PATENT	DOCUMENTS		
	Examiner Initials	Cite No. ¹	Foreign Patent Document Country Code ³ Number ⁴ Kind Code ⁶ (<i>It known</i>)	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
translatio			EP 1 092 689 A1	-04-18-2001	BPS Alzenau GmbH		
fransia	ph		EP 1068899 A1	01-17-2001	Nippon Sheet Glass Co., Ltd.		
	Ar	1	EP 0 639 655 A1	02-22-1995	Asahi Glass Co, Ltd.		
(MP		EP 0 652 308 A2	10-13-1994	Mega Chips Corp.		
	ple		JP 7-233469	09-05-1995	Asahi Glass Co, Ltd.		
C	BR		WO 00/21898 A1	04/20/2000	Samsung Electronics Co.		
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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.

Date Considered

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Examiner

Signature

EXPRESS MAIL NO. EV 727732357 US

IDS Form PTO/S	B/08: Substitute for for	m 1449A/PTO		Complete if Known		
				Application Number	10/954,182	
INFO	ORMATION D	ISCLOSU	RE	Filing Date	October 1, 2004	
	TEMENT BY			First Named Inventor	ZHANG, Hongmei	
514		AFFLICA		Art Unit	2823	
	(Use as many sheets	as necessary)		Examiner Name	ESTRADA, Michelle	
Sheet	2	of	2	Attorney Docket Number	9140.0016-01	

Examiner Initials	Cite No.1	NON PATENT LITERATURE DOCUMENTS 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),	Translation
		publisher, city and/or country where published.	
		Crowder, et al., "Low-Temperature Single-Crystal Si TFT's Fabricated on Si Films Processed via Seguential Lateral Solidification," IEEE, vol. 19, no. 8 (August 1998),	
MC		pages 306-308.	
BR		Greene et al., "Morphological and electrical properties of rf sputtered Y203-doped Zr02 thin films," J. Vac. Sci. Tecnol., vol. 13, no. 1 (Jan/Feb 1976), pages 72-75.	
Parti		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).	
MR.		Im, et al. "Controlled Super-lateral Growth of Si Films for Microstructural Manipulation and Optimization," Materials Science Program (1998), pages. 603-617.	
ARE		Im, et al., "Crystalline Si Films for Integrated Active-Matrix LiquidCrystal Displays," MrS Bulletin (March 1996), pages. 39-48.	
APE.		Im, et al., "Single-crystal Si films for thin-film transistor devices," American Institute of Physics (1997), pages. 3434-3436.	
PND		Tukamoto, H. et al., "Electronic Conductivity of LiCoO ₈ and Its Enhancement by	
yr		Magnesium Doping," J. Electrochem. Soc., vol. 44, no. 9, pages 3164-3168 (September 1997).	
PM .		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).	
file		Office Action issued on March 17, 2005 in U.S. Serial No. 09/903,081 (Attorney Docket No. 09140-0014-00).	
ally .		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).	
NG		Office Action dated January 13, 2005, received in Application No. 10/101,863 (Attorney Docket No. 09140.0016-00).	
bh		Response to office Action filed on June 10, 2005 in U.S. Serial No. 10/101,863 (Attorney Docket No. 09140.0016-00).	
fife		Office Action issued on March 14, 2005 in U.S. Serial No. 10/789,953 (Attorney Docket No. 09140-0030-00).	
RAR		Office Action issued March 24, 2005 in U.S. Application No. 10/851,542 (Attorney Docket No. 09140.0033-00).	

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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Examiner Signature

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	IDS Form PTO/S	B/08: Substitute for for	m 1449A/FTO	EC 2 1 2005	ж) С	omplete if Known
			(B)		Application Number	10/954,182
	INFO	ORMATION D	NSCLOS	BRE	Filing Date	October 1, 2004
	STA	ORMATION E		STRACENTE	First Named Inventor	Hongmei ZHANG
	517		AFFLICA		Art Unit	2823
		(Use as many sheets	as necessary)		Examiner Name	ESTRADA, Michelle
	Sheet	1	of	1	Attorney Docket Number	9140.0016-01

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS								
Examiner	Cite	Document Number	Issue or	Name of Patentee or	Pages, Columns, Lines, Where			
Initials ⁴	No."	Number-Kind Code ² (if known)	Publication Date MM-DD-YYYY	Applicant of Cited Document	Relevant Passages or Relevant Figures Appear			
-MU		US 2002/0076133 A1	06-20-2002	Li et al.				
INFU		US 5,478,456	12-26-1995	Humpal et al.				
ANKO	,	US 6,846,765 B2	01-25-2005	Imamura et al.	<u></u>			

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

	FOREIGN PATENT DOCUMENTS								
Examiner Initials	Cite No.'	Foreign Patent Document Country Code ³ Number ⁴ Kind Code ⁵ (<i>ij known</i>)	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			

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
RAR		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, 1 2 pages (2004).	
Alk		HOWSON, R.P., "The reactive sputtering of oxides and nitrides," Pure & Appl. Chem. 66(6):1311-1318 (1994).	
RR		Office Action issued September 21, 2005 in U.S. Application No. 11/100,856 (Attorney) Docket No. 09140.0015-01).	
PAR		Office Action issued on August 4, 2005, in U.S. Application No. 10/101,863 (Attorney Docket, No. 09140.0016-00).	
Ave		Office Action issued on August 8, 2005 in U.S. Application No. 10/101,341 (Attorney Docket , No. 09140-0017-00).	
file		Office Action issued on October 3, 2005 in U.S. Application No. 10/650,461 (Attorney Docket No. 09140-0025-00).	
ANA		Office Action issued on March 24, 2005 in U.S. Application No. 10/851,542 (Attorney Docket, No. 09140-0033-00).	
PAR		Response to Office Action filed July 25, 2005 in U.S. Application No. 10/851,542 (Attorney J Docket No. 09140-0033-00).	
HIG .		Office Action issued on October 19, 2005 in U.S. Application No. 10/851,542 (Attorney , Docket No. 09140.0033-00).	

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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.

· · ·	Application/Control No.	Applicant(s)/Patent Under	
Notice of References Cited	10/954,182	Reexamination ZHANG ET AL.	
Motice of References Offer	Examiner	Art Unit	
	Michelle Estrada	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-			
	С	US-			
	D	US-			
	Е	US-			
	F	US-			
	G	US-			
	Н	US-			
	Ι	US-			
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	L	US-			
	М	US-			

FOREIGN PATENT DOCUMENTS

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NON-PATENT DOCUMENTS

*		Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)
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	x	s reference is not being furnished with this Office action (See MDED § 707.05(c))

*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.

U.S. Patent and Trademark Office PTO-892 (Rev. 01-2001)

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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. (19872
For: BIASED PULSE DC REACTIVE SPUTTERING OF OXIDE FILMS) Confirmation No.: 9873)

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

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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:

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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 <u>43</u>, 59, 60, or 85, wherein the target is a metallic target and the process gas includes one or more of a set consisting of N_2 , NH_3 , CO, NO, CO₂, halide containing gasses.

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

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 <u>43</u>, 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 <u>43</u>, 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.

-3-

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

-4-

metallic target includes:

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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 <u>43</u>, 59, 60, or 85, furthercomprising: 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 thetarget; and

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

Claim 88 (Currently amended): The method according to claim 87, wherein the narrowband 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 biashas 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 ("<u>Smolonoff et al</u>.") in view of U.S. Patent No. 6,673,716 ("<u>D</u>'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.

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Claims 41, 42, 46, 48, and 50

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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.").

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 crossreference 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

-7-

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.

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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

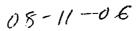
Gary J. Edwards Reg. No. 41,008

Date: August 9, 2006

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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 A	pplication of:)	
ZHAN	IG, Hongmei et al.)	Group Art Unit: 2823
Applic	ation 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 MBCANCO 00000010 060916 10954182 450.00 DA Rν J. E⁄dwards

Reg. No. 41,008

Date: August 9, 2006

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 App	lication of:)	
ZHANG,	Hongmei et al.)	Group Art Unit: 2823
Applicatio	on No.: 10/954,182)	Examiner: ESTRADA, Michelle
Filed: O	ctober 1, 2004)	Q () N 0072
	ASED PULSE DC REACTIVE)))	Confirmation No.: 9873

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 aftached.⁶⁶ CB₁AC9 of the listed non-patent literature documents are aftached.⁶⁶ CB₁AC9 of the listed non-patent 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: Jaw J. August

Reg. No. 41,008

Dated: August 9, 2006

Express Mail Label No. EV 901562545 US

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				Application Number	10/954,182	80
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT				First Named Inventor	ZHANG, Hongmei	AUG 0 9 2006
314		AFFLICA		Art Unit	2823	S
	(Use as many sheets	as necessary)		Examiner Name	ESTRADA, Michelle	.5
Sheet	1	of	3	Attorney Docket Number	9140.0016-01	ADENAN

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Examiner Initials"	Cite No. ¹	Document Number Number-Kind Code ² (if known)	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			
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Examiner	Date	
Signature	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.

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				Application Number	10/954,182	
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STATEMENT BY APPLICANT				First Named Inventor	ZHANG, Hongmei	
(Use as many sheets as necessary)			ALM I	Art Unit	2823	
				Examiner Name	ESTRADA, Michelle	
Sheet	2	of	3	Attorney Docket Number	9140.0016-01	

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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 Country Code ³ Number ⁴ Kind Code ⁵ (if known)	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		
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IDS Form PTO/SB/08: Substitute for form 1449A/PTO				Complete if Known	
				Application Number	10/954,182
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STATEMENT BY APPLICANT				First Named Inventor	ZHANG, Hongmei
	STATEMENT DI AFFLICANT			Art Unit	2823
(Use as many sheets as necessary)				Examiner Name	ESTRADA, Michelle
Sheet	3	of	3	Attorney Docket Number	9140.0016-01

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Examiner	Date	
Signature	Considered	

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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

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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 defurmation) 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

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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

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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 $\leq 88 \ \mu m$. 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.

INVENTOR: BERCHTOLD, L; ENGLISCH, U ; HAUSSELT, J ; KASTER, W ; SCHITTNY, S

PATENT-ASSIGNEE: DEGUSSA AG[DEGS]

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

PATENT-FAMILY:

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

APPLICATION-DATA:

PUB-NOAPPL-DESCRIPTORAPPL-NOAPPL-DATEDE 3738738C N/A1987DE-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., ، خبری

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 02 content.

CHOSEN- Dwg.0/0

DRAWING:

TITLE-HIGH PURE DENSE TARGET CATHODE SPUTTER PRODUCE RARE EARTHTERMS: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

7/21/2006, EAST Version: 2.0.3.0

Page 656 of 1053

BUNDESREPUBLIK DEUTSCHLAND C	 Patentsc DE 37387 		(5) Int. Cl. 4: B 22 F 7/00 H 01 L 21/203
DEUTSCHES PATENTAMT	 Aktenzeichen: Anmeldetag: Offenlegungstag: Veröffentlichungstag der Patenterteilung: 	P 37 38 738.3-24 14. 11. 87 – 26. 1. 89	E 31 38 738 C
Innerhalb von 3 Monaten nac	h Veröffentlichung der Erte	ilung kann Einspruch erhobe	En werden
(3) Patentinhaber:		② Erfinder:	
Degussa AG, 6000 Frankfu	urt, DE	Berchtold, Lorenz, E Jürgen, DrIng., 875 Wolfgang, DiplIng Udo, 6458 Rodenba	
		(56) Für die Beurteilung in Betracht gezogen	
		DE 35 37 19	1 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.

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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 Vakuden in Form von Vorlegierungen mit den entsprechenden Übergängsmetallen 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 Tar- 20 gets 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. 25

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 30 Schichten in der Elektronik und Datentechnik über Korrosions- und Verschleißschutzschichten bis zu optischen Schichten für dekorative und wärmetechnische Zwecke reicht.

(Target) und Gegenelektrode eine elektrische Gasentladung statt, bei der von den aufprallenden Ionen aus dem Target Teilchen von atomarer Größe herausgeschlagen und auf Substraten, die im Bereich der Gegenelektrode angeordnet sind, niedergeschlagen werden. 40

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 üblicher- 45 weise aus dem Material, aus dem die zu bildende Schicht bestehen soll, während beim Reaktivsputtern 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 55 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 Zusammen- 60 setzung 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. Dar- 65 über 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 gro-Ben spezifischen Oberfläche der Pulver und der daraus resultierenden Reaktionsfreudigkeit lassen sich keine hochwertigen Targetqualitäten mit niedrigem Sauerstoffgehalt pulvermetallurgisch herstellen.

Außerdem weisen pulvermetallurgisch hergestellte um, dadurch gekennzeichnet, daß die Seltenen Er- 10 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 insbeson-15 dere 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, Beim Kathodenzerstäuben findet zwischen Kathode' 35 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 gro-Ben 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 eutekti-50 scher 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.

Kompaktierungsbedingungen (Temperatur, Die 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 Erd-Phasen und der Übergangsmetallphasen gebildet werden. 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 Berührungsstellen der Pulverkörner und in den Körnern.

Es hat sich überraschenderweise gezeigt, daß aus Vorlegierungen der Seltenen Erden mit den entspre- 10 chenden 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 15 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 Zerkleinerungs- 20 vorgang 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 25 Inertgasverdüsen aus den niedriger schmelzenden Vorlegierungen aufgrund der bei den tieferen Prozeßtemperaturen geringeren Reaktionsgeschwindigkeiten mit dem Tiegelmaterial und mit der Atmosphäre und aufgrund der kürzeren Verweilzeit in der Anlage, niedrige-30 re Werte an Verunreinigungen und geringere Sauerstoffgehalte im Pulver erzielt. Desweiteren lassen sich hier die Verdüsungsparameter (Überhitzung, Abkühlrate, Prozeßgasdruck usw.) in weiten Maßen optimieren, so daß die Pulverausbeute und die Kornfraktion im Ver-35 gleich 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 50 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 55 Verfahren näher erläutern:

 Zur Herstellung von hochreinen, kompakten, mechanisch stabilen Sputtertargets wird zuerst eine Vorlegierung aus 80 Atom% Terbium und 20 60 Atom% Eisen in einem Vakuuminduktionsofen (10-⁵ Pa) hergestellt. Die anschließende Pulverisierung und Weiterverarbeitung erfolgt ausschließlich unter Argon-Schutzgas. Mit Hilfe eines Wellenfräsers wird der Gußblock zu grobem Pulver zerspant. 65 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-, Kobaltund 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 Kapselmaterials 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 to 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 Vorlegierungspulvern, da die Reaktion der Schmelze mit dem Tiegelmaterial und der Atmosphäre vergleichsweise gering ist. Des weiteren zeigt die Vorlegierungsschmelze 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 Mischund Kompaktierungsverfahren erhält man ebenfalls hochreine, kompakte, stabile Formkörper mit Sauerstoffgehalten <1000 ppm. Die Ungleichge-

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

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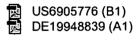
Da die intermetallischen Phasen in den Vorlegierungen fein verteilt vorliegen, und das Gefüge nicht sehr verspröden, lassen sich die Mengenanteile an freiem Eisen im Vergleich zu den intermetallischen Phasen durch Variation der Zusammensetzung der Vorlegierungen reduzieren. Die magnetischen Eigenschaften der Targets, die für das Magnetron-10 sputtern wichtig sind, lassen sich hierdurch verbessern. 6

*

Conducting transparent coatings and method for their production

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Publication date:	2001-04-18			
Inventor:	STOLLENWERK JOHANNES PROF DR (DE); KOEPPEL ANDREAS (DE); BENDER MARCUS (DE)			
Applicant:	BPS ALZENAU GMBH (DE)			
Classification:				
- international:	C03C17/36; C03C17/36; (IPC1-7): C03C17/36			
- european:	C03C17/36			
Application number:	EP20000119591 20000907			
Priority number(s):	DE19991048839 19991011			





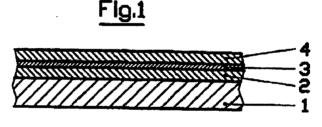
Cited documents:



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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 <-1> for the wavelengths 435, 545 and 610 nm at a flat resistance Rs 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.



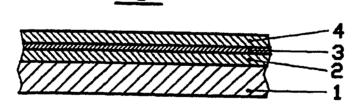
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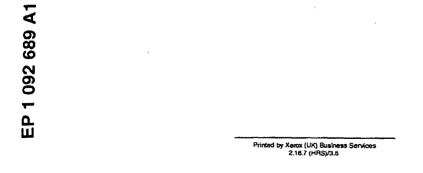


(54) Leitende transparente Schichten und Verfahren zu ihrer Herstellung

(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.





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Beschreibung

[0001] Die Erfindung betrifft leitende transparente Schichten nach dem Oberbegriff des Patentanspruches 1 sowie ein Verfahren zur Herstellung dieser leitenden 5 transparenten Schichten nach Patentanspruch 6.

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[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 niederer Flächenwiderstand angestrebt. Als Mass für die Güte leitender transparenter Schichten kann der Haakkesche Gütefaktor $\Phi_{IC} = T^{10}/R_S$, definiert in Journal of 15 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_{sg} . So besitzt beispielsweise eine Schicht mit einer Transmis-20 sion von 90% und einem Flächenwiderstand von 3 Ω_{so} einen Haackeschen Gütetaktor von 0.116 Ω^{-1} . Eine Schicht mit einer Transmission von 80% und einem Flächenwiderstand von 5 tug besitut einen Gütefaktor von $0.021 \Omega^{-1}$. 25

[0003] Eine weitere wichtige Eigenschaft eines solchen Schichtsystems ist seine Atroarkeit. Diese hängt von seiner chemischen Zusammensetzung und seiner Dicke ab. Fur eine kurze Atzzeit und gute Kantenschärfe ist es wichtig, dass die Schichtdicke möglichst 30 klein ist, d.h. unter 100 nm betragt.

[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 gleichzeltig Ihre Reflexion vermindert und dadurch die Transmission erhöht. Diese Schichtkombinationen besitzen ferner 40 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 -45 229), Damit können Ätzprozesse, wie sie bei der Herstellung von Displays üblich sind, schneller und mit geringerer Unterätzung hergestellt werden.

Solche Schichtsyteme sind z. B. beschrie-[0005] ben 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

In der EP 0 599 071 A1 wird ein Schichtsy-[0006] stern mit der Schichtfolge Indium-Zinn-Oxid, Silber bzw. verschiedene Silberlegierungen, Indium-Zinn-Oxid 55 beschrieben. Durch einstündige Temperung bei 300°C lassen sich Schichten mit einem Flächenwiderstand von 3,2 Ω_{sq} und gleichzeitig guter Transmission im

sichtbaren Bereich herstellen. Für die Wellenlängen 435, 545 und 610 nm ergibt sich ein gemittelter Haackescher 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 Ωsg und hoher Durchlässigkeit bei 550 nm erhalten. Die erhöhten Kosten durch die Goldbeimengung und der relativ hohe Flächenwi-

derstand müssen als Nachteile angesehen werden. In Thin Solid Films 341 beschreiben K. K. [0008] 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 Ω_{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 Ω_{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 Haackeschen Gütefaktor von 0,104 Ohm-1.
- [0010] Im Displaybereich für grossflächige flache LCD-Displays oder Computermonitore mit Bilddiagonalen vorzugsweise über 17" werden nun transparente Elektroden mit noch niedererem Flächenwiderstand bei gleichzeitig hoher Durchlässigkeit im sichtbaren Bereich, d. h. einem hohem Haackeschem 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 niedereren Flächenwiderstand bei einem hohen Haackeschen Gütefaktor zu erreichen
- [0012] Diese Aufgabe wird gelöst durch ein Schichtsystem nach Anspruch 1 sowie durch ein Verfahren nach Anspruch 6. Die abhängigen Patentansprüche beschreiben weitere bevorzugte Ausführungen der

inntin «FP 1092689A1 I.>

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 Ω_{sq} , vor- *s* zugsweise 2,5 Ω_{sq} und geringer auf, bei einem über die Wellenlängen 435, 545 und 610 nm gemittelten Haakkeschen Gütefaktor von grösser als 0,085 Ω^{-1} .

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[0014] Dabei ist es aus Gründen der Farbneutralität günstig, wenn bei einem Flächenwiderstand von 2,5 Ω_{sq} die optische Transmission bei 435 nm mindestens 89 %, bei 545 um 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 AL-% 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 % urd instesonoere 0,5 bis 1 %.

100181 Bei der Herstellung des beschriebenen Schichtsystems ist es entscheidend, wie in Anspruch 6 und weiteren achängigen Ansprüchen beschrieben, dass die Aufbringung der zweiten Oxidschicht nicht mit reiner CC Zerstaubung, sondern mit einer gepulsten DC-Zerstaubung oder mit einer AC-überlagerten DC-Zerstaubung erfcligt. Die AC-Überlagerung wird beiscielsweise dadurch erzeugt, dass das Ausgangssignal 35 uper ein Hitter auf die mit einer DC-Stromversorgung gespeiste Sputterquelle eingekoppelt wird. Eine weitere Mogicinaeit besteht beispielsweise auch darin, die DC-Stromversorgung entsprechend zu modulieren oder zu tasten (choppern). Es sind also verschiedene Modula-40 tionen mögich

[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, 45 definiert durch das Verhältnis der eingespeisten DCund 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/cm², vor- *so* zugsweise von 2 bis 2.2 W/cm².

[0022] Als Zerstäubungsmethode wird Magnetronsputtern 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.

- 10 [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
- 15 mit der Aufstäubung der ersten Oxidschicht aus Indiumund Ceroxid begonnen. Diese Oxidschicht wird teilreaktiv von einem Oxidtarget abgestäubt, d. h. in einer Argonatmosphäre von ca. 2,2x10⁻³ hPa mit einer Beimischung von Sauerstoff von maximal 5 %. Dieser Zer-
- stäubungsprozess ist ein reiner DC-Prozess. Typische 20 Zerstäubungsraten sind 5 bis 8 nmxm/minxcm²W. Anschliessend erfolgt als reiner nicht-reaktiver DC-Prozess das Aufstäuben der Silberschicht. Hier liegen die typischen Zerstäubungsraten bei 12 bis 15 25 nmxm/minxcm²/W. Ihm schliesst sich das Aufstäuben der zweiten Oxidschicht mit einer AC-überlagerten DC-Zerstäubung an. Dabel 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 Zerstäubungs-30 prozesses 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 Ω_{sg} .

[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, 5 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 20 Nickellegierungen mittels DC-Magnetron-Zerstäubung aufgebracht werden.

Patentansprüche

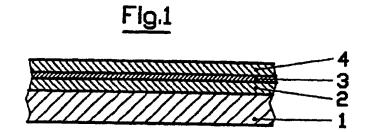
- 1. Leitendes transparentes Schichtsystem mit zwei Oxidschichten (2,4) und einer dazwischen gelagerten Silber-Schicht (3) auf einem Substrat (1), **dadurch gekennzeichnet, dass** bei einem Flächenwiderstand R_s von < 2,9 Ω_{sq} , vorzugsweise < 2,5 Ω_{sq} und weniger, der mittlere Haackesche Gütefaktor des Schichtsystems für die Wellenlängen 435, 545 und 610 nm ($\Phi_{Tc}=T^{10}/R_s$)>0,085 Ω^{-1} ¹beträgt.
- 2. Schichtsystem nach Anspruch 1, dedurch gekennzeichnet, dass bei einem Flächenwider- 45 stand von 2,5 Ω_{sq} die Durchlässigkeit T bei 435 nm mindestens 89 %, bei 545 nm mindestens 88 % und bei 610 nm mindestens75% beträgt.
- Schichtsystem nach einem der vorangehenden 50 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 < 55 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.

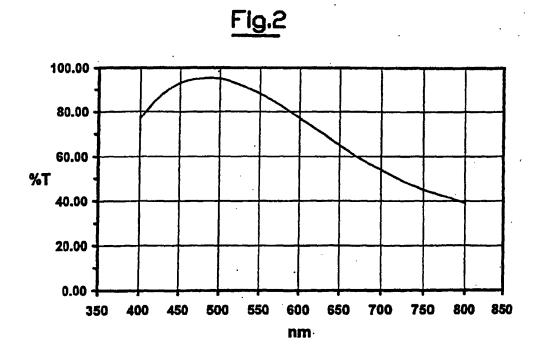
- Schichtsystem nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass die Silber-Schicht (3) bis zu 10 Gew.-% Kupfer enthält, vorzugsweise im Bereich o.5 bis 3 % und insbesondere 0,5 bis 1 %.
- Verfahren zur Herstellung eines leitenden transparenten Schichtsystems nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, dass bei der Aufbringung der zweiten Oxidschicht (4) eine gepulste DC-Zerstäubung oder eine ACüberlagerte DC-Zerstäubung verwendet wird.
- 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.
- 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.
- 30 9. Verlahren nach Anspruch 6, 7 und 8, dadurch gekennzelchnet, dass die totale Leistungsdichte (AC und DC) im Bereich 1 bis 3 W/cm², vorzugsweise aber bei 2 bis 2.2 W/cm² liegt.
 - Verfahren nach Anspruch 6 bis 9, dadurch gekennzeichnet, dass als Zerstäubungsverfahren Magnetronzerstäubung gewählt wird.
 - 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 gekennzelchnet, dass es nach dem Verfahren nach den Ansprüchen 6 bis 10 gefertigt wurde.

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UNVOLLSTÄNDIGE RECHERCHE ERGÄNZUNGSBLATT C

Nummer der Anmeidung

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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: P1: 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.

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

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06-02-2001

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

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THOMSON

MACHINE-ASSISTED TRANSLATION (MAT):

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

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- (71)【出願人】 (71)[PATENTEE/ASSIGNEE]
- 【識別番号】 [ID CODE] 000226688 000226688

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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 Prevent the crack by heat of a target material. れを防止すること。

【構成】

てバッキングプレート3に固着 bonding material 2. スリ割り溝(或いは切り込み、 け、ターゲット材1を多数の小 subsection 1_1 . の割れを防ぐことができる。予 target material. り付けても良い。

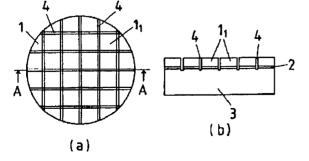
[CONSTITUTION]

所要の元素を含むターゲット The target material 1 containing a required 材1はボンディング材2によっ element adheres to the backing plate 3 by the

されている。ターゲット材1に It provides the offset rate slot (or an incision, the slot by cutting) 4 in a target material 1, and カッティングによる溝) 4を設 partitions a target material 1 into many

区画11に分割する。ターゲット The difference of the elongation in the thickness 材の厚さ方向位置での伸びの差 direction position of a target material etc. 等が小さくなり、ターゲット材 becomes smaller, it can prevent the crack of a

め作っておいたターゲット材の It is sufficient to bond on a backing plate the 小片をバッキングプレートに貼 fragment of the target material made beforehand.



【特許請求の範囲】

【請求項1】

バッキングプレートに固着され sticking. ていることを特徴とするスパッ タ・ターゲット。

[CLAIMS]

[CLAIM 1]

バッキングプレートに固着さ A sputter target, in which the target material れたターゲット材がスリ割り溝 which adhered to the backing plate is によって多数の小区画に分割さ partitioned into many subsections by the offset れているか、又はターゲット材 rate slot, or many fragments of a target material の多数の小片がタイル貼り状に adhere to the backing plate in the shape of a tile

【請求項2】 ットを備えていることを特徴と target of Claim 1. するスパッタ型イオン源。

【発明の詳細な説明】

[CLAIM 2] 請求項1のスパッタ・ターゲ A sputter type ion source, which has the sputter

[DETAILED DESCRIPTION OF THE INVENTION]

[0001]

【産業上の利用分野】 ターゲットに関する。

[INDUSTRIAL APPLICATION]

本発明は、加熱によるターゲッ This invention relates to the sputter target which ト材の割れを防いだスパッタ型 is used for the sputter type ion source which イオン源等に用いるスパッタ・ prevents the crack of the target material by heat.

[0002]

[0001]

[0002]

【従来の技術】

[PRIOR ART]

けるスパッタ・ターゲットは、

スパッタ型イオン源、イオンビ As shown in the front elevation of FIG.4(a), and ームスパッタリング装置等にお sectional drawing of said (b) in the A-A, the sputter target in a sputter type ion source, an

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 ion beam. いはイオンビームの衝撃により holder for the cooling. 加熱されるから、その冷却のた めにバッキングプレート3は冷 却ホルダーに取り付けられてい る。

図4(a)の正面図、そのA- ion-beam-sputtering apparatus, etc. adheres to A線での同(b)の断面図に示 a backing plate 3, and the bonding material 2 すように、プラズマイオン、イ comprises the tabular target material 1 オンビームでスパッタされる所 containing the required element by which a sputter is carried out from a plasma ion and an

ってバッキングプレート3に固 A target material 1 is heated by the shock of the 着して構成されている。ターゲ surrounding plasma, a plasma ion, or an ion ット材1はスパッタ時、周囲の beam at the time of a sputter, therefore

プラズマとプラズマイオン、或 The backing plate 3 is attached to the cooling

[0003]

[0003]

頿】

る等の理由により、ターゲット 1 at the time of a sputter. 材が割れてしまうことがある。

【発明が解決しようとする課 [PROBLEM TO BE SOLVED BY THE INVENTION]

かかるスパッタ・ターゲットの When the size of this sputter target becomes サイズが直径50mm程度の大 the size which is diameter 50 mm degree and a きさのものになると、ターゲッ target material 1 is the bad material of a heat ト材1 が熱伝導の悪い材質の場 conduction, a target material may break for the 合には、スパッタ時、ターゲッ reasons of a big difference arising on the ト材1の厚み方向位置での熱膨 elongation by the thermal expansion in the 張による伸びに大きな差が生じ thickness direction position of a target material

[0004]

[0004]

れることを防止したスパッタ・ heating by the plasma or an ion. ターゲットの提供を目的とする ものである。

本発明は、プラズマやイオンに This invention aims to provide the sputter target よる加熱で、ターゲット材が割 which prevents the crack of a target material by

[0005]

[0005]

【課題を解決するための手段】 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 of a tile sticking. トに固着されていることを主た 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る。

[MEANS TO SOLVE THE PROBLEM]

本発明は、スパッタ・ターゲッ As for this invention, in a sputter target, the トにおいて、バッキングプレー target material which adhered to the backing トに固着されたターゲット材が plate is partitioned into many subsections by the スリ割り溝によって多数の小区 offset rate slot, or many fragments of a target material adhere to a backing plate in the shape

イル貼り状にバッキングプレー And this sputter target is used for the sputter type ion source.

る特徴とするものであり、そし It is characterized by the above-mentioned.

[0006]

【作用】

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 form of a fragment, therefore 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 target does not arise. り、イオンビームが安定して引 出せる。

[0006]

[OPERATION]

ターゲット材がスリ割り或いは The target material is small partitioned according to an offset rate or the tile sticking

分割域における加熱による熱膨 Since the thermal expansion by the heat in each 張が他の分割域に波及すること partition region does not affect another partition がないから、ターゲット材の厚 region, the difference of the elongation in the み方向位置での伸びの差等が小 thickness direction position of a target material etc. is restrained small, the crack of a sputter

て、かかるターゲットをスパッ And it can pull out an ion beam with stability by タ型イオン源に用いることによ using this target for a sputter type ion source.

[0007]

[0007]

【実施例】

参照して説明する。図1 (a)、 Example of this design. びそのA-A線での断面図であ in the A-A. ンディング材2で固着されたタ 部分に形成されていれば充分で even the backing plate 3. あるが、図1(b)ではバッキ ングプレート3にまで達してい るものを示している。

[0008]

正面図とそのA-A線での断面 sticking form. 貼り形式でバッキングプレート 3に固着する。

[EXAMPLES]

本考案の実施例について図面を With reference to drawing, it demonstrates the

(b) はスリ割り形式によるス FIG. 1 (a), (b) is the front elevation of the sputter パッタ・ターゲットの正面図及 target by offset rate form, and sectional drawing

る。バッキングプレート3にボ It provides the offset rate slot (an offset rate or an incision, slot by cutting) 4 in the target ーゲット材1に、碁盤の目状に material 1 which adhered to the backing plate 3 スリ割り溝(スリ割りないし切 by the bonding material 2 in a grid pattern, and り込み、カッティングによる溝) one side small-partitions a target material 1 into 4を設け、ターゲット材1を一 many subsection 1_1 which are 10 mm level.

辺が10mm程度の多数の小区 If the offset rate slot 4 is formed in the target 画 11 に小分割する。スリ割り溝 material part at least, it is enough.

4は、少なくともターゲット材 However, FIG.1(b) shows what has reached

[0008]

図2(a)、(b) はタイル貼り FIG. 2(a), (b) is the front elevation and sectional 形式のスパッタ・ターゲットの drawing in an A-A of the sputter target of tile

図であり、予めターゲット材を One side partitions the target material into 一辺が10mm程度の小片12 fragment 12 which is 10 mm level beforehand, に分割しておき、かかる多数の and it adheres fragment 12 of the target material ターゲット材の小片 1_2 をボン of these many to a backing plate 3 in tile ディング材2によって、タイル sticking form by the bonding material 2.

[0009]

[0009]

このように、ターゲット材1は、 Thus, the target material 1 is partitioned into the 何れも一辺が10mm程度の小 subsection whose one side is all 10 mm level or 区画ないしは小片11、12 に分 fragment 11, and 12, therefore

熱膨張は、各小区画、小片11、 は波及せず、各小区画、小片1,、 差は小さく抑えられるから、タ restrained small, therefore い。

[0010]

源では、スパッタ・ターゲット 度に達し、スパッタ材が割れる break becomes higher. 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 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によってバッキングプレー ト3に固着しておき、スリ割り forming the offset rate slot 4. 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 heat, therefore タ・ターゲットの割れが防止で stably than an ion source.

割されているから、ターゲット Even if heat generation and heat arise at the 材1のスパッタ時に発熱、加熱 time of the sputter of a target material 1, thermal が生じても、ターゲット材等の expansion, such as a target material, is limited in each subsection, fragment 1_1 , and 1_2 , it does 12内に限定されて、他の部分に not affect the other part but the difference of the elongation in the thickness direction position of 12の厚み方向位置での伸びの each subsection, fragment 11, and 12 is

ーゲット材に割れが発生しな A crack does not occur in a target material.

[0010]

プラズマ・スパッタ型負イオン In the source of a plasma sputter type negative ion, in order to converge as a beam the 表面で発生した負イオンをイオ negative ion generated on the sputter target ン源の出口にビームとして集束 surface on the outlet of an ion source, it may させるために、スパッタ・ター make the surface of a sputter target into the ゲットの表面を球面状にえぐっ form scooped out to the spherical shape.

た形とする場合がある。このよ If it does in this way, in the periphery part of a うにすると、ターゲット材の周 target material, the thickness will amount to 5 辺部では、その厚みは5mm程 mm level, possibility that a sputter material will

> 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 ある。図1に示したものと同様 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

It can prevent the crack of the sputter target by

に分割する。加熱によるスパッ It becomes possible to pull out a beam more

きるから、イオン源より安定に It is sufficient to implement tile sticking form like ビームを引出すことが可能にな FIG.2. る。図2と同様に、タイル貼り 形式を実施してもよい。

[0011]

[0011]

【発明の効果】

[ADVANTAGE OF THE INVENTION]

ト材の割れを防止することがで target material. きる。

本発明は以上説明したように、 As for this invention, as explained above, the ターゲット材が小さく分割され target material is partitioned small, therefore ているから、スパッタ時に、タ At the time of a sputter, it can restrain small the ーゲット材における厚み方向の difference of the elongation by the thermal 熱膨張による伸びの差等を小さ expansion of the thickness direction in a target く抑えることができ、ターゲッ material etc., and can prevent the crack of a

[0012]

パッタ・ターゲットの場合に効 果的である。

[0012]

そして、本発明によるスパッ And in the case of the sputter target with which タ・ターゲットをイオン源に用 it could pull out the ion beam stably, and いることにより、イオンビーム particularly the surface was scooped out by the を安定に引出すことができ、特 spherical shape, it is effective by using the に表面が球面状にえぐられたス sputter target by this invention for an ion source.

【図面の簡単な説明】

【図1】

面図である。

[BRIEF DESCRIPTION OF THE DRAWINGS]

[FIG. 1]

本発明の実施例の正面図及び断 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 It is the front elevation and sectional drawing of である。 another Example.

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【図3】

面図である。

【図4】

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

3 バッキングプレート

2 ボンディング材

[FIG. 3]

更に他の実施例の正面図及び断 Furthermore, it is the front elevation and sectional drawing of another Example.

[FIG. 4]

従来のスパッタ・ターゲットの It is the front elevation and sectional drawing of a sputter target of the past.

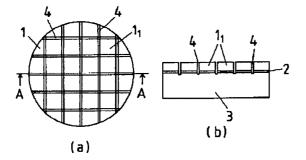
[DESCRIPTION OF SYMBOLS]

- $1, 1_1, 1_2$ target material
- 2 Bonding material
- 3 Backing plate
- 4 スリ割り溝

4 Offset rate slot

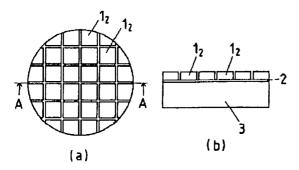
【図1】

[FIG. 1]





[FIG. 2]

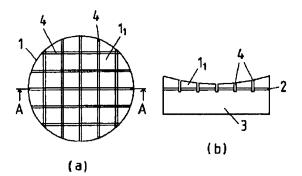


-

【図3】

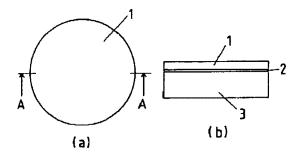
.

[FIG. 3]



【図4】

[FIG. 4]



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Page 1 of 2

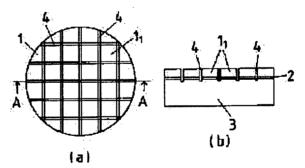
SPUTTERING TARGET

Publication number: JP5230642				
Publication date:	1993-09-07			
Inventor:	BABA TAKASHI; TAKEYAMA KUNIHIKO			
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			

Report a data error here

Abstract of JP5230642

PURPOSE: To prevent the cracks of a target material caused by heating. CONSTITUTION: A target material 1 contg. 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(JP)

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(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	FΙ	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)

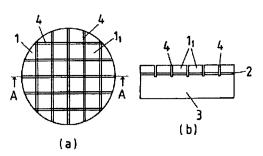
(21)出顧番号	特顧平4-69981	(71)出顧人 000226688
		日新ハイボルテージ株式会社
(22)出顧日	平成4年(1992)2月21日	京都府京都市右京区梅津高畝町47番地
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		新ハイボルテージ株式会社内
		(74)代理人 弁理士 成田 擴其

(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はポンディ ング材2によってバッキングプレート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 の小区面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 をパッキングプレートに貼り付けても良い。



【特許請求の範囲】

【請求項1】 バッキ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 にパッキ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。

1

【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をポン ディング材2によってバッキングプレート3に固着して 20 ッキングプレート3に固着する。

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キングプ 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 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、バッキ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 貼り状にパッキングプレートに固着されていることを主 40 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 50 ることができる。

2 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。パッキングプレート3にポンディング材2で固着 されたターゲット材1に、碁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 10 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) ではパッキングプレート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、タイル貼り形式でパ

【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、 加熱が生じても、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 による伸びに大きな差が生じる等の理由により、ターゲ 30 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をポンディング材2によってパッキングプレート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

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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】

3

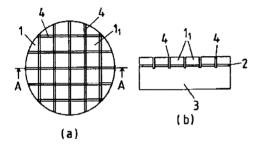
• 2. * • • •

【図1】本発明の実施例の正面図及び断面図である。

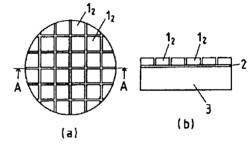
【図2】他の実施例の正面図及び断面図である。

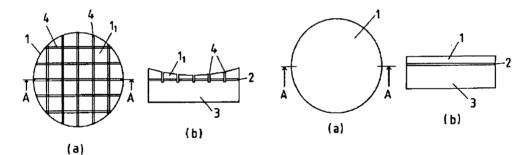
【図3】更に他の実施例の正面図及び断面図である。

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











PATENT ABSTRACTS OF JAPAN

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(43) Date of publication of application: 22.08.95

(51) Int. CI C23C 14/34		
(21) Application number: 06017653	(71) Applicant:	ULVAC JAPAN LTD
(22) Date of filing: 14.02.94	(72) Inventor:	OTA ATSUSHI HAGA HIDEAKI TANI NORIAKI SUU KOUKOU KOMATSU TAKASHI NAKAMURA KYUZO MOMONO TAKESHI KAWAMURA HIROAKI SUZUKI IKUO IKEDA SATOSHI ISHIKAWA MICHIO OTA YOSHIFUMI MATSUMOTO MASAHIRO

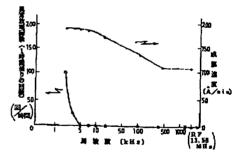
(54) SPUTTERING METHOD AND DEVICE THEREFOR

(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 negativepotential 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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mimosa

(12) 公開特許公報(A)

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特開平7-224379

(43)公阴日 平成7年(1995)8月22日

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C 2 3 C 14/34	R	8414-4K			
	А	8414-4K		•	
	М	8414-4K			

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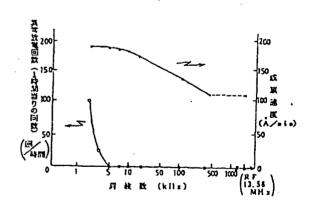
(21)出願番号	特願平6-17653	(71)出願人	000231464
	,		日本真空技術株式会社
(22)出願日	平成6年(1994)2月14日		神奈川県茅ヶ崎市萩園2500番地
		(72)発明者	太田 淳
	• • • •		千葉県山武郡山武町横田523 日本真空技
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	1 FN	(74)代理人	弁理士 北村 欣一 (外2名)
			最終頁に統く

(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】

(19)日本国特許庁(JP)

【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 常放む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 ットに正面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

監修 日本国特許庁

・【特許請求の範囲】

【訪求項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、T a、Tl、C、ITO、ZnO、SnO2 またはこれら の合金であり、また、反応ガスはN1, O2、H1, N 10 H1, CO、CO2、CH4、C2 H1、H1 O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。

【訪求項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、AI、T a、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、N H3、CO、CO2、CH4、C2 H2、H2 O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項に記載のスパック装置。

【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 30 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、 光学腺、保護膜等の膜を成膜するスパッタ方法に用いら 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 40 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13.56MIIz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、 道流電源を用いる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 緑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。

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【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。

【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、高速成版を必要とする 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。

【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置 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 20 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、A l、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、H2 O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、A 1、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、 II: NII: 、CO、CO2、CII: 、C2 II: 、II: O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発性ターゲットに直流沿 源より直流電圧を印加し、スパッタリングを行うとター 50 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(+)イオンが都積される。 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、アース電板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 10 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 ス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館位の印 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1 3.56MHz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にパルブ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の前面に導む 30 性ターゲット6をロウ材でポンデ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 べ等のガス供給源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 9 aにガス圧調節弁10 aを介して不活性ガス(例えば アルゴンガス)のガス供給源8 aを接続し、他方の分岐 管9 bにガス圧調節弁10 bを介して反応ガス(例えば 室素ガス)のガス供給源8 bを接続した。

【0021】また、ターゲットカソード5に直流電源1 1をパルスユニット12を介して接続し、パルスユニッ 40 ト12を調節して導発性ターゲット6に負発位と正社位 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の背面傾にマ 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に取り付けられた将れ性ター 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、真空処理次1内に茘板4と、ターゲットカソード 5に将乱性ターゲット6としてケイ素(S1)製ターゲ ットを装着した。

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【0025】 続いて、真空処理室1内を真空排気系2に より 6.7×10⁺Pa (5×10⁻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 8 b からの窒索(N1) 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⁻Pa (5×10⁻)T orr)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(SINx)の屈折率が2.03となるように ArガスとNa 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 900人の窒化ケイ素(SINx)膜を形成 した。尚、負重位のターゲットにバルス状に印加する正

20 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 2500 e 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載位に印加する 正載位の周波数の増加に伴い、異常放電回数は減少し、

- 0 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(13.55MHz)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電味がない。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。
- 40 【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 れし、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 50 るようにしている。

(4)

【0030】実施例2

反応ガスとして酸素(O₁)ガスを用い、スパッタ圧を 6.7×10⁻¹ Pa(5×10⁻³ Torr)とし、直流電力を2k Wとし、基板上に形成される酸化ケイ素(SiOx)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1000人の酸化ケイ素(SiOx)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 10 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S1Nxの 測定結果(瓜 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 20 を 6.7×10⁻¹ Pa(5×10⁻³ Torr)とし、直流電力を2 kW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 200人のダイヤモンドライクカー ポン(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 正元位の周波数は約5kllzで異常放乱をほぼ防止し得 30 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

【0039】また、前記実施例1では導電性ターゲット にS1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(SiNx)膜を形成したが、導電性ター ゲットとしてS1の代わりにIn:O1-SnO:焼結 体(ITO)、酸化亜鉛(ZnO)、酸化スズ(SnO 2)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(C O2)、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、負重位の

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ターゲットに印加する正知位の周波数を5~400kH z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、 SnOz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、負重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例の概略截断側面図。

【図2】 図1 装置による導発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実施例における成版 時の場波数と異常放電回数との関係、並びに周波数と成 脳速度との関係を示す特性線図、

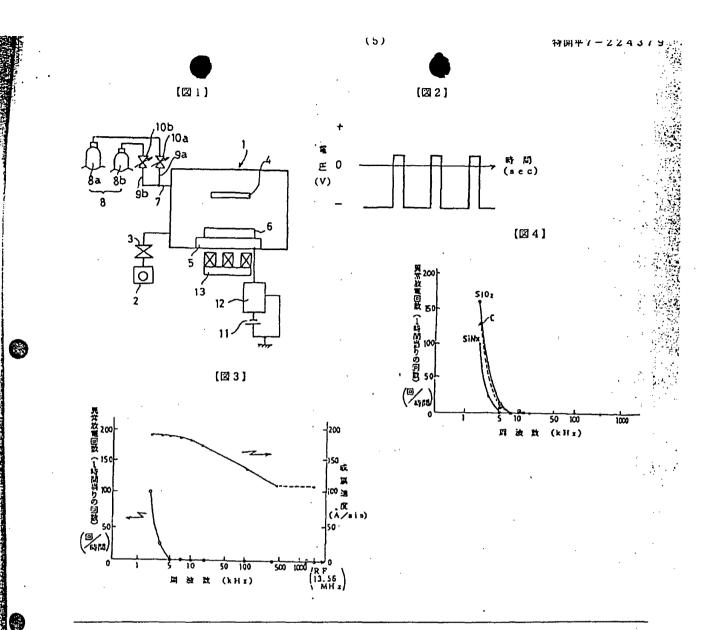
(図4) 本花明スパッタ方法の他の実施例における成 限時の周波数と異常放電回数との関係を示す特性線図。 (符号の説明)

 1
 真空処理室。
 2
 真空排気系、
 4

 塔板、6
 導電性ターゲット、
 7
 ガス導入

 宮、8
 スパッタガス供給源、
 1.1
 直流電

50 旅、12 パルスユニット。



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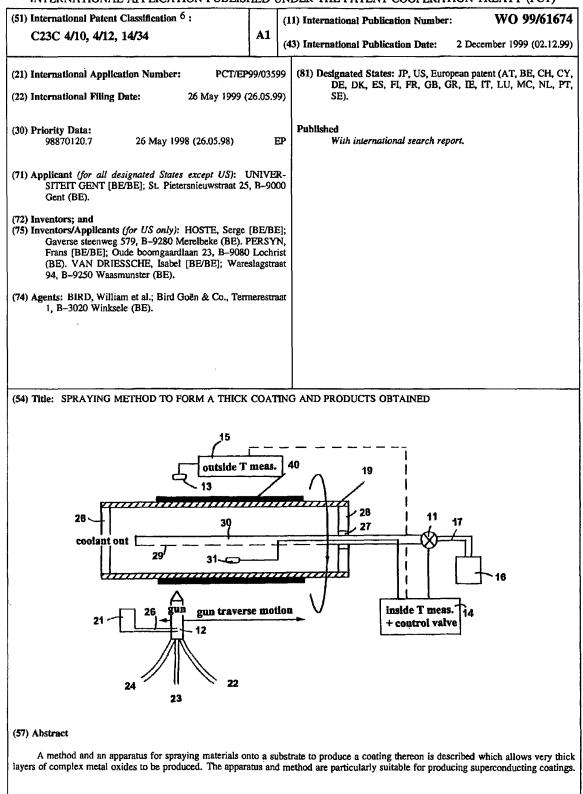
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WO 99/61674

1 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

formation of metallic or ceramic coatings such as superconductive or piezo-5 electric layers or for the production of targets for sputtering magnetrons having coatings which are precursors of such layers.

TECHNICAL BACKGROUND

- From EP-A-286 135 it is known to flame spray complex ceramic materials 10 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
- used for the flame spraving. Thickness of up to 3 mm are described. 15

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,

water cooling is unsuitable for superconductors due to the water vapour produced. 25

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 of Fe-C-Mo Alloys by Flame Spraying" describes up to 1.5 mm thick rapidly 30

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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
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
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
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,

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- 5 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 Wm⁻¹K⁻¹. When deposited on a steel substrate the thermal conductivity of the composite
- preferably lies within the range 25 to 125 Wm⁻¹K⁻¹. These values are particularly preferred for YBa₂Cu₃O₇ 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 Inalloy, for example. The deposited coating is preferably impact resistant, e.g.
- 15 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 in included as a salt or oxide, e.g. silver nitrate or silver oxide, in the material to be sprayed. Preferably, the electrical
- 20 resistivity of the deposited layer is lower than 15 x 10⁻⁶ Ohm.m, more preferably lower than 10 x 10⁻⁶ and most preferably less than 5 x 10⁻⁶ Ohm.m. Values below 1 x 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 YBa₂Cu₃O₇ 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.

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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.

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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

15 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. 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.

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The present invention may provide a method of flame spraying a

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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 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 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 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.

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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 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
- 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 (~25°C) and 150°C, more preferably between room temperature (~25°C) and 100 °C.

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The above methods may be used, for example, either as part of the direct 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

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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.

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 YBa₂Cu₃O₇ 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
- suitable for use as a sputtering magnetron target have been applied by oxyacetylene flame spraying with water cooling or by atmospheric pressure or lowpressure 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)

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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

5 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

- 10 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
- 15 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
- 20 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
- 25 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

30 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

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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
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 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.

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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

- 5 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
- 10 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
- 15 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

- 20 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 YBa₂Cu₃O₇ 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
- 30 be 10 to 50 micron thickness of the coating. About 10% to 15% of the deposited

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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

- 5 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 Wm⁻¹K⁻¹. When deposited on a steel substrate the thermal conductivity preferably lies within the range 25 to 125 Wm⁻¹K⁻¹. These values are particularly preferred for
- YBa₂Cu₃O₇ 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%
- 15 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 in 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 x 10⁻⁶ Ohm.m, more preferably lower than 10 x
- 20 10⁻⁶ and most preferably less than 5 x 10⁻⁶ Ohm.m. Values below 1 x 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 YBa₂Cu₃O₇ coatings.

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

- 25 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
- 30 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 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

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5 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}Cu_nO_{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
- 20 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

30 preferably be compatible with thermal decomposition to oxides in further

Page 709 of 1053

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
- 15 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

20 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.

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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 YBa₂Cu₃O₇ 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 YBa₂Cu₃O₇ 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.

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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

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5 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

- 10 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
- 15 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 as process is particularly economic when the target
- 20 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
- 25 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

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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.

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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

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.

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6. The target or a composite in accordance with any previous claim, wherein the coating has a thermal conductivity of between 1 and 5 $Wm^{-1}K^{-1}$.

7. The target or a composite in accordance with any previous claim, wherein the
 thermal conductivity of the composite or the target through the substrate and the
 coating is in the range 25 to 125 Wm⁻¹K⁻¹.

8. The target or a composite in accordance with any previous claim, wherein the coating has an electrical resistivity of lower than 15×10^{-6} Ohm.m, more preferably

30 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.

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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.

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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.

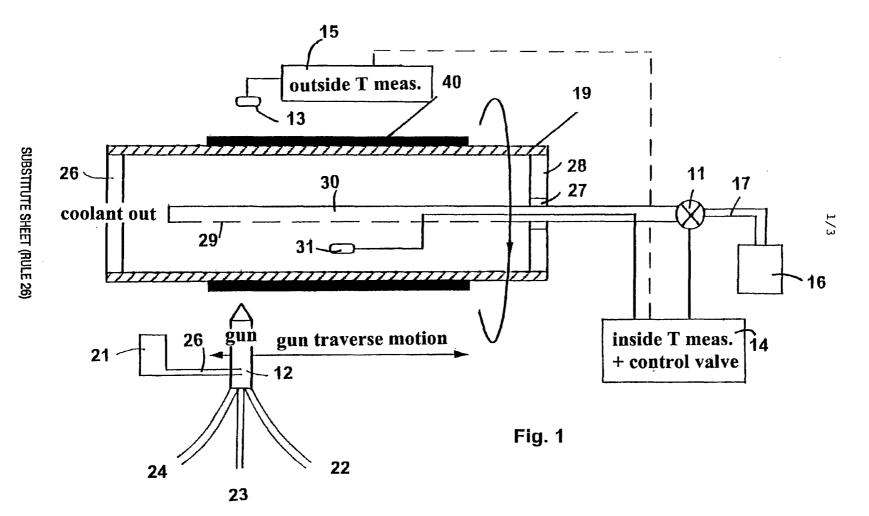
14. The method according to claim 13, wherein the spraying step includes spraying
a material through a spraying head, the material being in the form of a powder, a
slurry or a solution.

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

25 pressure plasma spraying target material into the erosion groove.

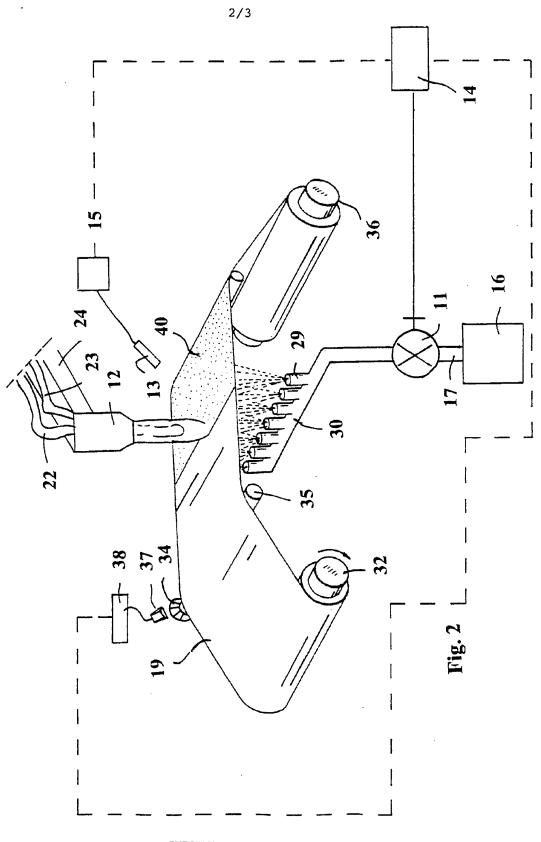
16. A reconditioned target for a sputtering magnetron, comprising: an erosion groove in the target material; and target material flame sprayed or atmospheric plasma sprayed into said groove to restore the thickness of the target

30 material to that of the unused material.

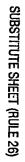


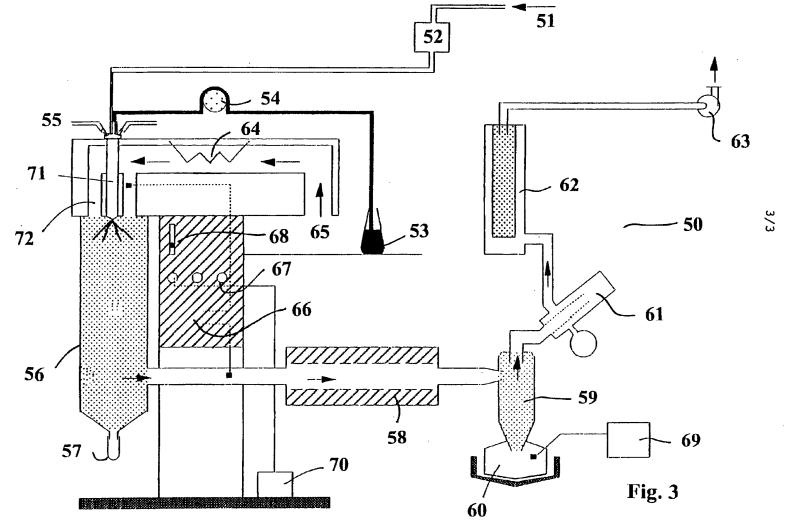
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SUBSTITUTE SHEET (RULE 26)





PCT/EP99/03599

	INTERNATIONAL SEARCH F		Intrational Application No	
			PUI/EP 99/03599	
A. CLASSI IPC 6	FICATION OF SUBJECT MATTER C23C4/10 C23C4/12 C23C14/3	4		
According t	o International Patent Classification (IPC) or to both national classific	ation and IPC		
	SEARCHED			
Minimum de IPC 6	ocumentation searched (classification system followed by classificati C23C	on symbols)		
Documenta	tion searched other than minimum documentation to the extent that s	uch documents are inclu	ded in the fields searched	
Electronic d	lata base consulted during the international search (name of data ba	se and, where practical,	search terms used)	
C. DOCUM	ENTS CONSIDERED TO BE RELEVANT			
Category *	Citation of document, with indication, where appropriate, of the ret	evant passages	Relevant to claim No.	
X	US 5 196 400 A (CHEN CHIOU T ET 23 March 1993 (1993-03-23) cited in the application column 3, line 52 - column 4, li		1-5, 10-14	
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X	DD 277 471 A (MANSFELD KOMBINAT W VEB) 4 April 1990 (1990-04-04) page 2, line 21 - line 23	I PIECK	15,16	
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X Furt	her documents are listed in the continuation of box C.	X Patent family n	nembers are listed in annex.	
"A" docum	ategories of citad documents : ent defining the general state of the art which is not tered to be of particular relevance	or priority date and	shed after the international filing date not in conflict with the application but the principle or theory underlying the	
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"P" docume later ti	ent published prior to the international filing date but han the priority date claimed	in the art. *&" document member of the same patent family		
	actual completion of the international search September 1999	Date of mailing of t	he International search report	
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	European Patent Office, P.B. 5818 Patentiaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 851 epo ni, Fax: (+31-70) 340-3016	Patterson, A		

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	ernational application No.				
INTERNATIONAL SEARCH REPORT	PCT/EP 99/03599				
Box I Observations where certain claims were found unsearchable (Continu	lation 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 Authonty. r	namety:				
2. X 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	ng ang 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	n. as follows:				
SEE FURTHER INFORMATION SHEET					
 As all required additional search fees were timely paid by the applicant, this internation searchable claims. 	onal Search Report covers all				
2. X As all searchable claims could be searched without effort justifying an additional fee.	this Authority aid not invite payment				
3. As only some of the required additional search fees were timely paid by the applicant covers only those claims for which fees were paid, specifically claims Nos.:	t. this International Search Report				
4. No required additional search fees were timely paid by the applicant. Consequently, the restricted to the invention first mentioned in the claims: it is covered by claims Nos.:	this International Search Report is				
Remark on Protest The additional search fees were No protest accompanied the pay	accompanied by the applicant's protest. ment of additional search tees.				

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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 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.

	hann			national Application No (/EP 99/03599		
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Form PCT/ISA/210 (patent family annex) (July 1992)

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•		al Publication Date 6 (15.06.2006)	PCT	(10) International Publication Number WO 2006/063308 A2	
•	(51) International Paten B05D 5/12 (2006.01 H01M 4/52 (2006.01) H01M 10/38 (2006.01)	4) Agent: EDWARDS, Gary, J.; Finnegan, Henderson, Farabow, Garrett & Dunner LLP, 901 New York Avenue, Washington, D.C., District of Columbia 20001-4413 (US).	
	(21) International Appli	cation Number: PCT/US2005/	•	 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, CH, CZ, DE, DK, DM, DZ, CH, EG, ES, KI, 	
	(22) International Filing	g Date: 7 December 2005 (07.12	2.2005)	CO, CR, CU, CZ, DE, DK, DM, DZ, BC, 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,	
	(25) Filing Language:(26) Publication Langua		English English	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.	
	(30) Priority Data: 60/634,818 5 60/651,363	8 December 2004 (08.12.2004) 8 February 2005 (08.02.2005)	US	4) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KR, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM),	
	MORPHIX, INC. Sunnyvale, CA 9408	lesignated States except US): [US/US]; 1278 Reamwood A 9-2233 (US).	venue,	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).	
	 (72) Inventors; and (75) Inventors/Applicant [US/US]; 1330 Rodr DEMARAY, Richar tola Valley, CA 9402 Nectar Circle, Elk Gr 	ts (for US only): ZHANG, Ho ey Drive, San Jose, CA 95118 d, E. [US/US]; 190 Fawn Lan 8 (US). SHAO, May [US/US] ove, CA 95757 (US).	ongmei — 8 (US). e, Por- Fa ; 5401 an	ublished: - without international search report and to be republished upon receipt of that report or two-letter codes and other abbreviations, refer to the "Guid- tice Notes on Codes and Abbreviations" appearing at the begin- ng of each regular issue of the PCT Gazette.	
	(54) Title: DEPOSITION	OF LICoO2			
				300	
		CURREN	VT COLL		
			Lipon		
82			LiCoO ₂		
808		SU	E <u>301</u>		
2006/063308 A2	the LiCoO ₂ layer. Some en	a deposition can provide a low or <003> orientation. Some on the utilized as the cathode lay ation can eliminate the high ten nbodiments of the process can	v-temperatur embodiment er in a solid nperature (>	n of LiCoO ₂ layers in a pulsed-dc physical vapor deposition re, high deposition rate deposition of a crystalline layer of Li- s of the deposition addresses the need for high rate deposition state rechargeable Li battery. Embodiments of the process ac- 700°C) anneal step that is conventionally needed to crystallize battery utilizing the LiCoO ₂ layer by utilizing a rapid thermal	
0M	anneal process with short r	amp rates.			

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DEPOSITION OF LICoO₂

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 LiCoO₂ 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

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the presence of the LiCoO₂, which can experience significant interfacial reactions with most materials utilized in a battery during these temperature cycles.

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Other lithium containing mixed metal oxides besides LiCoO2, including Ni, [004] 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 LiCoO₂, 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

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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.

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[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.

crystalline material, for example LiCoO₂ 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 <101> 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 °C) anneal step that is conventionally needed to crystallize the LiCoO₂ layer.

[012] A method of depositing a LiCoO₂ 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

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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 <101>.

[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 LiCoO₂ layer deposited over the conducting layer, a LiPON layer deposited over the LiCoO₂ 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 $LiCoO_2$ 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

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[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 LiCoO₂ 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 LiCoO₂ deposited according to some embodiments of the present invention on a thin substrate.

[024] Figure 6B illustrates a layer of LiCoO₂ 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 LiCoO₂ 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 LiCoO₂ layers deposited according to some embodiments of the present invention.

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[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

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invention provide a crystalline LiCoO₂ 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 LiCoO₂ 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 LiCoO₂ uniformity of 3% onesigma measured at 25 points at a deposition rate of 1.2 microns thickness an hour over an area of 2000 cm², without the need for secondary front side ion source or ion assistance.

[039] In one example process, a LiCoO₂ film was deposited utilizing a conductive ceramic LiCoO₂ target as described herein, with pulsed-dc power of 4 kW, no bias, with 60 sccm Ar and 20 sccm O₂ gas flows. A 3000 Angstrom layer of crystalline LiCoO₂ 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 μ m with a maximum of 3.09 μ m and a minimum of 2.70 μ m 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.

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[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 LiCoO₂ 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 LiCoO₂ film with crystallinity and with sufficient stoichiometry, a battery utilizing as-deposited LiCoO₂ films

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can be produced. Heat treating the LiCoO₂ layers may improve the crystallinity and lower the impedance.

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[044] In some embodiments, a crystalline layer of LiCoO₂ with a <101> or a <003> 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 LiCoO₂ 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.

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[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.

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[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.

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[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

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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 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.

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[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 LiCoO₂ 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 LiCoO₂ for deposition of LiCoO₂ 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.

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[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 LiCoO₂ 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 LiCoO₂ layer 303. Also according to some embodiments, LiCoO₂ 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 LiCoO₂ 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

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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 ($\text{Li}_x PO_y N_{z_3}$) is deposited over LiCoO₂ layer 303. LiPON layer 304 is the electrolyte for battery 300 while LiCoO₂ 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 LiCoO₂ layer 303. Li transport through bulk cathode LiCoO₂ 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

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structure, $LiCoO_2$ layer 303 should include crystals oriented in the <101> direction or <003> direction.

[065] In accordance with the present invention, $LiCoO_2$ 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 kQ can be utilized with dc-sputtering.

[066] In some embodiments, $LiCoO_2$ 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.

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[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 μ m/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 k Ω over the same distance by the same measurement technique or higher would not allow for such a high sputter efficiency or high rate of

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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 µm/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 μ 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 μ 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 μ m/hr of the example shown in Figure 5B to 0.67 μ m/hr of the example shown in Figure 5B to 0.67 μ 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/O_2 ratio was increased. As is shown in Figure 5D, increasing the Ar/O_2 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 μ m/hr from a rate of 0.67 μ m/hr illustrated in Figure 5C.

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[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 μ m/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 μ m/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 $LiCoO_2$ 602 deposited on a thin substrate 601 according to some embodiments of the present invention. Higher lithium-ion mobilities can be achieved utilizing crystalline $LiCoO_2$ 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 703.

[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 μ m 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 LiCoO₂ 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 LiCoO₂ 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

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by a LiCoO₂ 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 LiCoO₂ 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 LiCoO₂ 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 LiCoO₂ layer 602 is then deposited on conducting layer 603. LiCoO₂ 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, LiCoO2 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 LiCoO2 layer 602 is formed over LiPON layer 901 and finally another metal layer 603 is formed over LiCoO2 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 arrangment of battery stacking structure can be indicated as Current collector/LiCoO2/LiPON/Anode/current collector/Anode/LiPON/LiCoO2/current collector/LiCoO2 . . . /current collector. Figure 9B illustrates an alternative stacking corresponding to the battery structure current collector/LiCoO2/LiPON/anode/current collector/LiCoO2/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.

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[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 LiCoO₂ deposited as discussed above. The XRD analysis of the asdeposited layer demonstrates a shallow peak at $2\theta = 18.85^{\circ}$ denoting a <003> orientation of crystalline LiCoO₂, a sharper peak at about $2\theta = 38.07^{\circ}$ corresponding with the desired <101> crystallographic direction, and a peak at $2\theta = 40.57^{\circ}$ corresponding to the <111> direction of iridium. However, the position of the <101> LiCoO₂ peak indicates that the <101> LiCoO₂ peak is nonstoichiometric LiCoO₂. In order to be useful as a battery layer, stoichiometric LiCoO₂ provides for the best Li transport. One of ordinary skill in the art will notice that careful adjustment of deposition parameters can provide stoichiometric LiCoO₂ 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 <003> LiCoO₂ grows, indicating crystallization of LiCoO₂ into the <003> direction. Further, the <101> peak of LiCoO₂ shifts slightly to $2\theta = 38.53^\circ$, indicating a more stoichiometric crystallization of the <101> LiCoO₂. However, the crystalline LiCoO₂ 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 LiCoO₂ 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.

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[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 cyrstal 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° 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

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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, LiCoO₂ 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 freestanding battery of thickness about 5 μ m 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.

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[0102] Figures 12A through 12L illustrate the crystallinity of as-grown and post anneal LiCoO₂ 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.

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[0103] Figure 12A illustrates an XRD analysis of the as-deposited LiCoO₂ film on Al_2O_3 substrate (Example 32 in Table I). A broad <003> crystalline LiCoO₂ peak is observed. The remaining peaks in the analysis, which are not labeled in Figure 12A, result from the Al_2O_3 substrate. The <003> peak is characteristic of the layered structure in the as-deposited crystalline LiCoO₂ film according to embodiments of the present invention.

[0104] Figure 12B illustrates the crystallinity of the LiCoO2 film shown in Figure 12A after a 2 hr 700 °C anneal. As shown in Figure 12B, the <003> 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 <012> and <006> crystallinity peaks.

[0105] Figure 12C through 12F show SEM photos of the granularity of the asdeposited 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.

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[0107] Figures 13A through 13J illustrate rapid thermal anneal processes applied to a LiCoO₂ layer as in Example 49 of Table I. In that example, LiCoO₂ is deposited on alumina with a 2 kW pulsed DC power with no bias. Argon flow as set to 60 seem and oxygen flow was set to 20 seem. 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-thermalanneal (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 LiCoO₂ film after the RTA illustrated in Figure 13A. Figures 13E and 13F show the granularity of the LiCoO₂ 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, LiCoO2 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

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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 $LiCoO_2$ films according to embodiments of the present invention. The $LiCoO_2$ film in the battery profiled in Figure 17 was deposited according to Example 54 in Table I. The $LiCoO_2$ film was deposited on an alumina substrate with a gold current collector. The $LiCoO_2$ 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

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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/cm^2 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.

Example #	Target Power (kW)	Bias Power (W)	Reverse Time (µs)	Frequency (kHz)	Ar (sccm)	O ₂ (sccm)	Initial Substrate Temperature (temperature during deposit) (°C)	Deposition Time (sec)	Film Thickness (µm)
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	

TABLE I

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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

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4	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]	2 0 [°]	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	LiCoO2	rhombohedral	strong [101]	2.370(1)	37.92	~1700
PDF	LiCoO ₂	Rhombohedral	random	2.408(1)	37.31	

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WHAT IS CLAIMED IS:

1. A method of depositing a LiCoO₂ layer, comprising:

placing a substrate in a reactor;

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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 μ m per hour.

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13. The method of claim 1 wherein the target is a ceramic LiCoO₂ sputter target with a resistance measured across about 4 cm of surface of less than about 500 k Ω .

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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 LiCoO₂ layer deposited over a low-temperature substrate.

22. The structure of claim 21, further including a conducting layer deposited between

the crystalline LiCoO₂ 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

LiCoO₂ 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:

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one or more battery stacks deposited on a thin substrate, wherein each battery stack comprises:

a conducting layer,

a LiCoO₂ layer deposited as a crystalline layer over the conducting layer,

a LiPON layer deposited over the crystalline LiCoO₂ 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.

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36. A method of producing a battery, comprising:

loading a substrate into a cluster tool;

depositing a crystalline LiCoO₂ layer over a conducting layer in a chamber of the cluster tool with a pulsed-dc PVD process.

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37. The method of claim 36, wherein depositing a crystalline $LiCoO_2$ layer includes

depositing crystalline LiCoO₂ 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 LiCoO₂ 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

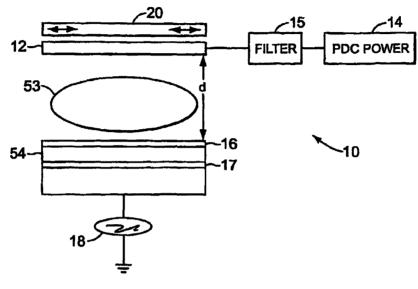
the thin substrate is held when the top portion is attached to the bottom

portion.

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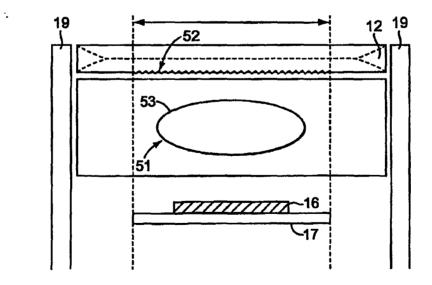
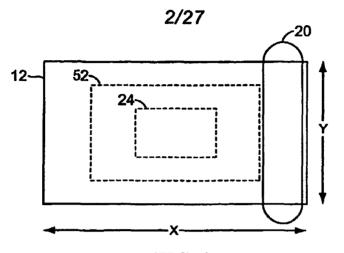


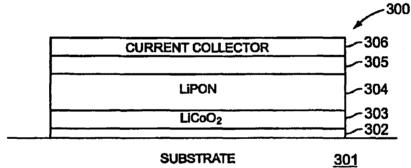
FIG. 1B

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LiCoO ₂	
SUBSTRATE	

FIG. 6A

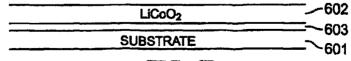


FIG. 6B

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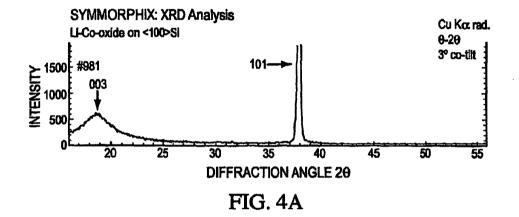
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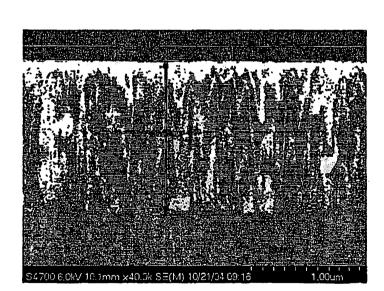


FIG. 4B

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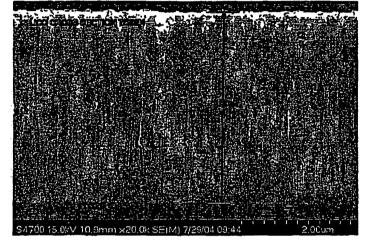


FIG. 5A

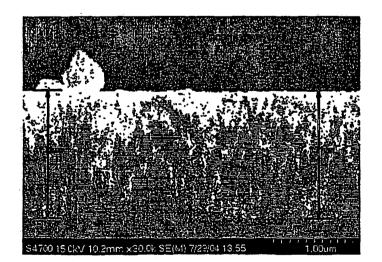


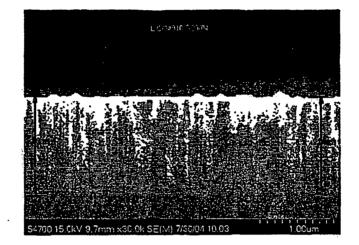
FIG. 5B

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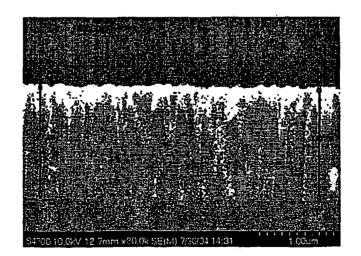


FIG. 5D

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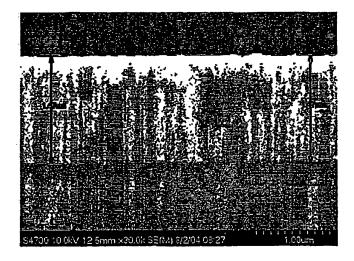


FIG. 5E

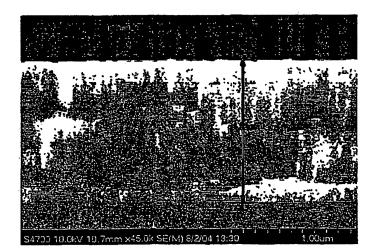
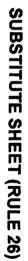
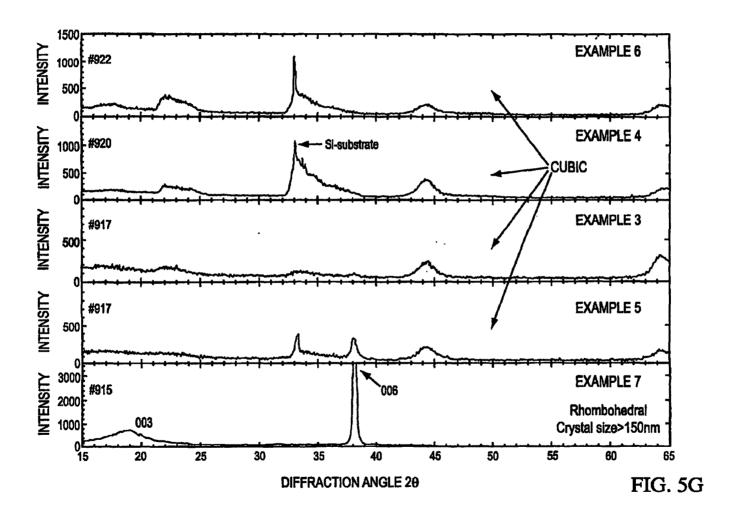


FIG. 5F

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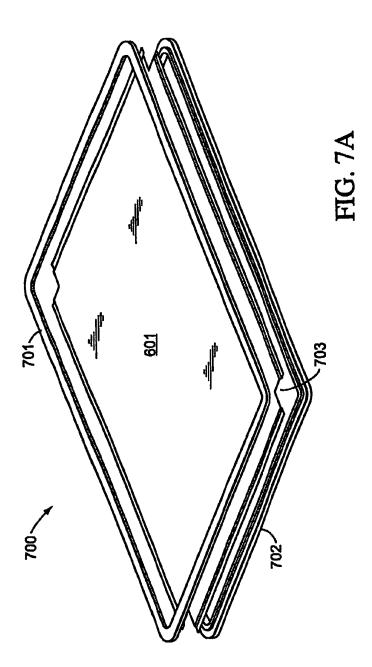
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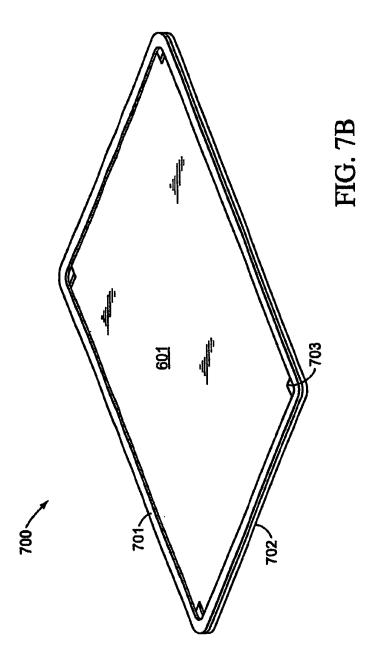
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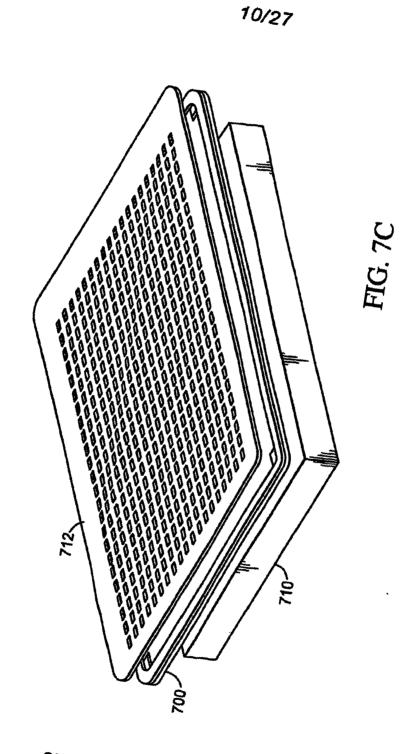
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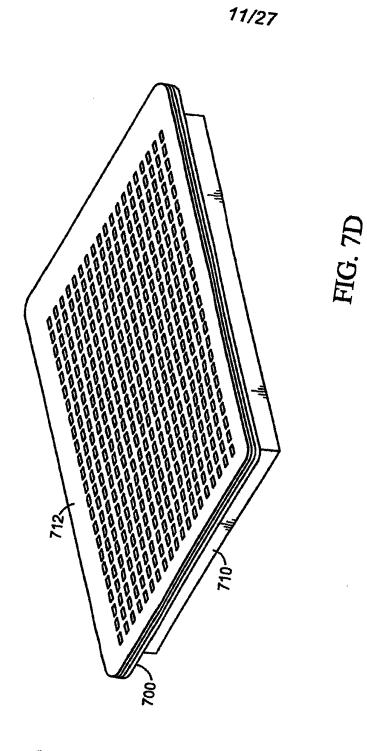
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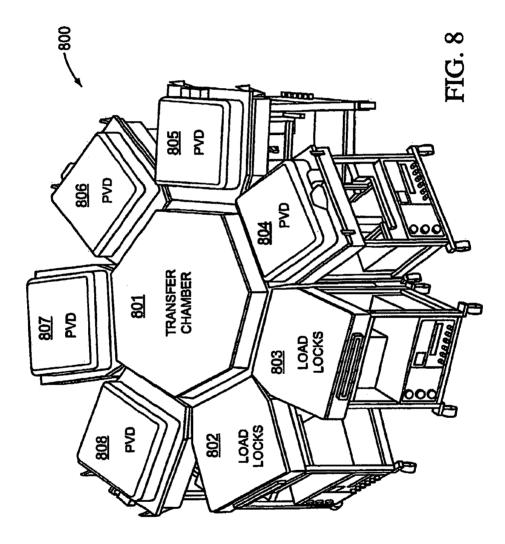
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CURRENT COLLECTOR	
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CURRENT COLLECTOR	
	602
LIPON	
ANODE	
CURRENT COLLECTOR	
ANODE	
LiPON LiCoO ₂	
CURRENT COLLECTOR	
SUBSTRATE	

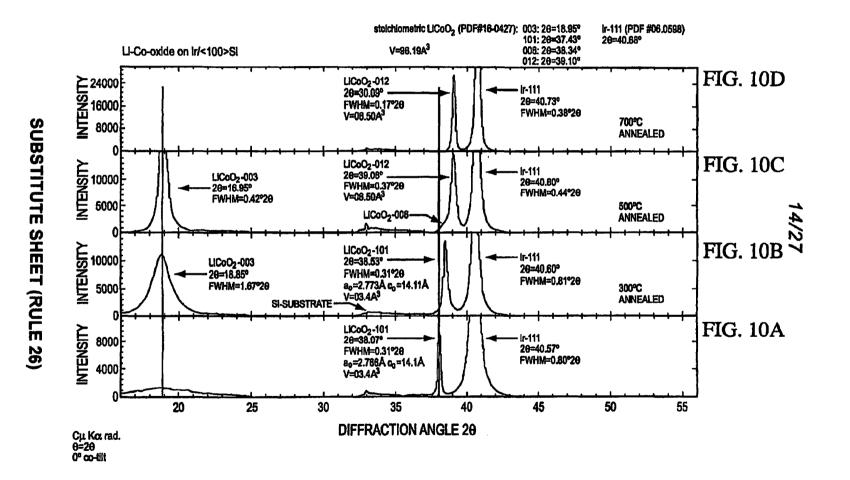
FIG. 9A

CURRENT COLLECTOR	
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CURRENT COLLECTOR	
ANODE	902
Lipon	-607
LiCoO2	
CURRENT COLLECTOR	903
ANODE	
Lipon	901
LiCoOz	602
CURRENT COLLECTOR	
SUBSTRATE	

FIG. 9B

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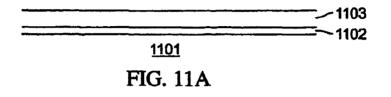
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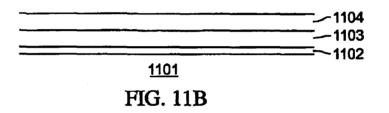
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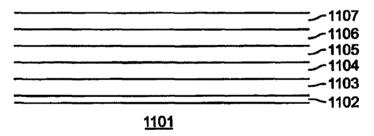
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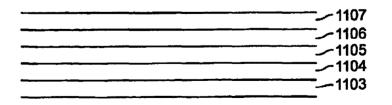


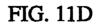












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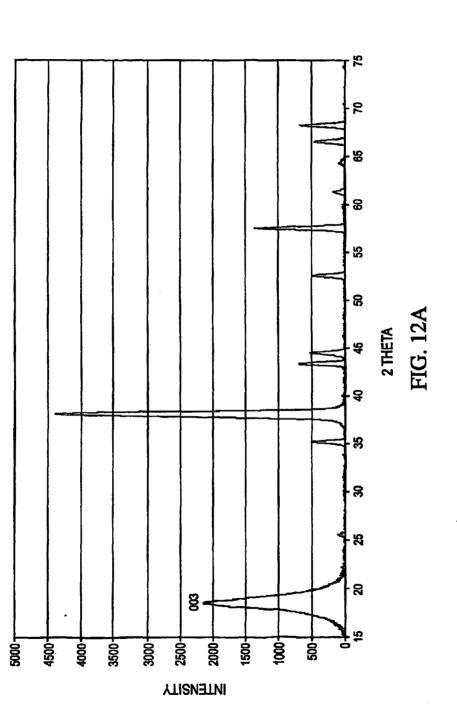
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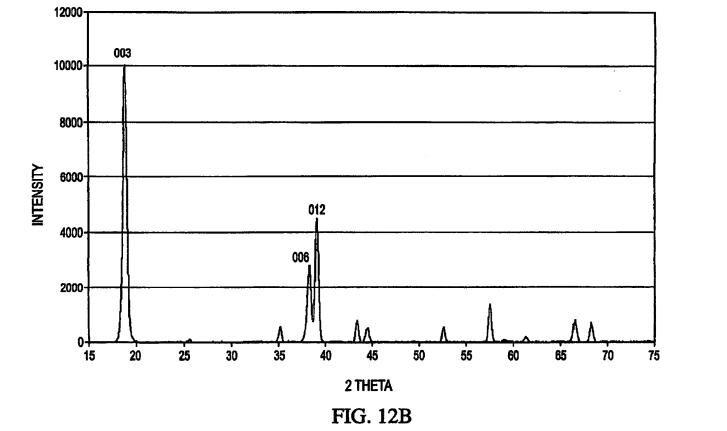
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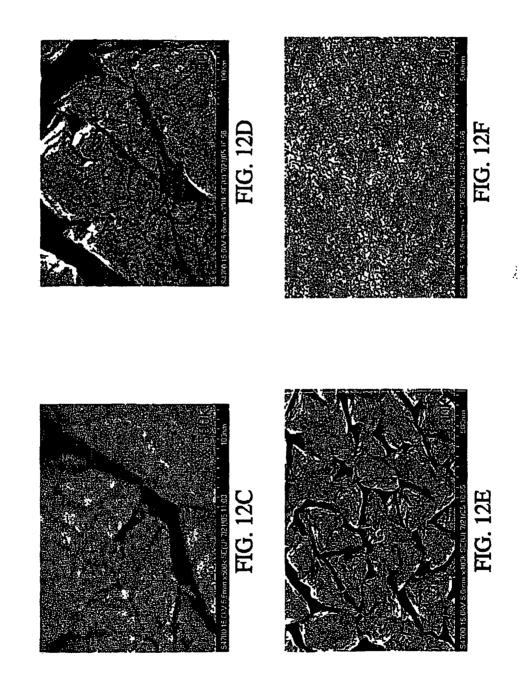
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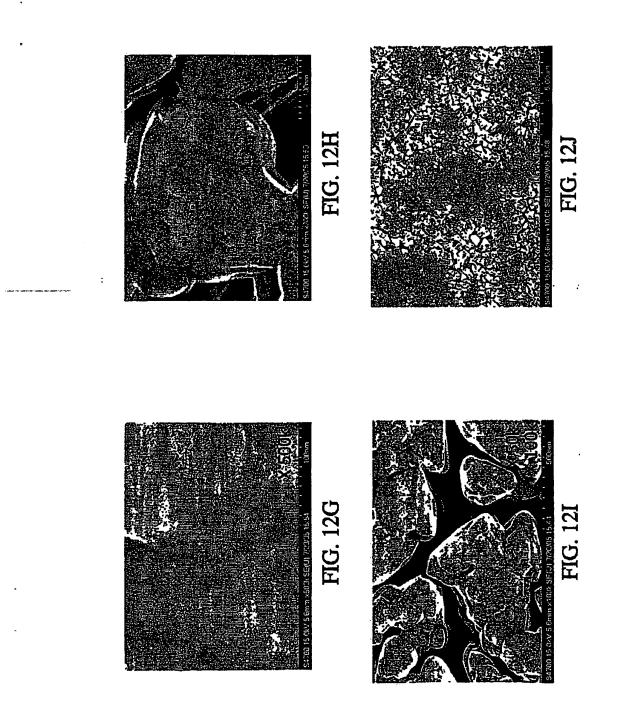
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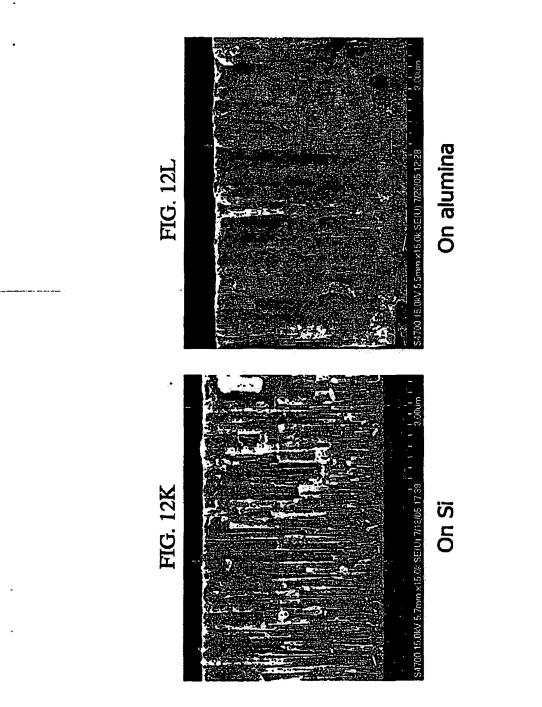




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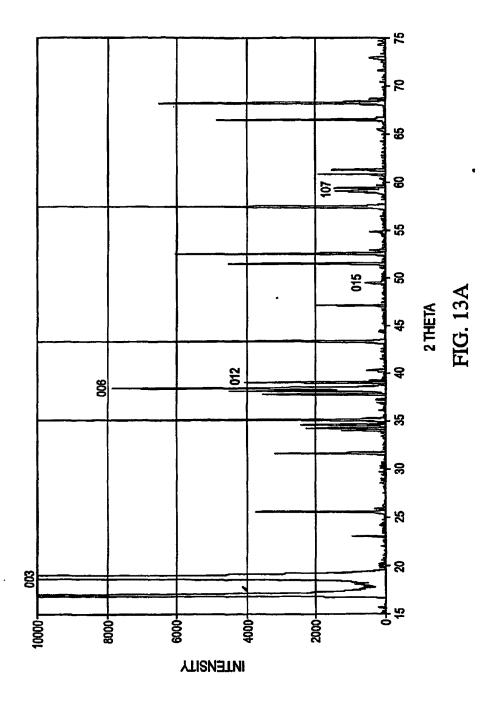
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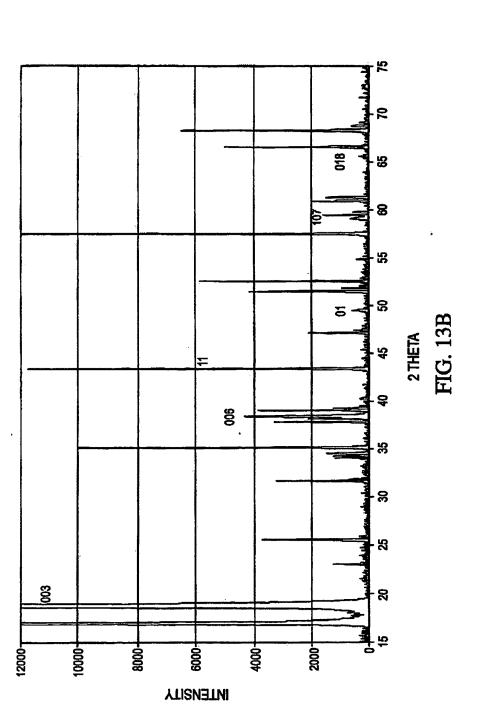


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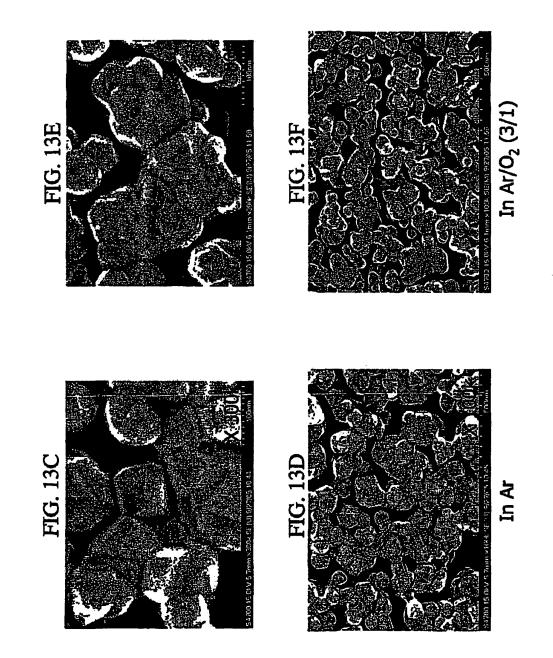
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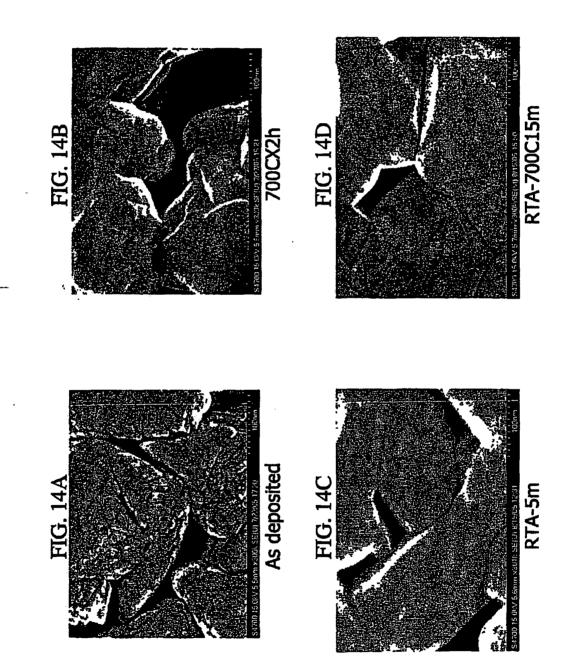




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FIG. 15A

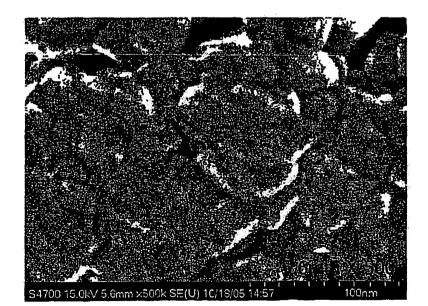


FIG. 15B

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FIG. 16

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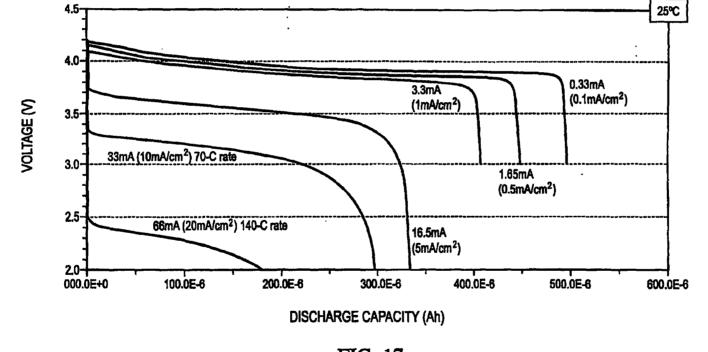


FIG. 17

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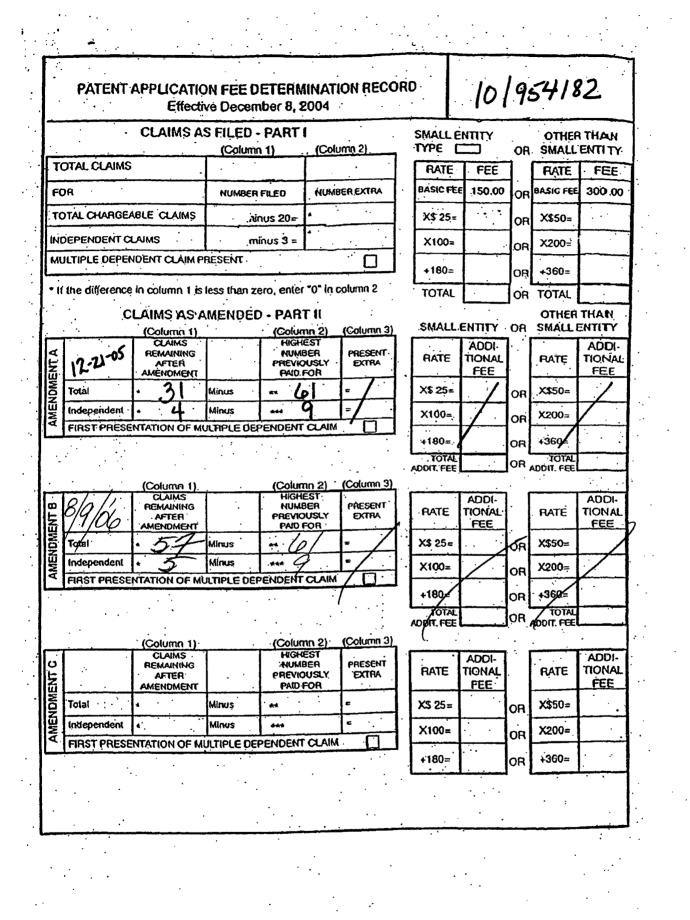
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	ED STATES PATENT	T AND TRADEMARK OFFICE	UNITED STATES DEPAR United States Patent and Address: COMMISSIONER F P.O. Box 1450 Alexandria, Virginia 22: www.uspto.gov	Trademark Office OR PATENTS
APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/954,182	10/01/2004	Hongmei Zhang	09140-0016-01000	9873
22852 7.	590 10/31/2006		EXAM	INER
FINNEGAN, LLP	HENDERSON, FAR	ABOW, GARRETT & DUNNER	ESTRADA,	MICHELLE
	RK AVENUE, NW		ART UNIT	PAPER NUMBER
	N, DC 20001-4413	X	2823	
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DATE MAILED: 10/31/2006

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

PTO-90C (Rev. 10/03)

	Application No.	Applicant(s)
	10/954,182	ZHANG ET AL.
Office Action Summary	Examiner	Art Unit
	Michelle Estrada	2823
The MAILING DATE of this communication priod for Reply	n appears on the cover sheet w	ith the correspondence address
A SHORTENED STATUTORY PERIOD FOR RE WHICHEVER IS LONGER, FROM THE MAILIN - Extensions of time may be available under the provisions of 37 CF after SIX (6) MONTHS from the mailing date of this communication - If NO period for reply is specified above, the maximum statutory pr - Failure to reply within the set or extended period for reply will, by s Any reply received by the Office later than three months after the r earned patent term adjustment. See 37 CFR 1.704(b).	G DATE OF THIS COMMUNI FR 1.136(a). In no event, however, may a n. eriod will apply and will expire SIX (6) MOI statute, cause the application to become Al	CATION. reply be timely filed NTHS from the mailing date of this communication. BANDONED (35 U.S.C. § 133).
atus		
 1) Responsive to communication(s) filed on <u>(</u> 2a) This action is FINAL. 2b) 3) Since this application is in condition for all closed in accordance with the practice und 	This action is non-final. owance except for formal mat	
sposition of Claims		
 4) Claim(s) <u>41-43,45-62,85 and 87-89</u> is/are 4a) Of the above claim(s) is/are with 5) Claim(s) <u>61,62 and 85</u> is/are allowed. 6) Claim(s) <u>41-43,45-60 and 87-89</u> is/are rejected to. 7) Claim(s) is/are objected to. 8) Claim(s) are subject to restriction and an expectation of the second sec	ndrawn from consideration.	
plication Papers		
9) The specification is objected to by the Exar	niner.	
10) The drawing(s) filed on is/are: a)		by the Examiner.
Applicant may not request that any objection to	the drawing(s) be held in abeya	nce. See 37 CFR 1.85(a).
Replacement drawing sheet(s) including the co 11) The oath or declaration is objected to by th		
iority under 35 U.S.C. § 119		
 12) Acknowledgment is made of a claim for for a) All b) Some * c) None of: 1. Certified copies of the priority documents 		§ 119(a)-(d) or (f).
2. Certified copies of the priority docum		
3. Copies of the certified copies of the		received in this National Stage
application from the International Bu * See the attached detailed Office action for a		received
achment(s)		
 Notice of References Cited (PTO-892) Notice of Draftsperson's Patent Drawing Review (PTO-948 		Summary (PTO-413) s)/Mail Date
☐ Notice of Draftsperson's Patent Drawing Review (P10-948) ☑ Information Disclosure Statement(s) (PTO/SB/08)		nformal Patent Application

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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 negatived 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 bandrejection) 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).

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 $N_{\rm 2}$ or $NH_{\rm 3}.$

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).

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

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 Woodruf*, 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 Page 8

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	2	NO.	Number-Kind C	Code ² (If known)	MM-DD-YY		Applicant of Cited Docu			igures Appear
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TAK			US 2004/00435	557 A1	03/04/2004		Haukka et al.			

Examiner Signature

Date Considered

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IDS Form PTO/SI	B/08: Substitute for for	n 1449A/PTO		C	Complete if Known				
-				Application Number	10/954,182				
INFO	ORMATION D	ISCLOSU	IRE	Filing Date	October 1, 2004				
	TEMENT BY			First Named Inventor	ZHANG, Hongmei				
514	TEMENID	AFFLICA		Art Unit	2823				
	(Use as many sheets	as necessary)		Exeminer Name	ESTRADA, Michelle				
Sheet	2	of	3	Attorney Docket Number	9140.0016-01				

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Vol	US 2006/0071592 A1	04/06/2006	Narasimhan et al.	
My	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.

		I OKI		DOCUMENTS	T	
Examiner Initials	Cite No. ¹	Foreign Patent Document Country Code ³ Number ⁴ Kind Code ⁵ (if known)	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	Translation
MM		DE 37 38 738 C1	01/26/1989	Degussa AG		
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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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ha		TOMASZEWSKI, H. et al., "Yttria-stabilized zirconia thin films grown by reactive r.f. magnetron sputtering," <i>Thin Solid Films 287</i> :104-109 (1996).										

Examiner Signature

Date Considered 2n

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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				Application Number	10/954,182	
INF	ORMATION D	ISCLOS	IRF	Filing Date	October 1, 2004	
	TEMENT BY			First Named Inventor	ZHANG, Hongmei	
317				Art Unit	2823	
	(Use as many sheets	as necessary)		Examiner Name	ESTRADA, Michelle	
Sheet	3	of	3	Attorney Docket Number	9140.0016-01	

FRAG T	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).
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	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).
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	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).
IM	Specification as filed September 2, 2005, for U.S. Appl. No. 11/218,652 (Atty. Docket No. 09140.0052-00)

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12-08-06



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 D SPUTTERING OF	, , ,	

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

Sir:

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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 $\frac{59, 60, 61}{0}$ 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

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wherein a material is deposited on the substrate, <u>The method of claims 61 or 85</u> 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.

-3-

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 metallic conductive target and a substrate;

providing pulsed DC power to the target;

providing a magnetic field to the target; and

reconditioning a metallic 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).

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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 ("<u>Smolonoff et al</u>.") in view of U.S. Patent No. 6,673,716 ("<u>D'Couto et al.</u>").

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 ("<u>Chen et</u> <u>al.</u>").

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 ("<u>Milonopoulou et al.</u>").

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.

Reg. No. 56,170 By:

Gary J. Edwards Reg. No. 41,008

Date: December 6, 2006

EXPRESS MAIL LABEL NO. EV 746096154 US

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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 A	Application of:)	
ZHAN	IG, Hongmei et al.)) Group Art Unit: 2823	
Applic	cation No.: 10/954,182)) Examiner: ESTRADA, Miche	lle
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.

Date: December 6, 2006

Gary J. Edwards Reg. No. 41,008

Bv:

EXPRESS MAIL LABEL NO. EV 746096154 US

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IDS Form PTO/S	B/08: Substitute for for	m 1449A/PTO DI	EC U 6 2006	C C	omplete if Known
		13		Application Number	10/954,182
INFO	ORMATION D ATEMENT BY	NSCLOST	IRE .	Filing Date	October 1, 2004
STA	TEMENT BY	APPIICA	BADEMARIA	First Named Inventor	ZHANG, Hongmei
				Art Unit	2823
	(Use as many sheets	as necessary)		Examiner Name	ESTRADA, Michelle
Sheet	1	of	3	Attorney Docket Number	9140.0016-01

		U.S. PATENTS	AND PUBLISH	ED U.S. PATENT APPLICAT	TIONS
Examiner Initials	Cite	Document Number Issue or		Name of Patentee or	Pages, Columns, Lines, Where
	No.'	Number-Kind Code ² (if known)	Publication Date MM-DD-YYYY	Applicant of Cited Document	Relevant Passages or Relevant Figures Appear
		US-6,088,492	07-11-2000	Kaneko et al.	
		US-6,288,835 B1	09-11-2001	Nilsson et al.	
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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 Country Code ³ Number ⁴ Kind Code ⁵ (<i>if known</i>)	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			
		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	Date	
Signature	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.

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

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IDS Form PTO/S	B/08: Substitute for for	m 1449A/PTO		Complete if Known		
				Application Number	10/954,182	
INFO	DRMATION D	ISCLOSU	IRF	Filing Date	October 1, 2004	
				First Named Inventor	ZHANG, Hongmei	
	STATEMENT BY APPLICANT			Art Unit	2823	
	(Use as many sheets as necessary)			Examiner Name	ESTRADA, Michelle	
Sheet 3 of 3			3	Attorney Docket Number	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).	
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PCT International Search Report and Written Opinion for Application No. PCT/US05/44781 dated October 3, 2006 (Attorney Docket No. 9140.0042- 00304).	

Examiner	Date	
Signature	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.

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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	II ISTE	Application Number	Kind	Date	Update	Туре
KR 2003088236	Α	20031119	KR 200226187	Α	20020513	200421	B
KR 507142	В	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 No	tes		
KR 2003088236	A	ко	1	. 10		*		
KR 507142	B	ко			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

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(74) 대리인 한양특허법인

심사관 : 방승훈

(54) 방열과 강성 강화용 플라이 될

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 발생되는 마찰열의 방열성도 향상시 켜 균열을 방지함에 그 목적이 있다.

상기와 같은 목적을 달성하기 위한 본 발명은, 플라이휠(10)이 링기어(11)가 외주 측에 압입되면서 크랭크축(5)에 결합 되는 지지플레이트(10a)와, 이 지지플레이트(10a)에 일단이 고정되면서 절곡된 내부로 수용공간을 형성하는 커버플레이 트(10b), 이 커버플레이트(10b)의 안쪽에서 고정되는 인서트플레이트(10c) 및 이 인서트플레이트(10c)에 접촉됨과 더불 어 커버플레이트(10b)내에 삽입·고정되어 클러치 조작에 따라 클러치플레이트(22)로부터 클램핑 로드(F')를 받는 마찰플 레이트(10d)로 이루어진 것을 특징으로 한다.

대표도

도 4

명세서

도면의 간단한 설명

도 1은 일반적인 엔진의 구성도

도 2는 중래에 따른 플라이 휠부위의 구성도

도 3은 중래에 따른 플라이휠의 구성 단면도

-1-

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 : 갭

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 방 지하도록 강성을 강화함과 더불어 클러치와의 마찰에 의한 마찰열의 방열성도 향상할 수 있도록 된 방열과 강성 강화용 플 라이휠에 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 회전 력을 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 클러치폐달에 의해 플라이휠 (10)과 마찰되는 클러치플레이트(22)를 감싸는 클러치커버(21)로 이루어져 엔진과 변속기사이의 회전 전달을 분리·연결 하는 클러치어셈블리(20)가 구비되어진다.

이러한 플라이횔(10)은 엔진의 4행정 중에서 폭발행정에서 얻어진 토크(Torque)를 흡입·압축·배기 행정에 고르게 분배 하여 승차감 및 차량 출발성에 맞춘 형상으로 설계됨은 물론 엔진과 변속기를 연결하는 역할로 클러치의 용량에 맞게 설계 됨은 물론이다.

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그러나, 이와 같은 플라이휠(10)은 도 3에 도시된 바와 같이, 크랭크축(5)에 보을트등을 매개로 고정된 상태에서 엔진의 회전력을 변속기로 전달하기 위해 고속으로 회전하고, 또한 클러치의 조작에 따라 클러치플레이트(22)와 서로 강하게 마 찰되어 엔진의 회전력을 단속하는데 이로 인해, 포트(Port)형 클러치인 경우에는 클러치플레이트(22)의 취부면과 플라이 휠(10)좌면 사이의 노치(C: Notch)부위가 회전에 따른 버스트 포스(Burst Force)에 의해 균열(Crack)이 발생될 소지가 있는 문제가 있게 된다.

또한, 트럭과 버스와 같이 대형 디젤엔진을 사용하는 경우에는 플라이휠(10)의 형상도 이너시아(Inertia)값이 매우 큰 형 상을 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 발 생시킬 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 균 열을 방지함에 그 목적이 있다.

상기와 같은 목적을 달성하기 위한 본 발명은, 플라이 휠이 초기 시동시 스타트 모터에 의해 회전되는 링기어를 외주면으 로 압입 고정하면서 엔진의 초기 구동력 발생하는 크랭크축으로 링기어의 회전력을 전달하도록 크랭크축에 결합된 지지플 레이트와,

상기 지지플레이트에 고정되면서 지지플레이트로부터 일정간격을 두도록 절곡되어 그 내부로 수용공간을 형성하는 커버 플레이트,

상기 커버플레이트의 내부 수용공간인 안쪽에서 고정되는 인서트플레이트 및

상기 커버플레이트내에서 인서트플레이트에 접촉되도록 삽입·고정되어 클러치 조작 시 클러치어셈블리의 클러치플레이트 에 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 플라리휠의 구성 단면도를 도시한 것인바, 본 발명은 피스톤(2)의 왕복 운동을 회전운동으로 변환 하는 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 더 불어 보을트등의 고정부재를 통해 크랭크축(5)에 결합되는 지지플레이트(10a)와, 이 지지플레이트(10a)에 보을트등의 고 정부재를 통해 고정된 일단으로부터 서로 간격을 두도록 절곡되어 그 내부로 수용공간을 형성하는 컵 형상의 커버플레이 트(10b), 이 커버플레이트(10b)의 안쪽에서 보옵트등의 고정부재를 통해 고정되는 인서트플레이트(10c) 및 이 인서트플 레이트(10c)에 접촉됨과 더불어 커버플레이트(10b)내에 삽입·고정되어 클러치 조작 시 클러치어셈블리(20)의 클러치플레 이트(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 퓰라이휠(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 의 해 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 테 두리를 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 보올트등의 고정부재를 이용해 인서트플레이트(10c)와 마찰플레이트(10d) 순차적으로 고정한 후, 상기 커버플레이트(10b)와 지지플레이트(10a)를 보올트등의 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 플라이뵐(10)에 클러치어셈블리(20)를 결합하여 클러치 조작시 클러치플레이트(22)에 의해 플라이뵐(10)의 마찰플레이트(10d)쪽으로 강하게 작용하는 클램핑로드(F')를 통해 엔진과 변속기사이의 회전력을 단속하게 된다.

이때, 본 발명의 플라이횥(10)은 뮬러치의 조작시 엔진의 회전에 의해 플라이횥(10)에 발생되는 버스트 포스(F)가 강재 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 플라이횔(10)은 클러치의 빈번한 조작에 의한 클램핑로드(F')에 의해 클러치플레이트(22)와 마찰플레이 트(10d)사이에서 마찰열이 발생되면, 상기 마찰플레이트(10d)에 접촉된 인서트플레이트(10c)로 전달됨과 더불어 마찰플 레이트(10d)의 측면 테두리를 따라 형성된 다수의 슬랏(10d')을 통해 보다 빠른 열의 방출이 이루어지게 된다.

이때, 상기 인서트플레이트(10c)로 전달된 마찰열이 방열되는 것보다 적충되는 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 클러 치 조작에 따라 클러치플레이트로부터 클램핑 로드를 받는 부분으로 분리 제작한 후 서로 조립·결합시켜, 플라이휠이 적용 되는 차종에 관계없이 플라이휠에서 발생되는 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 크랭 크축(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 형 성하는 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 힅.

청구항 2.

제 1항에 있어서, 상기 지지플레이트(10a)와 인서트플레이트(10c)는 주철로 이루어지고, 상기 커버플레이트(10b)는 탄 성을 갖는 강재질로 이루진 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.

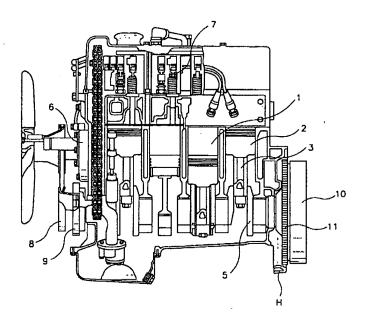
삭제

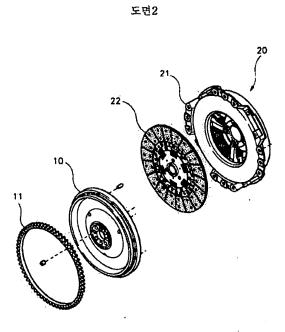
청구항 5.

제 1항에 있어서, 상기 마찰플레이트(10d)는 클러치 조작에 따른 클러치플레이트(22)와의 마찰열을 외부로 발산하도록 그 측면 테두리를 따라 다수의 슬랏(10d')이 형성되어진 것을 특징으로 하는 방열과 강성 강화용 플라이 휠.

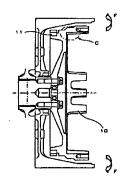
도면1

도면





도면3

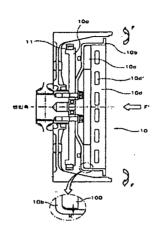


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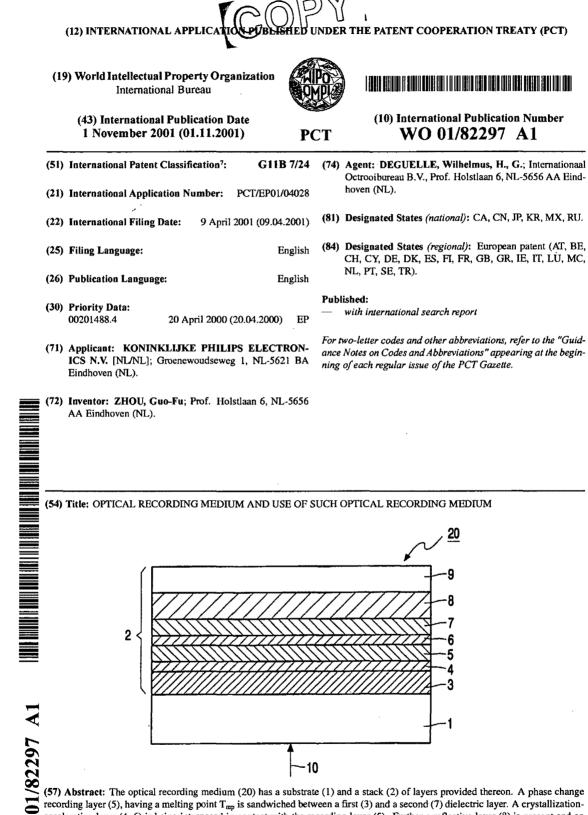
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(57) Abstract: The optical recording medium (20) has a substrate (1) and a stack (2) of layers provided thereon. A phase change recording layer (5), having a melting point T_{mp} is sandwiched between a first (3) and a second (7) dielectric layer. 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).

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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.

WO 01/82297

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

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- 5 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.
- 10 This causes a high noise level. A high crystallization speed is particularly required in highdensity 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
- 15 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 Sb₂Te₃ layer as crystallization accelerating layer, a Sb layer as a composition correcting layer, a recording layer of a phase-change Sb₇₂Te₂₈ alloy, a second dielectric layer and a metal

- 25 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 Sb₂Te₃, has been arranged between the first dielectric layer and the correction and the recording layer to
- 30 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

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cycles, whereafter the crystallization accelerating layer N is no longer present and its crystallization-accelerating action is lost.

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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 nucleationpromoting 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

10 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 10³ 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, semiconductors elements and semiconductor alloys and

- has a melting point T_{mg} at least 250°C higher than the melting point T_{mp} of the 25 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

30 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.

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- An advantage of the G-layer, comprising a material selected from the group 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 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 Glayer 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

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- 5 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
- 10

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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 $Q_a \ln_b Sb_c Te_d$ (in atomic percentages),

- 15 wherein Q is selected from the group consisting of Ag and Ge;
 - 2≤a≤9
 - 0 < b ≤ 6
 - $\cdot 55 \le c \le 80$

 $16 \le d \le 30$; 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} (in atomic percentages), wherein $0.166 \le x \le 0.444$; 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 Sb₂Te₃ in the triangular Ge-Sb-Te composition diagram and includes the stoichiometric compounds Ge₂Sb₂Te₅ (x = 4/9), GeSb₂Te₄ (x = 2/7) and GeSb₄Te₇ (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 SiO₂, e.g. (ZnS)₈₀(SiO₂)₂₀. The layers may alternatively be made of SiO₂, TiO₂, Ta₂O₅, ZnS, AlN and/or Si₃N₄. The dielectric layer through which the laser light enters the stack preferably has a thickness of 70 to (70+λ/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.

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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 10³ DOW-cycles.

As mentioned above the total thickness of the first dielectric layer is preferably 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 10 to 40 nm. Preferably the thickness of the dielectric layer adjacent to the reflective layer is

15 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.

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.

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

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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.

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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

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.

inversely proportional to the crystallization rate.

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 shortwavelength 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

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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

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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 .

Embodiments of the optical recording medium of the invention will be described with reference to the drawings.

In the drawings:

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.

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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(SiO₂)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

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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 λ=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 Ag₂Te, CrTe, Ge or Si. The melting points T_{mg} of bulk PbTe, Ag₂Te, 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 10³. 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 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.

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In application JP-09161316 A the crystallization accelerating layer N of Sb₂Te₃, 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 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 μ m. 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+/-3 μm 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.

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

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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 μ m 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. $Ge_2Sb_2Te_5$.

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 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 10³ 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

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5 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)

- 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).

An optical recording medium (20) as claimed in Claim 1 characterized in that
 the crystallization accelerating layer (4, 6) comprises a material selected from the group
 consisting of PbTe, Ag₂Te, CrTe, Ge and Si.

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
 recording layer (5) and the second dielectric layer (7).

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).

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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).

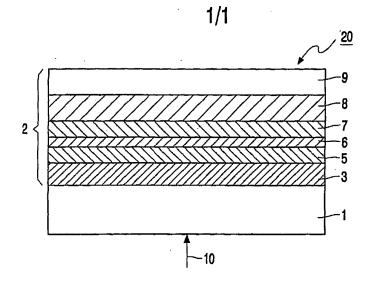
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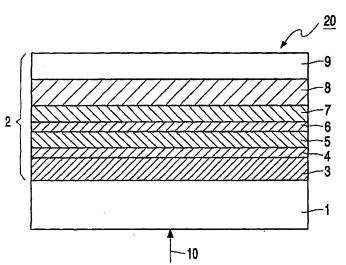
9. The use of an optical recording medium (20), which medium is claimed in any one of the preceding claims, characterized in that the linear velocity of the medium relative to a laser-light beam (10) is at least 7.2 m/s.

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FIG. 1





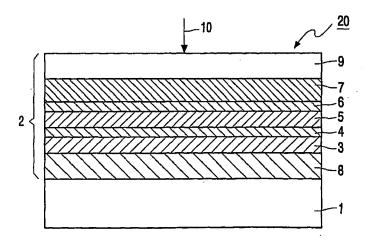


FIG. 3

FIG. 2

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INTERNATIONAL SEARCH REPORT

		PCT	/EP 01/04028			
A. CLASSI	FICATION OF SUBJECT MATTER G11B7/24					
	b International Patent Classification (IPC) or to both national classific SEARCHED	ation and IPC				
	cumentation searched (classification system followed by classificat	ion symbols)				
IPC 7	G11B					
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C. DOCUM	ENTS CONSIDERED TO BE RELEVANT					
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A" document defining the general state of the art which is not cited to understand the principle or theory underlying the						
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page 1 of 2

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INTERNATIONAL SEARCH REPORT

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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 A	Application of:)	
ZHAN	IG, Hongmei et al.)	Group Art Unit: 2823
Applic	cation No.: 10/954,182)	Examiner: ESTRADA, Michelle
Filed:	October 1, 2004)	Confirmation No. 6072
For:	BIASED PULSE DC REACTIVE SPUTTERING OF OXIDE FILMS)	Confirmation No.: 9873

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

Sir:

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<u>SEVENTH SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT</u> <u>UNDER 37 C.F.R. § 1.97(c)</u>

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.

03/07/2007 DEMMANU1 00000038 060916 10954182 01 FC:1806 180.00 DA

Page 848 of 1053

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

EXPRESS MAIL LABEL NO. EV 977728687 US

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IDS Form PTO/S	SB/08: Substitute for for	n 1449A/PTQ	MAR 0 1 200	ببر	C	omplete if Known
		1	.	Anp	lication Number	10/954,182
INF	ORMATION D	ISCLOSŮ	RE	Filin	ig Date	October 1, 2004
I STA	TEMENT BY		NTRADE	Firs	t Named Inventor	ZHANG, Hongmei
317		AFFLICA		Art	Unit	2823
	(Use as many sheets	as necessary)		Exa	miner Name	ESTRADA, Michelle
Sheet	1	of	2	Atto	rney Docket Number	9140.0016-01

	U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS								
Examiner	Cite	Document Number	Issue or	Name of Patentee or	Pages, Columns, Lines, Where				
Initials	No.'	Number-Kind Code ² (if known)	Publication Date MM-DD-YYYY	Applicant of Cited Document	Relevant Passages or Relevant Figures Appear				
		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
	Ν.,	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).	
	k.	SCHOLL, R., "Power Supplies for Pulsed Plasma Technologies: State-Of-The- Art And Outlook," Advances Energy Industries, Inc., pages 1-8 (1999).	
	1	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).	
	i	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).	
	1	Response to Office Action dated January 26, 2007, in U.S. Application No.	

		فاستبيت كربرتية ككر ميلان بيست
Examiner	Date	
Signature	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 977728687 US

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IDS Form PTO/S	B/08: Substitute for for	n 1449A/PTO		Complete if Known		
				Application Number	10/954,182	
INFO	ORMATION D	ISCLOSU	IRE	Filing Date	October 1, 2004	
	TEMENT BY			First Named Inventor	ZHANG, Hongmei	
514		AFFLICA		Art Unit	2823	
	(Use as many sheets as necessary)			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	Date	te	
Signature		nsidered	

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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	<u>ted States Paten</u>	t 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	09140-0016-01000	9873	
22852 FINNEGAN	7590 03/14/2003	, OW, GARRETT & DUNNER	EXAMINER ESTRADA, MICHELLE		
LLP	,	ow, oracler a bounder			
	RK AVENUE, NW DN, DC 20001-4413		ART UNIT	PAPER NUMBER	
			2823		
SHORTENED STATUTO	RY PERIOD OF RESPONSE	MAIL DATE	DELIVER	Y MODE	
3 M(ONTHS	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.

PTOL-90A (Rev. 10/06)

Page 852 of 1053

	Application No.	Applicant(s)
	10/954,182	ZHANG ET AL.
Office Action Summary	Examiner	Art Unit
	Michelle Estrada	2823
The MAILING DATE of this communication a Period for Reply	ppears on the cover sheet w	ith the correspondence address
 A SHORTENED STATUTORY PERIOD FOR REP WHICHEVER IS LONGER, FROM THE MAILING Extensions of time may be available under the provisions of 37 CFR - after SIX (6) MONTHS from the mailing date of this communication. If NO period for reply is specified above, the maximum statutory period Failure to reply within the set or extended period for reply will, by state Any reply received by the Office later than three months after the mai earned patent term adjustment. See 37 CFR 1.704(b). 	DATE OF THIS COMMUNI 1.136(a). In no event, however, may a pd will apply and will expire SIX (6) MON ute, cause the application to become Al	CATION. reply be timely filed ITHS from the mailing date of this communication. BANDONED (35 U.S.C. § 133).
Status		
1) Responsive to communication(s) filed on $\underline{06}$	December 2006	
	his action is non-final.	
3) Since this application is in condition for allow		ers, prosecution as to the merits is
closed in accordance with the practice under	•	•
Disposition of Claims		
4)⊠ Claim(s) <u>41-58,61,62,85 and 87-89</u> is/are pe	nding in the application	
4a) Of the above claim(s) is/are withdr		
5)⊠ Claim(s) <u>85 and 87-89</u> is/are allowed.		, ,
6)⊠ Claim(s) <u>61</u> is/are rejected.		•
7) Claim(s) <u>41-58 and 61</u> 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 Examination 10) The drawing(s) filed on is/are: a) and acceleration 		by the Eveniner
		•
Applicant may not request that any objection to the	e drawing(s) be held in abeyar	nce. See 37 CFR 1.85(a).
Applicant may not request that any objection to the Replacement drawing sheet(s) including the corre	e drawing(s) be held in abeyar action is required if the drawing	ice. See 37 CFR 1.85(a). (s) is objected to. See 37 CFR 1.121(d)
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Applicant may not request that any objection to the Replacement drawing sheet(s) including the correct 11) The oath or declaration is objected to by the B Priority under 35 U.S.C. § 119 12) Acknowledgment is made of a claim for foreign a) All b) Some * c) None of: 1. Certified copies of the priority document 2. Certified copies of the priority document 3. Copies of the certified copies of the priority document * See the attached detailed Office action for a list	e drawing(s) be held in abeyar ection is required if the drawing Examiner. Note the attached on priority under 35 U.S.C. § Ints have been received. Ints have been received in A fority documents have been au (PCT Rule 17.2(a)). st of the certified copies not	nce. See 37 CFR 1.85(a). (s) is objected to. See 37 CFR 1.121(d) d Office Action or form PTO-152. 119(a)-(d) or (f). pplication No received in this National Stage received.

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Application/Control Number: 10/954,182 Art Unit: 2823

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 negatived by the manner in which the invention was made.

Claim 61 ia 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 Application/Control Number: 10/954,182 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. Application/Control Number: 10/954,182 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).

nelle Estrada

Primary Examiner Art Unit 2823

ME March 5, 2007

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IDS Form PTO/S	B/08: Substitute for for	m 1449A/PID DI	EC U 6 2005	⁶) C	omplete if Known
		凶	- 2000	Application Number	10/954,182
	ORMATION D	NSCLOS		Filing Date	October 1, 2004
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516		AFFLICA		Art Unit	2823
	(Use as many sheets			Examiner Name	ESTRADA, Michelle
Sheet	1	of	3	Attorney Docket Number	9140.0016-01

	U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS					
Examiner	Cite	Document Number	Issue or	Name of Patentee or	Pages, Columns, Lines, Where	
Initials	No.'	Number-Kind Code ² (if known)	Publication Date MM-DD-YYYY	Applicant of Cited Document	Relevant Passages or Relevant Figures Appear	
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 Country Code ³ Number ⁴ Kind Code ⁶ (# known)	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		
ME		KR 2002-26187	04-06-2002	Hyundai Motor Co Ltd		Abstract		
ME	,	WO 01/82297 A1		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
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	

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IDS Form PTO/S	B/08: Substitute for for	m 1449A/PTO		Complete if Known		
				Application Number	10/954,182	
INFO	DRMATION D	ISCLOSU	IRE	Filing Date	October 1, 2004	
	TEMENT BY			First Named Inventor	ZHANG, Hongmei	
5.7			1.01	Art Unit	2823	
	(Use as many sheets	as necessary)		Examiner Name	ESTRADA, Michelle	
Sheet 2 of 3				Attorney Docket Number	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		Date	
Signature	/Michelle Estrada/	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/S	B/08: Substitute for for	m 1449A/PTO		Complete if Known		
				Application Number	10/954,182	
	ORMATION D	ISCLOSU	IRE	Filing Date	October 1, 2004	
				First Named Inventor	ZHANG, Hongmei	
317	STATEMENT BY APPLICANT			Art Unit	2823	_
	(Use as many sheets	as necessary)		Examiner Name	ESTRADA, Michelle	
Sheet	3	of	3	Attorney Docket Number	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 Date Considered /Michelle Estrada/ 03/02/2007

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.		
Notice of References Cited	Examiner Art Unit			
	Michelle Estrada	2823	Page 1 of 1	
U	S. PATENT DOCUMENTS			

Document Number Date * Name Classification Country Code-Number-Kind Code MM-YYYY * US-2001/0034106 10-2001 Moise et al. 438/396 Α USв US-С US-D US-Е US-F US-G USн US-T US-J USк US-L US-М

	FOREIGN PATENT DOCUMENTS					
*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Country	Name	Classification
	N					
	0					
	Р					
	Q					
	R					
	S					
	Т					

NON-PATENT DOCUMENTS

*		Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)
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*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.

U.S. Patent and Trademark Office PTO-892 (Rev. 01-2001)

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Notice of References Cited

Part of Paper No. 20070305

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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 A	Application of:)	
ZHAN	IG, Hongmei et al.) Group Art Unit: 2823	
Applic	cation No.: 10/954,182) Examiner: ESTRADA, Mi	chelle
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:

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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 N_2 , NH_3 , 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:

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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;

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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.

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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.

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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

-9-

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 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

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 A	application of:)	
ZHAN	IG, Hongmei et al.)	Group Art Unit: 2823
Applic	cation 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.

04/02/2007 EEKUBAY1 00000017 060916 10954182 02 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 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

Reg. No. 41,008

EXPRESS MAIL LABEL NO. EV 977728506 US

~	IDS Form PTO	/SB/08: Substitute for for	m 1449A/PTO		Complete if Known		
					Application Number	10/954,182	
1	INI	FORMATION F	DISCLOSU	RF	Filing Date	October 1, 2004	
17	STATEMENT BY APPLICANT				First Named Inventor	ZHANG, Hongmei	
1					Art Unit	2823	
A MAR 3	3	(Use as many sheets	as necessary)		Examiner Name	ESTRADA, Michelle	
a .	20nSheet	1	of	1	Attorney Docket Number	9140.0016-01	
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THOEMA	KOT	U.S	. PATENTS	AND PUB	LISHED U.S. PATENT A	PPLICATIONS	

Examiner	Cite	Document Number	Issue or	Name of Patentee or	Pages, Columns, Lines, Where
Initials	No."	Number-Kind Code ² (if known)	Publication Date MM-DD-YYYY	Applicant of Cited Document	Relevant Passages or Relevant Figures Appear
		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				
		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		Date	
Signature	,	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

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

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)
ZUANC Hongmoi et al)
ZHANG, Hongmei et al.) Group Art Unit: 2823
Application No.: 10/954,182) Examiner: ESTRADA, Michelle
Filed: October 1, 2004)
Flied. 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	A	dditional Fee
Total	39	-	45	0	x \$ 50	\$	0
Indep.	19	-	2	17	x \$200		3,400.00
First	Presentation of Multip	le De	p. Claim(s)	I	+\$360		0
			<u>, on an an</u>		Subtotal	\$	3,400.00
	<u> </u>		Reduction	on by ½ if sn	nall 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

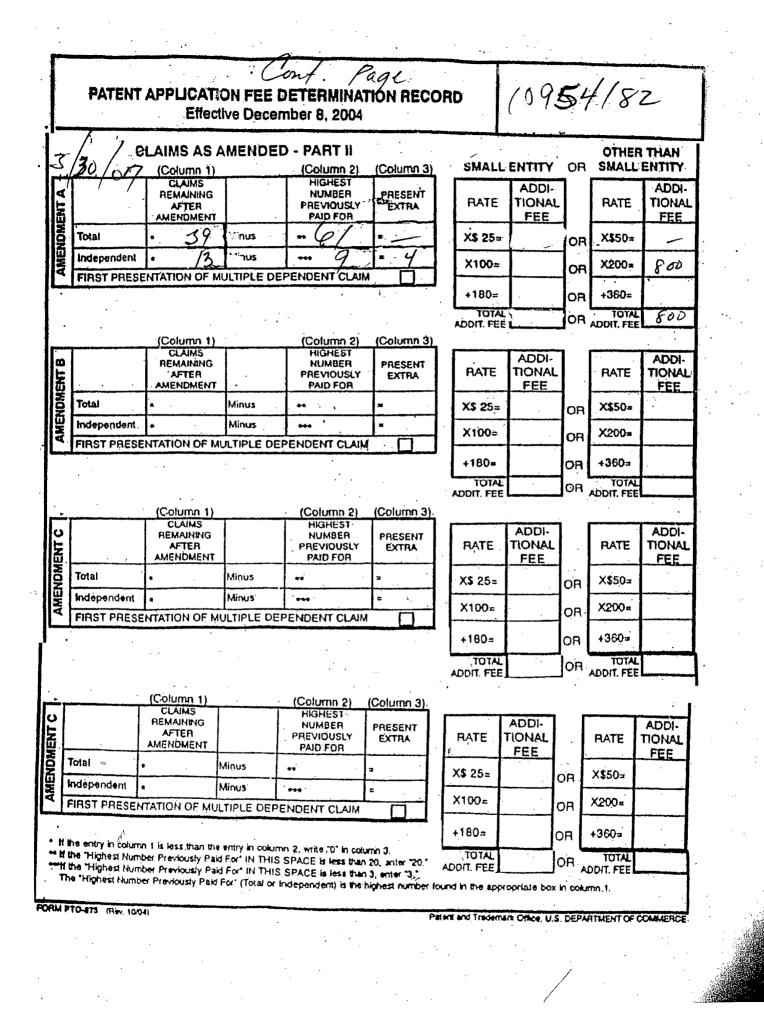
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By:

Gary J. Edwards Reg. No. 41,008

EXPRESS MAIL LABEL NO. EV 977728506 US



Page 877 of 1053

	ed States Patent a	ND TRADEMARK OFFICE	UNITED STATES DEPAR United States Patent and Address: COMMISSIONER F P.O. Box 1450 Alexandria, Virginia 222 www.uspto.gov	Trademark Office OR PATENTS	
APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.	
10/954,182	10/01/2004	Hongmei Zhang	09140-0016-01000	9873	
	7590 04/12/2007 ENDERSON, FARABOW	EXAMINER			
LLP	·	, on define bounder	ESTRADA, MICHELLE		
	K AVENUE, NW N, DC 20001-4413		ART UNIT	PAPER NUMBER	
			2823		
SHORTENED STATUTORY PERIOD OF RESPONSE MAIL DATE			DELIVER	Y MODE	
30 DAYS 04/12/2007			PAF	'ER	

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.

PTOL-90A (Rev. 10/06)

	Application No.	Applicant(s)
Notice of Non-Compliant	10954182	
Amendment (37 CFR 1.121)	Examiner	Art Unit
The MAILING DATE of this communica	ation appears on the cover sheet v	with the correspondence address
The amendment document filed on <u>30 March 20</u> requirements of 37 CFR 1.121 or 1.4. In order fo tem(s) is required.		
THE FOLLOWING MARKED (X) ITEM(S) CAUS 1. Amendments to the specification: A. Amended paragraph(s) do not B. New paragraph(s) should not C. Other	t include markings.	ENT TO BE NON-COMPLIANT:
 2. Abstract: A. Not presented on a separate s B. Other 	sheet. 37 CFR 1.72.	
"Annotated Sheet" as required B. The practice of submitting pro	d by 37 CFR 1.121(d).	Replacement Sheet," "New Sheet," or en eliminated. Replacement drawings th 37 CFR 1.84 are required.
of each claim cannot be identi number by using one of the fo	include the text of all pending cla ided with the proper status identi ified. Note: the status of every c ollowing status identifiers: (Origin), (Not entered), (Withdrawn) and	fier, and as such, the individual status claim must be indicated after its claim al), (Currently amended), (Canceled), I (Withdrawn-currently amended).
5. Other (e.g., the amendment is unsign	ned or not signed in accordance	with 37 CFR 1.4):
5. Other (e.g., the amendment is unsign	-	
5. Other (e.g., the amendment is unsign ————————————————————————————————————	at required by 37 CFR 1.121, see	
5. Other (e.g., the amendment is unsign For further explanation of the amendment forma	at required by 37 CFR 1.121, see S NOTICE: e non-compliant amendment is a on (only). If applicant wishes to r	MPEP § 714. n after-final amendment, an amendment esubmit the non-compliant after-final
 5. Other (e.g., the amendment is unsigned of the amendment formation of the amendment is given no new time period if the filed after allowance, or a drawing submission amendment with corrections, the entire corrections, the entire corrections, the entire corrections, the entire corrections. 	at required by 37 CFR 1.121, see S NOTICE: e non-compliant amendment is a on (only). If applicant wishes to r rected amendment must be res days, whichever is longer, from t is one of the following: a prelimin ntinued examination (RCE) under under 37 CFR 1.103(a) or (c), a . are checked, the correction req	MPEP § 714. n after-final amendment, an amendment esubmit the non-compliant after-final ubmitted. the mail date of this notice to supply the nary amendment, a non-final amendment r 37 CFR 1.114), a supplemental ind an amendment filed in response to a
 5. Other (e.g., the amendment is unsigned of the second sec	at required by 37 CFR 1.121, see S NOTICE: e non-compliant amendment is a on (only). If applicant wishes to r rected amendment must be res days, whichever is longer, from t is one of the following: a prelimin ntinued examination (RCE) unde under 37 CFR 1.103(a) or (c), a . are checked, the correction req ith 37 CFR 1.121. 37 CFR 1.136(a) <u>only</u> if the non-c	MPEP § 714. n after-final amendment, an amendment esubmit the non-compliant after-final ubmitted. the mail date of this notice to supply the nary amendment, a non-final amendment r 37 CFR 1.114), a supplemental ind an amendment filed in response to a uired is only the corrected section of the
 5. Other (e.g., the amendment is unsigned. For further explanation of the amendment formation of the amendment formation. TIME PERIODS FOR FILING A REPLY TO THE Applicant is given no new time period if the filed after allowance, or a drawing submission amendment with corrections, the entire correction, if the non-compliant amendment (including a submission for a request for corramendment filed within a suspension period Quayle action. If any of above boxes 1. to 4. non-compliant amendment in compliance within a mendment filed in restance. Extensions of time are available under 3 amendment or an amendment filed in restance. 	at required by 37 CFR 1.121, see S NOTICE: e non-compliant amendment is a on (only). If applicant wishes to r rected amendment must be res days, whichever is longer, from t is one of the following: a prelimin ntinued examination (RCE) under a under 37 CFR 1.103(a) or (c), a . are checked, the correction req ith 37 CFR 1.121. 37 CFR 1.136(a) <u>only</u> if the non-o sponse to a <i>Quayle</i> action. will result in: e non-compliant amendment is a r	MPEP § 714. In after-final amendment, an amendment esubmit the non-compliant after-final ubmitted. The mail date of this notice to supply the hary amendment, a non-final amendment r 37 CFR 1.114), a supplemental ind an amendment filed in response to a uired is only the corrected section of the compliant amendment is a non-final non-final amendment or an amendment

Part of Paper No.

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U.S. Patent and Trademark Office

04-25.07

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

N 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-compliancy 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:

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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 N_2 , NH_3 , CO, NO, CO_2 , 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.

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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 thetarget, 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:

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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).

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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;

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providing pulsed DC power to the target; providing a magnetic field to the target; and

reconditioning the target;

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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.

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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.

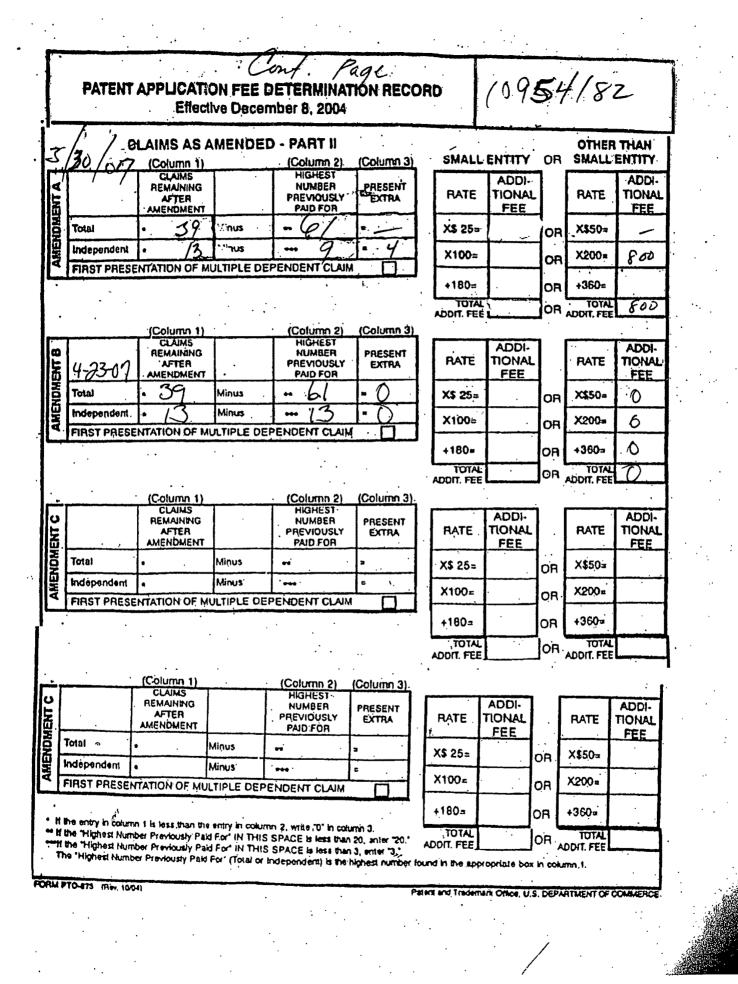
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Date: April 23, 2007

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Page 889 of 1053

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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
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			07/24/2007	PAPER

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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.

PTOL-90A (Rev. 04/07)

		Applicatio	on No.	Applicant(s)
	·	10/954,18	32	ZHANG ET AL.
	Office Action Summary	Examiner		Art Unit
		Michelle E	strada	2823
Period fo	The MAILING DATE of this communicatio or Reply	on appears on the	e cover sheet with the c	correspondence address
WHIC - Exte after - If NC - Failu Any	ORTENED STATUTORY PERIOD FOR F CHEVER IS LONGER, FROM THE MAILIN nsions of time may be available under the provisions of 37 O SIX (6) MONTHS from the mailing date of this communicati 0 period for reply is specified above, the maximum statutory ire to reply within the set or extended period for reply will, by reply received by the Office later than three months after the ed patent term adjustment. See 37 CFR 1.704(b).	NG DATE OF TH CFR 1.136(a). In no even on. period will apply and w statute, cause the app	IIS COMMUNICATION ant, however, may a reply be tin II expire SIX (6) MONTHS from lication to become ABANDONE	N. nely filed the mailing date of this communication. D (35 U.S.C. § 133).
Status				
1)🖂	Responsive to communication(s) filed on	4/23/07.		
1		This action is n	on-final.	
	Since this application is in condition for a		•	osecution as to the merits is
	closed in accordance with the practice un	-		
Disposit	ion of Claims	·		
· _		2 100 is/ara pap	ting in the application	
4)	Claim(s) <u>41-43,45-58,62,85,87-89 and 93</u> 4a) Of the above claim(s) is/are wit			
5)	Claim(s) is/are allowed.			
1 .	Claim(s) <u>93-109</u> is/are rejected.			
	Claim(s) <u>55 765</u> Israre objected to.			
1	Claim(s) are subject to restriction a	and/or election r	equirement	
			equilation.	
Applicat	ion Papers			
9)	The specification is objected to by the Exa	aminer.		
10)	The drawing(s) filed on is/are: a)	accepted or b)	objected to by the	Examiner.
	Applicant may not request that any objection t	to the drawing(s) t	e held in abeyance. Se	e 37 CFR 1.85(a).
	Replacement drawing sheet(s) including the c	-		
11)	The oath or declaration is objected to by t	he Examiner. No	ote the attached Office	e Action or form PTO-152.
Priority	under 35 U.S.C. § 119			
,	Acknowledgment is made of a claim for fo	preign priority un	der 35 U.S.C. § 119(a	ı)-(d) or (f).
	1. Certified copies of the priority docu	iments have hee	n received	
	2. Certified copies of the priority docu			ion No
	3. Copies of the certified copies of the			
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*	See the attached detailed Office action for	-	,	ed
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Page 891 of 1053

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 negatived 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).

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

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	U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS												
Examiner	Cite	Document Number	Issue or	Name of Patentee or	Pages, Columns, Lines, Where								
	No.'	Number-Kind Code ² (# known)	Publication Date MM-DD-YYYY	Applicant of Cited Document	Relevant Passages or Relevant Figures Appear								
HORA	•	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.									
hfe		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).									
		SCHOLL, R., "Power Supplies for Pulsed Plasma Technologies: State-Of-The- Art And Outlook," Advances Energy Industries, Inc., pages 1-8 (1999).									
	1	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).									
	ï	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).									
7		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).									
V		Response to Office Action dated December 21, 2006, in U.S. Application No. 11/228,805 (Attorney Docket No. 9140.0030-01).									
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Examiner Signature

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.

Date Considered

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	NON PATENT LITERATURE DOCUMENTS	
100	10/851,542 (Attorney Docket No. 9140.0033-00).	
ME	Notice of Allowance dated February 22, 2007, in U.S. Application No. 10/851,542 (Attorney Docket No. 9140.0033-00).	
MA	Continuation application and Preliminary Amendment dated December 13, 2006 (Attorney Docket No. 9140.0042-01).	

Date Considered Examiner Signature

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

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.								
Hab,		Response to Office Action dated March 19, 2007, in U.S. Application No. 09/903,081 (Attorney Docket No. 9140.0014-00).								
1		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).								
1 CALAY	/	Application filed March 22, 2007 (Attorney Docket No. 9140.0033-01).	Ì							

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	II Stop RCE nmissioner for Patents b. Box 1450 xandria, VA 22313-1450 s is a Request for Continued Examination (RCE) uses for Continued Examination (RCE) practice under une 8, 1995, or to any design application. Submission required under 37 C.F.R. § 1.114: Material Mate			First Named Inventor: ZHANG, Hongmei et al.					
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PATENT Customer No. 22,852 Attorney Docket No. 10655.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re A	Application of:)
ZHAN	IG, Hongmei et al.) Group Art Unit: 2823
Applic	cation 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:

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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 N_2 , NH_3 , CO, NO, CO_2 , 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.

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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).

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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.

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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).

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<u>REMARKS</u>

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.

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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,

Gáry / Edwards Reg. No. 41,008

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

Dated: October 24, 2007

EM 100825779 US

EXPRESS MAIL LABEL NO.

-7-



PATENT Customer No. 22,852 Attorney Docket No. 10655.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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

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.

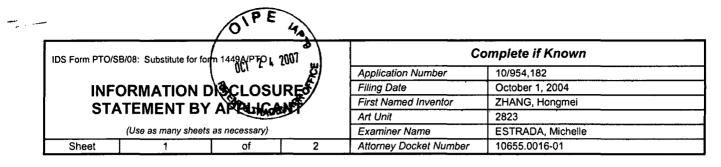
Respectfully submitted,

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

Dated: October 24, 2007

Reg. No. 41,008

EXPRESS MAIL LABEL NO. EM 100825779 US



	U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS							
Examiner	Cite	Document Number	Issue or	Name of Patentee or	Pages, Columns, Lines, Where			
Initials	No.'	Number-Kind Code ² (if known)	Publication Date MM-DD-YYYY	Applicant of Cited Document	Relevant Passages or Relevant Figures Appear			
		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 Country Code ³ Number ⁴ Kind Code ⁵ (<i>if known</i>)	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			
		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
		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. EM 100825779 US

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- (71) Applicant (for all designated States except US): SYM-MORPHIX, INC. [US/US]; 1278 Reamwood Avenue, Sunnyvale, California 94089-2233 (US).
- (72) Inventors; and
- (75) Inventors/Applicants (for US only): ZHANG, Hongmei [US/US]; 1330 Rodney Drive, San Jose, California 95118 (US). DEMARAY, Richard, E. [US/US]; 190 Fawn Lane, Portola Valley, California 94028 (US).
- (74) Agent: EDWARDS, Gary J.; Finnegan, Henderson, Farabow, Garrett & Dunner LLP, 901 New York Avenue, N.W., Washington, D.C., District Of Columbia 20001-4413 (US).

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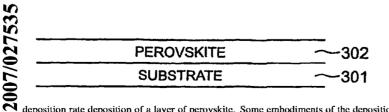
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(54) Title: DEPOSITION OF PEROVSKITE AND OTHER COMPOUND CERAMIC FILMS FOR DELECTRIC APPLICA-TIONS



(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.

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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

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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

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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.

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[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

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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.

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[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.

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[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

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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

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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

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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 amphotaric 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

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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

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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

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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 O_2/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

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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 μ m 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

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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 Ω 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, IrO₂ and also Ti₄O₇, Ti₃O₅, 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.

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				Ţ	ABLE 1			•
: Example #	Film Thickness (nm)	Target Power (W)r	Bias Power (W)	Ar/O ₂ Ratio	Vbd	Ebd	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			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			· · · · · · · · · · · · · · · · · · ·	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			75/25	1.4	0.063665	21950	1048.85
BST Pt 1 step	· 2918	2000	· 0	50/50		Ì	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	Ö	100/0-50/50	· · · ·		1230	45.14
BST IrO2 2 steps	1689	2000	0	100/0-50/50			684	25.10

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Sample#	thickness	target power	bias power	Ar/02	dep time (sec)	Vbđ	Ebd	C(PF)	Dielectric Constant, k		/bd (V)	C(PF)	Dielectric Constant, k (after 500C' anneal)
ALDOEN++-1	840.4					70	8.329367	380	6.939418			1	
	840.4							377	6.884633			1	
ALDOEN++-2	5767.2							60.5	7.581825				
		·		•				1200	0.				
experiment 109	1000							1200	26.07562				
	840					75	8.928571	405	7.392438				
ebonex,BST (A)	1140	900	100		3600		0		0				
Ir Coated #2	2220	900	0	50/50	5400		0	508	24.50587			16800	810.4302
		1										5000	
	2220				1							21530	1038.605
IrO2 Coated #2	2220	900	0	50/50	5400		0	365	17.60756	9	0.405	22000	1061.278
	2220 -	1					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	1		0	3030	59.91525			450	8.898305
	910	1		,	1			2962	58.57062				
	910	1			1			2860	56.55367				
Ti407 (B)	1490	1.	·	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				

Table II

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	•		. ·	•				•	•				
Ir #4	870	900	500 sec no bias/ 4900 sec 50w bias	50/50	5400			very	/ leaky				
Ir #5	2000	900	1500sec no bias/5700 sec 50W bias	50/5 0	7200			very	/ leaky				
IrO2 (Tsub=450c)	2000	900	no bias	50/50	5400	10	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	12	1.284797	870	17.65711	12		2857	57.98431
n++ (7) (room)	2541	900	no bias	50/50	5400			219		13	0.512	2210	122.0254
n++(8) (room)	2504	900	75 w bias	50/50	7200		÷.	224	12.18809	22	0.879	2218	120.6839
n++ (9) (room)	10000		75 w bias	50/50	28800			58	12.60322	55.	0.55	954.7	207.4533
n++ (10) (room)	5000		75w bias	50/50	14400								

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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.

- 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.
- 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.
- The method of claim 6, wherein the perovskite layer is a barium strontium titanite (BST) layer.
- The method of claim 6, wherein the formed perovskite layer is more than about 0.1 micron thick.
- The method of claim 6 wherein the formed perovskite layer is less than about 1 micron thick.
- The method of claim 6, further comprising annealing the perovskite layer formed on the substrate.

- 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 amphotaric 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.

- 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:

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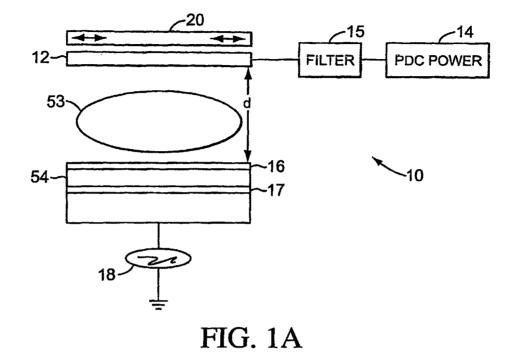
a top portion; and

a bottom portion, wherein

the thin substrate is held when the top portion is attached to the bottom

portion.





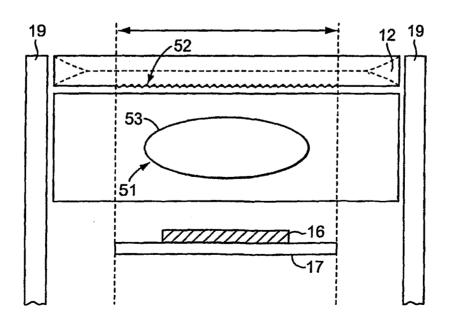


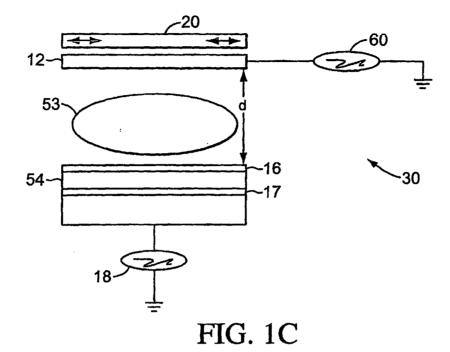
FIG. 1B

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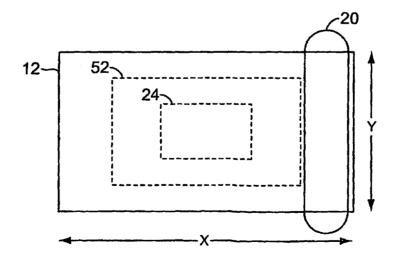


FIG. 2

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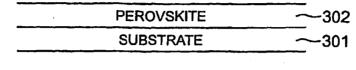


FIG. 3A

and the second s		
	SECOND ELECTRODE	
	PEROVSKITE	~-302
	FIRST ELECTRODE	~303
	SUBSTRATE	~-301

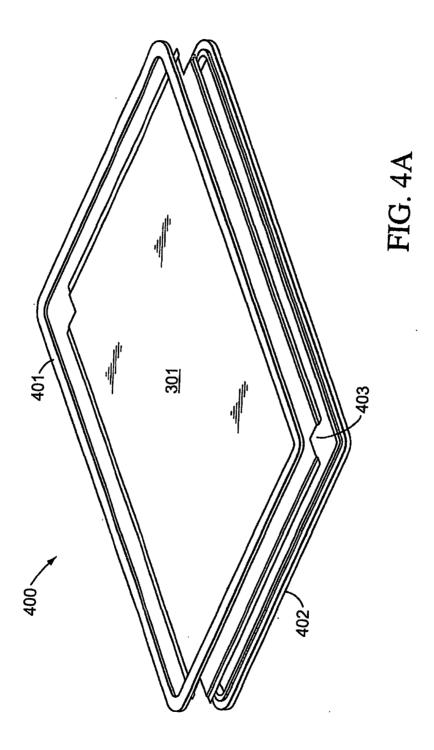
FIG. 3B

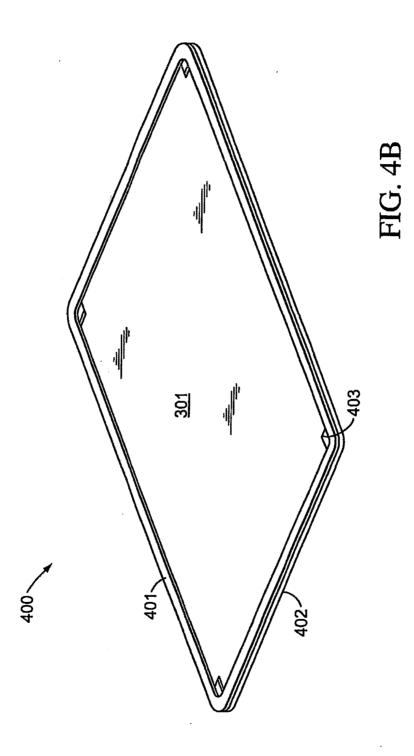
TOP ELECTRODE	~304
PEROVSKITE	~302
•	
•	
•	
PEROVSKITE	
THIRD ELECTRODE	
PEROVSKITE	~- 302
SECOND ELECTRODE	
PEROVSKITE	
FIRST ELECTRODE	303
SUBSTRATE	~-301

FIG. 6

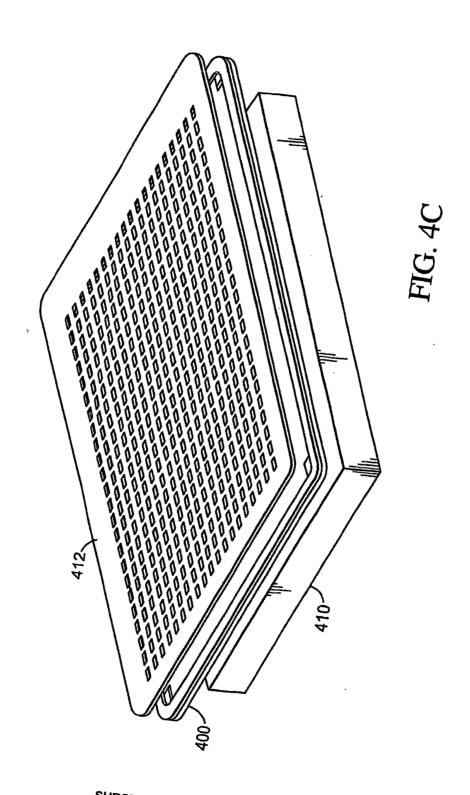
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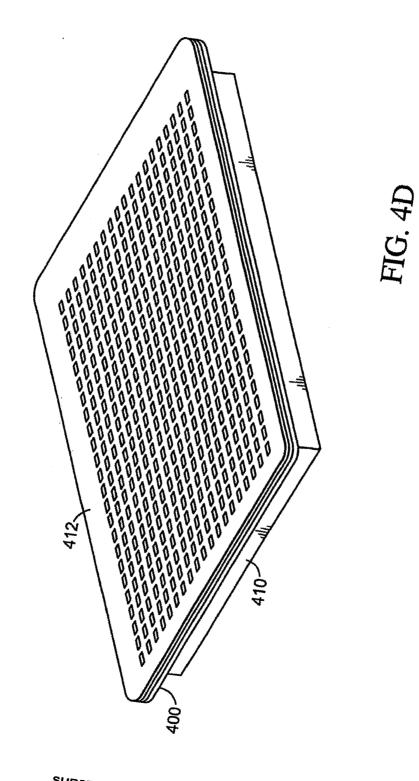
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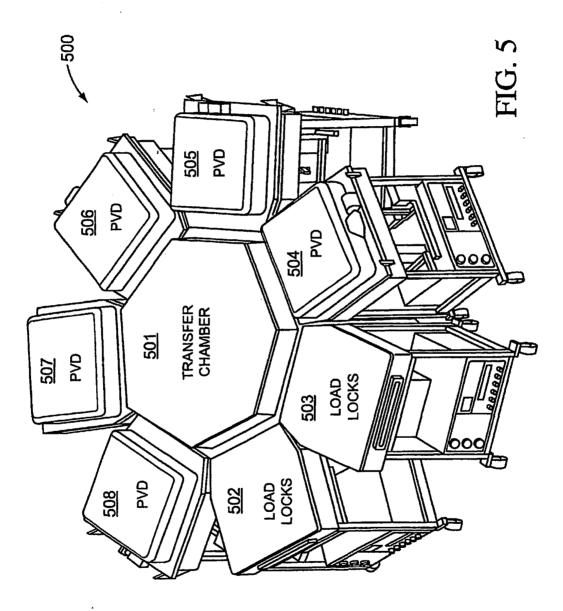




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PTO/SB/06 (07-06)

Approved for use through 1/31/2007. OMB 0651-0032 U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

P/					pplication or l	Docket Number 4,182	Fil	ing Date 01/2004	To be Maile		
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	SEARCH FEE (37 CFR 1.16(k), (i), (i), (i), (i), (i), (i), (i), (i		N/A		N/A		N/A			N/A	
	EXAMINATION FE (37 CFR 1.16(o), (p),	E	N/A		N/A		N/A		1	N/A	
	TAL CLAIMS CFR 1.16(i))		mir	ius 20 = *			X \$ =		OR	X \$ =	
	EPENDENT CLAIM CFR 1.16(h))	S	m	inus 3 = *			X \$ =			X \$ =	
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		(Column 1) CLAIMS		(Column 2) HIGHEST	(Column 3)		SMAL	L ENTITY	OR		R THAN LL ENTITY
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	Total (37 CFR 1.16(i))	* 25	Minus	** 61	= 0		X \$ =		OR	X \$50=	0
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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		LAIM	Application 10/954,	n Number		Filing Date			it displays a valid OMB control numbe			
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							* May be 1	used for addit	tional claims	or amendme	ents		
CLAIMS	AS F	ILED	AMEN	R FIRST DMENT /2007	AFTEI AMENI			*			*	*	
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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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Page 1

			UNITED STATES DEPAR United States Patent and Address: COMMISSIONER F P.O. Box 1450 Alexandria, Virginia 22: www.uspto.gov	Trademark Office OR PATENTS
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 FINNEGAN H	7590 11/15/2007	, OW, GARRETT & DUNNER	EXAM	IINER
LLP	·	OW, OARIETT & DOUNER	ESTRADA,	MICHELLE
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,			MAIL DATE	DELIVERY MODE
			1/15/2007	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.

PTOL-90A (Rev. 04/07)

	Application No.	Applicant(s)
	10/954,182	ZHANG ET AL.
Office Action Summary	Examiner	Art Unit
	Michelle Estrada	2823
The MAILING DATE of this communication		
Period for Reply		
 A SHORTENED STATUTORY PERIOD FOR F WHICHEVER IS LONGER, FROM THE MAILII Extensions of time may be available under the provisions of 37 after SIX (6) MONTHS from the mailing date of this communicat If NO period for reply is specified above, the maximum statutory Failure to reply within the set or extended period for reply will, by Any reply received by the Office later than three months after the earned patent term adjustment. See 37 CFR 1.704(b). 	NG DATE OF THIS COMMUNI CFR 1.136(a). In no event, however, may a ion. period will apply and will expire SIX (6) MOI y statute, cause the application to become A	CATION. reply be timely filed NTHS from the mailing date of this communication BANDONED (35 U.S.C. § 133).
Status		
1) Responsive to communication(s) filed on	24 October 2007	
	This action is non-final.	
3) Since this application is in condition for a	-	ters, prosecution as to the merits is
closed in accordance with the practice u	•	
Disposition of Claims		
· _		
4) \square Claim(s) <u>41-43,45-58,62,85 and 87-89</u> is		
4a) Of the above claim(s) is/are wi5) Claim(s) is/are allowed.		
	lara raiaatad	
 6) Claim(s) <u>41-43,45-58,62,85 and 87-89</u> is 7) Claim(s) is/are objected to. 	are rejected.	
8) Claim(s) are subject to restriction	and/or algotion requirement	
	and/or election requirement.	
Application Papers		
9) The specification is objected to by the Exa	aminer.	
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 abeya	nce. See 37 CFR 1.85(a).
Replacement drawing sheet(s) including the o	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 t	he Examiner. Note the attache	d Office Action or form PTO-152.
Priority under 35 U.S.C. § 119		
12) Acknowledgment is made of a claim for fo a) All b) Some * c) None of:	preign priority under 35 U.S.C. {	§ 119(a)-(d) or (f).
1. Certified copies of the priority docu	ments have been received.	
2. Certified copies of the priority docu	iments have been received in A	opplication No
3. Copies of the certified copies of the	e priority documents have been	received in this National Stage
application from the International E	lureau (PCT Rule 17.2(a)).	
* See the attached detailed Office action for	a list of the certified copies not	received.
•		
Attachment(s)		
		Summary (PTO-413)
1) X Notice of References Cited (PTO-892)		
	48) Paper No(s)/Mail Date nformal Patent Application

Page 2

Application/Control Number: 10/954,182 Art Unit: 2823

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 negatived 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 Application/Control Number: 10/954,182 Art Unit: 2823 Page 3

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 bandwith and frequency through routine experimentation to achieve a desired device associated characteristics and rate of sputtering.

In addition, the selection of the bandwith 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) Application/Control Number: 10/954,182 Art Unit: 2823 Page 4

(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 bandwith and frequency or any unexpected results arising therefrom. Where patentability is said to be based upon particular chosen bandwith and frequency or upon another variable recited in a claim, the Applicant must show that the chosen bandwith and frequency are critical. *In re Woodruf*, 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 or 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 N_2 .

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Chen et al. disclose wherein the target is a metallic target and the process gas includes N_2 (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.

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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 prealloy atomization and hot isostatic pressing of a powder (Page 2, Paragraph [0020]).

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Application/Control Number: 10/954,182 Art Unit: 2823

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

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U.S. PATENT DOCUMENTS

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NON-PATENT DOCUMENTS Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages) Li, Ning et al., "Enhancement of aluminum oxide physical vapor deposition with a secondary plasma", November 28, 2001, Scien Direct, pages, 1-11.

*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.

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257	E23.132,E21.091,E21.169,E21.2,E21.462	11/6/07	ME

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PATENT Customer No. 22,85² Attorney Docket No. 10655.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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

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. Techol. 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 N_2 , NH_3 , 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): $\overline{}$ method of claim 85 wherein the Oxygen flow is adjusted to adjust the index of refraction of the f¹lm.

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 pow^er 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]] <u>an insulating</u> substrate, comprising:

providing a process gas between a conductive target and [[a]] <u>the</u> substrate; providing pulsed DC power to the target <u>through a narrow band rejection filter such that</u> <u>the target alternates between positive and negative voltages;</u> 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]] <u>an insulating</u> 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;

<u>and</u>

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]] <u>85</u>, wherein the narrow band-rejection filter has a bandwidth of about 100 kHz.

Claim 89 (Previously presented): The method according to claim [[87]] <u>85</u>, 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. <u>Therefore, Smolanoff</u>

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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 <u>62.</u> 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,

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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

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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. Techol. B* 13 2 (1995), pp. 203-208, and S. M. Rossnagel 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

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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

Gáry J. Edwards Reg. No. 41,008 (650) 849-6622

Ptta h P.F. C ng t al., J. a C f is o refer c d a tic e gJ.F. C ng t al., J. a C f c hol B f 2 (19 5)' pp 203-208, and S. M. Rossnagel et al., Appl. Phys. Lett. 63 (1993), p. 24.

Electronic Acl	knowledgement 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)

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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	Amendment_Response_to_	215331	no	14			
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Approved for use through 1/31/2007. OMB 0651-0032

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process) an application. Confidentially is governed by 35 0.5.0. 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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Fax No.:	(571) 273-8300 Application No. 10/954,182		lo.: <u>(571) 272-1858</u>
Subject:	Confirmation No. 9873 Atty. Doc. 10655.0016-01000	Date:	January 17, 2008
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	Gary J. Edwards	Phone No.:	
Fax # Ver	ified by: _A. Wong; x6738	No. of Page	s (incl. this page) 2
	Confirmation C	opy to Follow: <u>No</u>	
	CERTIFICATE OF TRANSM	155ION UNDER 3	5/ G.F.K § 1.0
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IDS Form PTO/SB/08: Substitute for form 1449A/PTO		Complete if Known				
		Application Number 10/954,182 RECEIVED				
INFORMATION DISCLOSU	RF	Filing Date	October 1, 2004	CENTRAL FAX CE	ITED	
STATEMENT BY APPLICA	First Named Inventor	ZHANG, Hongmei	CENTRALINA DE	j i i i i i i		
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(Use as many sheets as necessary)	Examiner Name	ESTRADA, Michelle	JAN 1 T. MA	T		
Sheet 1 of	1	Attorney Docket Number	10655.0016-01		J	

	U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS									
Examiner	Cite	Document Number	Issue or	Name of Patentee or	Pages, Columns, Lines, Where					
Iniŭals	No.'	Number-Kind Code ² (it known)	Publication Date MM-DD-YYYY	Applicant of Cited Document	Relevant Passagos or Relevant Figures Appear					
		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										
Examinar Iniliais	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					
,		WO 2007/027535 A2	03-08-2007	Symmorphix, Inc.							

		NON PATENT LITERATURE DOCUMENTS	
Examiner Initials	Cite No.4	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 ^d
		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.

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PAGE 2/2 * RCVD AT 1/17/2008 4:28:32 PM [Eastern Standard Time] * SVR:USPTO-EFXRF-4/1 * DNIS:2738300 * CSID:650 849 6666 * DURATION (mm-ss):01-08

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



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

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

ESTRAD	A, MICHELLE
ART UNIT	PAPER NUMBER
2823	· · · · ·
ATE MAILED: 02/01/2	008

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

THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT. <u>PROSECUTION ON THE MERITS IS CLOSED</u>. 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 <u>THREE MONTHS</u> FROM THE MAILING DATE OF THIS NOTICE OR THIS APPLICATION SHALL BE REGARDED AS ABANDONED. <u>THIS STATUTORY PERIOD CANNOT BE EXTENDED</u>. 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:	If the SMALL ENTITY is shown as NO:
A. If the status is the same, pay the TOTAL FEE(S) DUE shown above.	A. Pay TOTAL FEE(S) DUE shown above, or
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	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.

PTOL-85 (Rev. 08/07) Approved for use through 08/31/2010.

Page 989 of 1053

		PART E	- FEE(S) TRANSM	AITTAL		
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						(Date)
APPLICATION NO.	FILING DATE		FIRST NAMED INVENTOR		ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/954,182 TITLE OF INVENTION	10/01/2004 : BIASED PULSE DC F	EACTIVE SPUTTERIN	Hongmei Zhang G OF OXIDE FILMS		10655.0016-01	9873
APPLN, TYPE	SMALL ENTITY	ISSUE FEE DUE	PUBLICATION FEE DUE	PREV. PAID ISSUE	E FEE TOTAL FEE(S) DUE	DATE DUE
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This collection of inform an application. Confident submitting the completed this form and/or suggesti Box 1450, Alexandria, V Alexandria, Virginia 223	ation is required by 37 C iality is governed by 35 I application form to the ons for reducing this bui irginia 22313-1450. DC 13-1450.	FR 1.311. The informatic U.S.C. 122 and 37 CFR USPTO. Time will vary rden, should be sent to the NOT SEND FEES OR (n is required to obtain or r 1.14. This collection is est depending upon the indiv chief Information Office COMPLETED FORMS TO	etain a benefit by th imated to take 12 n ridual case. Any con r, U.S. Patent and D THIS ADDRESS	he public which is to file (and ninutes to complete, including mments on the amount of tim Trademark Office, U.S. Depa . SEND TO: Commissioner fi lisplays a valid OMB control i	by the USPTO to process) g gathering, preparing, and te you require to complete rtment of Commerce, P.O. or Patents, P.O. Box 1450,

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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
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FINNEGAN, HE	ENDERSON, FARAE	BOW, GARRETT & DUNNER	ESTRADA,	MICHELLE
LLP			ART UNIT	PAPER NUMBER
901 NEW YORK WASHINGTON,		·	2823 DATE MAILED: 02/01/200	8

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.

	Application No.	Applicant(s)
Notice of Allowability	10/954,182 Examiner	ZHANG ET AL.
	Michelle Estrada	2823
The MAILING DATE of this communication app All claims being allowable, PROSECUTION ON THE MERITS IS herewith (or previously mailed), a Notice of Allowance (PTOL-85 NOTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT F of the Office or upon petition by the applicant. See 37 CFR 1.31 1. X This communication is responsive to <u>12/18/07</u> .	S (OR REMAINS) CLOSED in 5) or other appropriate commun RIGHTS. This application is su	this application. If not included nication will be mailed in due course. THIS
2. X The allowed claim(s) is/are <u>41-43,45-58,62,85,88 and 89</u> .		
 3. Acknowledgment is made of a claim for foreign priority of a) All b) Some* c) None of the: Certified copies of the priority documents have Certified copies of the priority documents have Copies of the certified copies of the priority documents have Copies of the certified copies of the priority documents have Copies of the certified copies of the priority documents have Copies of the certified copies of the priority documents have Copies of the certified copies of the priority documents have Copies of the certified copies of the priority documents have Copies of the certified copies of the priority documents have Certified copies not received: Applicant has THREE MONTHS FROM THE "MAILING DATE" noted below. Failure to timely comply will result in ABANDON THIS THREE-MONTH PERIOD IS NOT EXTENDABLE. A SUBSTITUTE OATH OR DECLARATION must be subrink for the priority documents have a subrink of the priority documents have a subrink of the priority documents have a subrink of the priority documents have a subrink of the priority of th	ve been received. ve been received in Application ocuments have been received " of this communication to file a MENT of this application. nitted. Note the attached EXAI	No in this national stage application from the a reply complying with the requirements MINER'S AMENDMENT or NOTICE OF
 5. CORRECTED DRAWINGS (as "replacement sheets") muture (a) including changes required by the Notice of Draftsper 1) hereto or 2) to Paper No./Mail Date (b) including changes required by the attached Examiner Paper No./Mail Date Identifying indicia such as the application number (see 37 CFR each sheet. Replacement sheet(s) should be labeled as such in 6. DEPOSIT OF and/or INFORMATION about the deport attached Examiner's comment regarding REQUIREMENT 	son's Patent Drawing Review 's Amendment / Comment or i 1.84(c)) should be written on the the header according to 37 CFR psit of BIOLOGICAL MATE	n the Office action of e drawings in the front (not the back) of t 1.121(d). RIAL must be submitted. Note the
 Attachment(s) 1. ☐ Notice of References Cited (PTO-892) 2. ☐ Notice of Draftperson's Patent Drawing Review (PTO-948) 3. ☑ Information Disclosure Statements (PTO/SB/08), Paper No./Mail Date <u>1/17/08</u> 4. ☐ Examiner's Comment Regarding Requirement for Deposit of Biological Material 	6. ☐ Interview Sur Paper No./M 7. ☐ Examiner's A	Amount of Reasons for Allowance Mail Date Itatement of Reasons for Allowance Multiple ESTRADA PRIMARY EXAMINER
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	U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS										
Examiner	Cite	Document Number	lasue or	Name of Patentee or	Pages, Columns, Lines, Where						
Initiats	No.'	Number-Kind Code ² (it known)	Publication Date MM-DD-YYYY	Applicant of Cited Document	Relevant Pessages or Relevant Figures Appear						
(VAL)		US-6,391,166 B1	05-21-2002	Wang							
Alle Alle		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									
Examinar Iniliais	Cite No.'	Forsign Patent Document Country Code ⁶ Number ⁴ Kind Code ⁶ (# known)	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				
ARE		WO 2007/027535 A2	03-08-2007	Symmorphix, Inc.						

		NON PATENT LITERATURE DOCUMENTS	
Examiner Iniliais	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
AR		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).	
V		Supplemental Notice of Allowance dated July 5, 2007, in U.S. Application No. 11/228,805 (Attorney Docket No. 9140.0030-01).	
MR		Second Supplemental Preliminary Amendment filed May 31, 2007, in U.S. Application No. 177297.057 (Application No. 9140.0042-00).	

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

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PAGE 2/2* RCVD AT 1/17/2008 4:28:32 PM [Eastern Standard Time]* SVR:USPTO-EFXRF-4/1* DNIS:2738300 * CSID:650 849 6666 * DURATION (mm-ss):01-08

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	Application/Control No.	Applicant(s)/Patent Under Reexamination
Issue Classification	10954182	ZHANG ET AL.
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	Estrada, Michelle	2823

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	Application/Control No.	Applicant(s)/Patent Under Reexamination
Search Notes	10954182	ZHANG ET AL.
	Examiner	Art Unit
	Estrada, Michelle	2823

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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
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SEARCH NOTES		,
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					E	strada, Mic	helle			2823					
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	Index of Claims				10 Ex	Application/Control No. 10954182 Examiner Estrada, Michelle				Reexa ZHANC	Applicant(s)/Patent Under Reexamination ZHANG ET AL. Art Unit 2823				
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02-15-08



OIPE 47001 FEB 13 2008

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)
For: BIASED PULSE DC REACTIVE) Confirmation No.: 9873
SPUTTERING OF OXIDE FILMS	

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

Sir:

- 3

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

Page 1001 of 1053

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.

Reg. No. 41,008 (650) 849-6622

Dated: February 13, 2008

EXPRESS MAIL LABEL NO. EM 074697408 US

P	IDS FORDEPTO/S	B/08: Substitute for for	m 1449A/PTO		C	omplete if Known	
/0	\				Application Number	10/954,182	
	13 2008 1000 000		ISCLOSU	IRE	Filing Date	October 1, 2004	
FEB	ST A	TEMENT BY			First Named Inventor	Hongmei ZHANG	
\land	37		AFFLICA		Art Unit	2823	
S. Cha	ATGARIEN	(Use as many sheets	as necessary)		Examiner Name	Michelle ESTRADA	
A. S.	Sheet	1	of	1	Attorney Docket Number	10655.0016-01	

	U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS										
Examiner	Cite	Document Number	Issue or	Name of Patentee or	Pages, Columns, Lines, Where						
Initials	No.1	Number-Kind Code ² (if known)	Publication Date MM-DD-YYYY	Applicant of Cited Document	Relevant Passages or Relevant Figures Appear						
		US-									

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 Country Code ³ Number ⁴ Kind Code ⁵ (<i>if known</i>)	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							

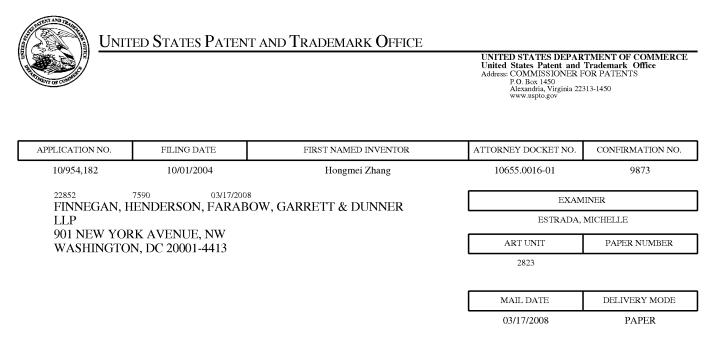
		NONPATENT LITERATURE DOCUMENTS	
Examiner Initials	Cite No. ¹	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	Translation ⁶
		Response to Office Action dated 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

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



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.

PTOL-90A (Rev. 04/07)

Page 1004 of 1053



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
				EXAMINER
	, , ,	RRETT & DUNNER	Mi	chelle Estrada
901 NEW YORK AVEN WASHINGTON, DC 2	·		ART UNIT	PAPER
			2823	20080304
			DATE MAILED	:

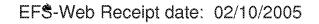
Please find below and/or attached an Office communication concerning this application or proceeding.

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

PTO-90C (Rev.04-03)



10954182 - G



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.

Application No.: 10/954,182

Filed: October 1, 2004

Group Art Unit: 2882

Examiner: Not Yet Assigned

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.

EFS-Web Receipt date: 02/10/2005

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

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /ME/

Page 1007 of 1053

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.

Bv

Gary J. Edwards Reg. No. 41,008

Dated: February 9, 2005

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				Application Number	10/954,182	C.
INF	ORMATION DI	ISCLOSU	RE	Filing Date	October 1, 2004	✓ ►
	ATEMENT BY			First Named Inventor	Zhang et al.	FEB 1 0 2005 W
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Examiner Initials	Cite . No. ¹	Document Number Number-Kind Code ² (<i>if known</i>)	Issue or Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Docume	ent	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
		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.		, <u>, , , , , , , , , , , , , , , , , , </u>
		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.		<u> </u>
		US 5,107,538	Apr. 21, 1992	Benton et al.		<u> </u>
		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	T	/Michelle Estrada/		Date Conside	ered	03/04/2008

EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not conformation of the conformation

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S Form PTO/SB/	08: Substitute for fo	orm 1449A/PTO		Complete if Known			
				Application Number	10/954,182		
INFO	RMATION	DISCLOSU	IRF	Filing Date	October 1, 2004		
				First Named Inventor	Zhang et al.		
JIAI		AFFLICA		Art Unit	2882		
(Use as many shee	ts as necessary)		Examiner Name	Not Yet Assigned		
Sheet	2	of	9	Attorney Docket Number	09140-0016-01000		

		U.S. PATENTS	AND PUBLISHE	D U.S. PATENT APPLICA	ATIONS
Examiner Initials	Cite No.1	Document Number Number-Kind Code ² (if known)	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
		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.	
Examiner Signature	T	/Michelle Estrada/		Date Considered	03/04/2008

EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not the matter of the second seco

IDS Form PTO/S	SB/08: Substitute for for	n 1449A/PTO		C	omplete if Known
				Application Number	10/954,182
INF	ORMATION D	ISCLOSU	RF	Filing Date	October 1, 2004
	ATEMENT BY			First Named Inventor	Zhang et al.
317		AFFLICA		Art Unit	2882
	(Use as many sheets as necessary)			Examiner Name	Not Yet Assigned
Sheet	3	of	9	Attorney Docket Number	09140-0016-01000

		U.S. PATENTS	AND PUBLISHE	ED U.S. PATENT APPLICAT	IONS
Examiner Cite		Document Number	Issue or	Name of Patentee or	Pages, Columns, Lines, Where
Initials	No.1	Number-Kind Code ² (if known)	Publication Date MM-DD-YYYY	Applicant of Cited Document	Relevant Passages or Relevant Figures Appear
		US 6,413,645 B1	Jul. 2, 2002	Graff et al.	
		US 6,416,598 B1	Jul. 9, 2002	Sircar	
		US 6,423,776 B1	Jul. 23, 2002	Akkapeddi et al.	
		US 6,433,380 B2	Aug. 13, 2002	Shin	
		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. ¹	Foreign Patent Document Country Code ³ Number ⁴ Kind Code ⁵ (<i>it known</i>)	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				
		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 DOCU	JMENTS				
Examiner Cite 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.							
		AFFINITO et al., "PML/oxide/PML Barrier Layer Performa of UV or Electron Beam Polymerization of the PML Layers pp. 19-25 (1997)					
		AFFINITO et al., "Polymer-Oxide Transparent Barrier Laye 39th Ann. Technical Conference Proceedings, May 5-10, 19 (1996).					
		ALDER, T. et al., "High-Efficiency Fiber-to-Chip Coupling Mode Fiber," <i>IEEE Photonics Technology Letters</i> , 12(8):10					
Examiner Signature		/Michelle Estrada/	Date Considered	03/04/2008			

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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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Date Considered

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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. (19872)
For: BIASED PULSE DC REACTIVE) Confirmation No.: 9873
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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

Gary 1/Edwards Reg. No. 41,008

Express Mail Label No. EV 860819695 US

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	IDS Form PTO/SB/08: Substitute for form 1449A/PTO				C	Complete if Known		
1	E	,			Application Number	10/954,182		
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		TEMENT B			First Named Inventor	Hongmei ZHANG		
	2 2006		TAFFLICA		Art Unit	2823		
IAK 9	2 2000 W	(Use as many shee	ets as necessary)		Examiner Name	ESTRADA, Michelle		
	Sheet	1	of	3	Attorney Docket Number	9140.0016-01		
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	C '.		<u> </u>	ED U.S. PATENT APPLICAT	
Examiner Initials	Cite No. ¹	Document Number Number-Kind Code ² (if known)	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
		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.	
	N.	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 Country Code ³ Number ⁴ Kind Code ⁵ (<i>if known</i>)	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		
		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	/Michelle Estrada/	Date	03/04/2008
Signature		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 ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /ME/

EXPRESS MAIL NO. EV 860819695 US

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

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

Complete if KnownApplication Number10/954,182Filing DateOctober 1, 2004First Named InventorHongmei ZHANGArt Unit2823Examiner NameESTRADA, Michelle

9140.0016-01

Attorney Docket Number

(Use as many sheets as necessary)
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. 09.140.0015-01).					
ľ	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	/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/

EXPRESS MAIL NO. EV 860819695 US

IDS Form PTO/S	S Form PTO/SB/08: Substitute for form 1449A/PTO		Complete if Known			
				Application Number	10/954,182	
INFORMATION DISCLOSURE		Filing Date	October 1, 2004			
STATEMENT BY APPLICANT		First Named Inventor	Hongmei ZHANG			
(Use as many sheets as necessary)				Art Unit	2823	
				Examiner Name	ESTRADA, Michelle	
Sheet	3	of	3	Attorney Docket Number	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	/Michelle Estrada/	Date Considered	03/04/2008
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APPLICATION NO.	FILING DATE		FIRST NAMED INVENTOR		Annie Wong	DOCKET NO.	CONFIRMATION NO.
	10/01/2004		Hongmei Zhang			.0016-01	
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 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 Image: Structure (1) the names of up to 3 registered patent attorneys or agents OR, alternatively, Image: Structure (2) the name of a single firm (having as a member a registered patent 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. Image: Structure				
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				Application Number	10/954,182	
INF	ORMATION D	ISCLOSU	RE	Filing Date	October 1, 2004	
	TEMENT BY	-		First Named Inventor	Zhang et al.	
517		AFFLICA		Art Unit	2882	
	(Use as many sheets	as necessary)		Examiner Name	Not Yet Assigned	
Sheet	8	of	9	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
		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/Jun 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," Journal of Lightwave Technology, 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).	
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		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). l/w^{c3}	1
		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 12/04 (Attorney Docket No. 09140-0002-02). 12/04	
		Office Action issued on May 2, 2002 in U.S. Patent No. 6,533,907 3/03 (Attorney Docket No. 09140-0004-00). 3/03	
,		Response to Office Action filed on September 3, 2002 in U.S. Patent No. 6,533,907 (Attorney Docket No. 09140-0004-00).	
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		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).	,
	<i>.</i>	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	/Michelle Estrada/	Date	03/04/2008
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				Application Number	10/954,182	
INF	ORMATION D	ISCLOSU	RE	Filing Date	October 1, 2004	
	TEMENT BY			First Named Inventor	ZHANG, Hongmei	
514	VIEWENT DI	AFFLICA		Art Unit	2823	
	(Use as many sheets as necessary)			Examinar Nama	ESTRADA, Michelle	
Sheet	3	of	3	Attorney Docket Number	9140.0016-01	

1	NON PATENT LITERATURE DOCUMENTS	
4119	Response to Final Office Action filed April 14, 2006, in U.S. Appl. No. 10/291,179 (Atty. Docket No. 9140.0001-00).	
1	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).	61
	Response to Office Action filed February 28, 2006 in U.S. Application No. 09/903,081 (Atty. Docket No. 09140-0014-00).	41
	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).	
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	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).	
V	Response to Office Action filed July 26, 2006 in U.S. Application No. 10/851,542 (Atty. Docket No. 09140.0033-00).	
PANY	Specification as filed September 2, 2005, for U.S. Appl. No. 11/218,652 (Atty. Docket No. 09140.0052-00)	

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IDS Form PTO/S	B/08: Substitute for for	m 1449A/PTO		Complete if Known		
				Application Number	10/954,182	
INFO	DRMATION D	ISCLOSU	IRE	Filing Date	October 1, 2004	
	TEMENT BY			First Named Inventor	ZHANG, Hongmei	
3.7			1.01	Art Unit	2823	
	(Use as many sheets as necessary)			Examiner Name	ESTRADA, Michelle	
Sheet	2	of	3	Attomey Docket Number	9140.0016-01	

		NON PATENT LITERATURE DOCUMENTS		
		No. 10,291,179 (Attorney Docket No. 9140.0001-00).		
M	Æ	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). Demarcy et al. 12/2004		80 411710
		Notice of Allowance issued on October 8, 2002, in U.S. Patent No. 6,533,907 (Atty. Docket No. 09140-0004-00). Demarcy efal 3/2003	·	,,,,,,,
		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).		Ľ.
		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).		
M	E	Response to Office Action from Singapore Patent Office in Appl. No. 200505388-9, dated August 11, 2006 (Atty. Docket No. 9140.0030-00256).		

Signature /Michelle Estrada/ Considered 03/02/2007	Examiner		Date	
	Signature	/Michelle Estrada/	Considered	03/02/2007

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EFS-Web Receipt date: 02/10/2005

10954182 - GAU: 2823

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	Demaray etd. 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	<i>B</i> 41171
09140-0034-00	10/850,968	Pemavay del, US 2005/0000794 A1 1/2005	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.

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /ME/

IDS Form PTO/SB/08: Substitute for form 1449A/PTO				Complete if Known	OIPE	
				Application Number	10/954,182	S.
INFORMATION DISCLOSURE				Filing Date	October 1, 2004	Þ
				First Named Inventor	Zhang et al.	FEB 1 0 2005 H
514	STATEMENT BY APPLICANT			Art Unit	2882	2
	(Use as many sheets	as necessary)		Examiner Name	Not Yet Assigned	in it
Sheet	1	of	9	Attorney Docket Number	09140-0016-01000	& TRADEMA

<u>.</u>.

U.S. PATENTS AND PUBLISHED U.S. PATENT APPLICATIONS						
Examiner Initials	Cite . No.1	Document Number Number-Kind Code ² (if known)	Issue or Publication Date	Name of Pate Applicant of Cited		Pages, Columns, Lines, Where Relevant Passages or Relevant
		US 2001/0027159 A1	• MM-DD-YYYY Oct. 4, 2001	Kaneyoshi	······	Figures Appear
		US 2002/0033330 A1	Mar. 21, 2001	Demaray et al.		
		US 2002/0134671 A1	Jul: 17, 2002	Demaray et al.	9102-	Annmm
		US 2002/0170821 A1	Nov. 21, 2002	Sandlin et al.	1100	· · ·
	·	US 2003/0097858 A1	May 29, 2003	Strohhofer et al.		
		US 2003/0127319 A1	Jul. 10, 2003	Demaray et al.		
		US 2003/0124919 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 2003/01/4391 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.		ana tana dikamananan ara para ara ara ara ara ara ara ara ar
		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, 1992	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.		••••••••••••••••••••••••••••••••••••••
	<u> </u>	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.	<u></u>	
		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		/Michelle Estrada/			Date	03/04/2008
Signature					Considered	03/04/2000

EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if no孩儿如何要要要问题的问题。/ME/

k



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.	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;

IR103 (Rev. 11/05)

Page 1031 of 1053

PATENT Customer No. 22,852 Attorney Docket No. 10655.0016-01

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re U	S. Patent No.: 7,381,657)
Invente	ors: Hongmei ZHANG et al.)
Issue I	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

Ron k By: Gary J. Edwards

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:	Ga	ary James Edward	s/Annie Wonç]				
Attorney Docket Number:	10	655.0016-01						
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:	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 Acl	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)

Image: Information: Request for Certificate of Correction Request Certificate of Correction 35334 extended procession no 3 Warnings: Information:	Document Number	Document Description	File Name	File Size(Bytes) /Message Digest	Multi Part /.zip	Pages (if appl.			
pdf consecutor/secundences Warnings: Information: 2 Fee Worksheet (PTO-06) fee-info.pdf 8157 overlead-action no 2 Total Files Size (in bytes): 43491 43491 This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated document 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. <td></td> <td></td> <td></td> <td>35334</td> <td></td> <td>0</td>				35334		0			
Information: 8157 no 2 Fee Worksheet (PTO-06) fee-info.pdf 8157 no 2 Warnings: Information: 754050 100 2 Warnings: Information: 43491 1 This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated document 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 1.54) 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 conditio 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 i	1	Request for Certificate of Correction			no	3			
2 Fee Worksheet (PTO-06) fee-info.pdf 8157 no 2 Warnings: Information: Total Files Size (in bytes): 43491 This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated document 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 condition of 35 U.S.C. 371 and other applicable requirements a Form PCT/D0/E0/903 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 filed with 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 file of the International Application of the Internati	Warnings:								
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Total Files Size (in bytes): 43491 This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated document 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 conditio 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 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	Warnings:								
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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. <u>New Applications Under 35 U.S.C. 111</u> 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. <u>National Stage of an International Application under 35 U.S.C. 371</u> If a timely submission to enter the national stage of an international application is compliant with the condition 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. <u>New International Application Filed with the USPTO as a Receiving Office</u> 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									
	characterize similar to a l <u>New Applica</u> If a new app	d by the applicant, and including Post Card, as described in MPEP ations Under 35 U.S.C. 111 lication is being filed and the app	ceipt on the noted date by t page counts, where applic 503. lication includes the neces	the USPTO of the ind able. It serves as ev sary components fo	dicated do vidence of or a filing d	receipt ate (see			

UNITED STATES PATENT AND TRADEMARK OFFICE CERTIFICATE OF CORRECTION

Page 1 of 1

PATENT NO.: 7,381,657 B2APPLICATION NO.: 10/954182DATED: June 3, 2008INVENTOR(S): Hongmei Zhang et al.

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

JON W. DUDAS Director of the United States Patent and Trademark Office

PATENT Attorney Docket No. 10655.0016-01000

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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

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

Bv:

C. Larry O'Rourke Reg. No. 26,014 (650) 849-6600

Electronic Acl	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 wit	th Payment		no				
File Listing	g:						
Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)		
1	Petition to withdraw attorney or agent		Request_for_Withdrawal_1065	11787	no	1	
	(SB83)	5-0016-01.PDF	c431d494a4cc3cab23a44aa0e61de91d766 5d00f				
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: as H. Kel Name: Title:

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	Assigment Reel/Frame	Recorded Date	
43668.3	11/191.643	7/27/2005	H. Zhang M. Narasimhan	Biased Pulse DC Reactive Sputtering Of	SpringWorks LLC	From Inventors to Symmorphix	014766 / 0601	12/2/2003	
45000.5	11/191,045	112112005	R. Mullapudi R. Demaray	Oxide Films	Spring works LEC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.4	11/100,856	4/6/2005	T. Pan R. Demaray Y. Chen	Mode Size Converter	SpringWorks LLC	From Inventors to Symmorphix	020035 / 0110	10/30/2007	
	1,1,100,000		Y. Xie R. Pethe	For A Planar Waveguide	Spring , one bbo	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.5	10/850,968	5/20/2004	R. Demaray	Transparent Conductive	SpringWorks I I C	From Inventors to Symmorphix	014945 / 0661	8/4/2004	
43008.5	10/050,900	5/20/2004	M. Narasimhan	Oxides	SpringWorks LLC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.22	10/650,461	8/27/2003	D. Dawes	Optically Coupling Into		SpringWorks LLC	From Inventors to Symmorphix	014897 / 0768	1/16/2007
	10/050,401	0/2//2005	D. Dawes	Waveguides		From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
12668.20	11/70 (070	2/22/2005	R. Demaray H. Zhang	Energy Conversion And Storage Devices By Physical Vapor			From Inventors to Symmorphix	014948 / 0097	8/5/2004
43668.39	11/726,972	3/22/2007	M. Narasimhan V. Milonopoulou	Deposition Of Titanium And Titanium Oxides And Sub-Oxides		From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.44	11/297,057	12/7/2005	H. Zhang R. Demaray	Deposition Of LiCoO2	Spring Works LLC	From Inventors to Symmorphix	017196 / 0699	12/21/2006	
43008.44	11/297,037	12/7/2003	M. Shao	Deposition Of LiCoO2	SpringWorks LLC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.53	11/218,652	9/2/2005	H. Zhang	Deposition Of Perovskite And Other Compound Ceramic	SpringWorks LLC	From Inventors to Symmorphix	016821 / 0220	11/28/2005	
		51212005	R. Demaray	Films From Dielectric Applications	Spring works LLC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.6	10/291,179 US Pat No.	ISSUED -	R. Demaray,	Low temperature zirconia based thermal	Serie Wester LLC	From Inventors to Symmorphix	014756 / 0416	12/2/2003	
43000.0	7,404,877	7/29/08	V. Milonopoulou	barrier layer by PVD	SpringWorks LLC	From Symmorphix to SpringWorks LLC	20134 / 0102	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	Assigment Reel/Frame	Recorded Date	
43668.8	09/903,050 US Pat No.	ISSUED -	R. Demaray, K. Wang, R. Mullapudi, D.	Planar optical devices and methods for their	SpringWorks LLC	From Inventors to Symmorphix	012010 / 0318	07/10/2001	
15000.0	6,506,289	1/14/03	Stadtler, H Zhang, R. Peth	manufacture	opining works EEC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.9	10/288,278 US Pat No.	ISSUED -	R. Demaray, K. Wang, R. Mullapudi, D.	Planar optical devices and methods for their	SpringWorks LLC	From Inventors to Symmorphix	012010 / 0318	07/10/2001	
15000.9	6,827,826	12/7/04	Stadtler, H Zhang, R. Pethe	and methods for their manufacture	Spring works LEC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.13	09/766,463	ISSUED -	R. Demaray, J. Shan,	Method of Producing amorphous silicon for hard mask and waveguide applications		From Inventors to Symmorphix	011504 / 0738	01/19/2001	
43008.13	U.S. Pat No. 6,533,907	3/18/03	K. Wang, R. Mullapudi		and Spring Works LLC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.14	09/903,081 US Pat No.	ISSUED -	R. Demaray, K. Wang, R. Mullapudi, Q. Zhu,	As-deposited planar optical waveguides with low scattering loss and methods for their manufacture	optical waveguides with	Series Wester LLO	From Inventors to Symmorphix	012010 / 0752	07/10/2001
43008.14	7,469,558	12/30/08	H. Zhang, H. Ackler, J. Egermeier, R. Pethe		Spring works LLC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.15	10/101,492 US Pat No.	ISSUED -	T. Pan, R. Demaray, Y. Chen, Y. Xie,	Mode size converter for	SpringWorks LLC	From Inventors to Symmorphix	020035 / 0110	10/30/2007	
45008.15	6,884,327	4/26/05	R. Pethe	a planar waveguide	Spring works LLC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.17	10/101,863 US Pat No.	ISSUED -	H. Zhang, M. Narasimhan,	Biased pulse DC reactive sputtering of	SpringWorks LLC	From Inventors to Symmorphix	014766 / 0601	12/2/2003	
43000.17	7,378,356	5/27/08	R. Mullapudi, R. Demaray	oxide films	Spring works LLC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.21	10/954,182 US Pat No.	ISSUED -	H. Zhang, M. Narasimhan,	Biased pulse DC reactive sputtering of	SpringWorks LLC	From Inventors to Symmorphix	014766 / 0601	12/2/2003	
-13000.21	7,381,657	6/30/08	R. Mullapudi, R. Demaray	oxide films	opring works ELC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.19	11/228,834 US Pat No.	ISSUED -	H. Zhang, M. Narasimhan,	Biased pulse DC reactive sputtering of	SpringWorks LLC	From Inventors to Symmorphix	014766 / 0601	12/2/2003	
15000.15	7,544,276	6/9/09	R. Mullapudi, R. Demaray	oxide films	oping works ELC	From Symmorphix to SpringWorks LLC	20134 / 0102	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	Assigment Reel/Frame	Recorded Date	
43668.20	11/228,717 U.S. Pat No.	ISSUED -	H. Zhang, M. Narasimhan,	Biased pulse DC reactive sputtering of oxide films	SpringWorks LLC	From Inventors to Symmorphix	014766 / 0601	12/2/2003	
43000.20	7,413,998	8/19/08	R. Mullapudi, R. Demaray		Spring works LLC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.25	10/789,953 US Pat No. ISSUED - M. Narasimhan, Dielectric Barrier Layer	Spring Works LLC	From Inventors to Symmorphix	014948 / 0111	08/05/2004				
13000.23	7,205,662	4/17/07	P. Brooks, R. Demaray	Films	SpringWorks LLC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.32	11/228,805 US Pat No.	ISSUED -	M. Narasimhan,	Dielectric Barrier Layer	Dielectric Barrier Layer	ectric Barrier Layer SpringWorks LLC	From Inventors to Symmorphix	014948 / 0111	08/05/2004
45000.52	7,262,131	8/28/07	P. Brooks, R. Demaray	Films	Spring works LLC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007	
43668.34 U	10/851,542 US Pat No.			Energy Conversion and Storage Devices by Physical Vapor	Serie - Wester LLC	From Inventors to Symmorphix	014948 / 0097	8/5/2004	
	7,238,628 7/3/07	V. Milonopoulou	Deposition of Titanium and Titanium Oxides and Sub-Oxides	SpringWorks LLC	From Symmorphix to SpringWorks LLC	20134 / 0102	11/19/2007		

SpringWorks LLC

Βv Name: Title:



PTO/SB/122 (11-08)

U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE 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	Application Number	10/954,182		
CORRESPONDENCE ADDRESS	Filing Date	10-1-2004		
Application	First Named Inventor	H. Zhang		
Address to:	Art Unit	2823		
Commissioner for Patents P.O. Box 1450	Examiner Name	Michelle Estrada		
Alexandria, VA 22313-1450	Attorney Docket Number	43668.21		
Please change the Correspondence Address for the above-identified patent application to:				
Image: The address associated with Customer Number: 27683				
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I am the:				
Applicant/Inventor				
Assignee of record of the entire interest. Statement under 37 CFR 3.73(b) is encl				
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 Gary J. Edwards				
Date December 2, 2009	Telephone 408-660-4120			
NOTE: Signatures of all the inventors or assignees of record of the entire inte forms if more than one signature is required, see below*.		uired. Submit multiple		
*Total of forms are submitted.				
This collection of information is required by 37 CFR 1.33. The information is to process) an application. Confidentiality is governed by 35 U.S.C. 122 an				

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 wit	h Payment	no			
File Listing:					
Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1		43668 POA.pdf	482094	Vec	4
		43008_r OA.pdi	50be7508dee6f2cb790d6ae139f6af2655b5 3fda	yes	4

	Multi	part Description/PDF files i	n .zip description		
	Document Description		Start	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
		d99d50b20ef1f621e331386a1b218c7bf1aa 07ba			
Warnings:					
Information:					
		Total Files Size (in byte	(s): 780	011	

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.

			450 a, Virginia 22313-1450
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
27683		POA ACC	EPTANCE LETTER
HAYNES AND BOONE, L	LP		
IP Section			DC000000039121160*
2323 Victory Avenue			000000039121160*
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 Patent and Trademark Office Address COMMISSIONER FOR PATENTS PC Box 1450 Alexandra, Virginia 22313-1450 www.uspl.opv				
APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE	
10/954,182	10/01/2004	Hongmei Zhang	43668.21	
22852 FINNEGAN, HENDERSO LLP 901 NEW YORK AVENUE WASHINGTON, DC 2000			CONFIRMATION NO. 9873 F ATTORNEY NOTICE	

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 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	REQUEST ID
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:
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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).

Signature:	/Gary Edwards/
Name:	Gary Edwards
Registration Number:	41008