EXHIBIT B

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(54) **BIASED PULSE DC REACTIVE SPUTTERING OF OXIDE FILMS**

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See application file for complete search history.

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(57) **ABSTRACT**

A biased pulse DC reactor for sputtering of oxide films is presented. The biased pulse DC reactor couples pulsed DC at a particular frequency to the target through a filter which filters out the effects of a bias power applied to the substrate, protecting the pulsed DC power supply. Films deposited utilizing the reactor have controllable material properties such as the index of refraction. Optical components such as waveguide amplifiers and multiplexers can be fabricated using processes performed on a reactor according to the present inention.
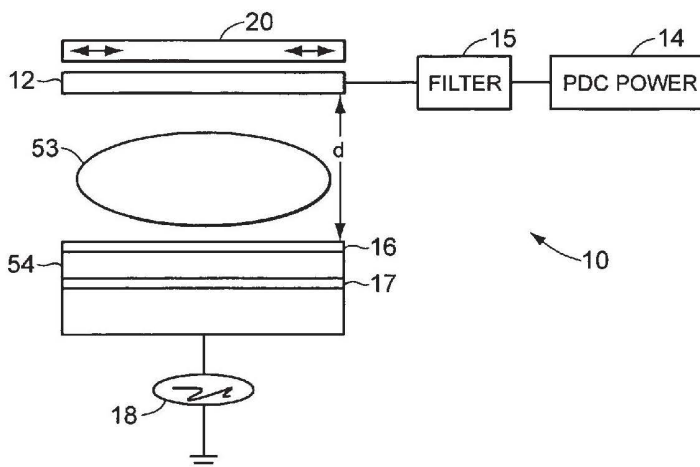
21 Claims, 27 Drawing Sheets

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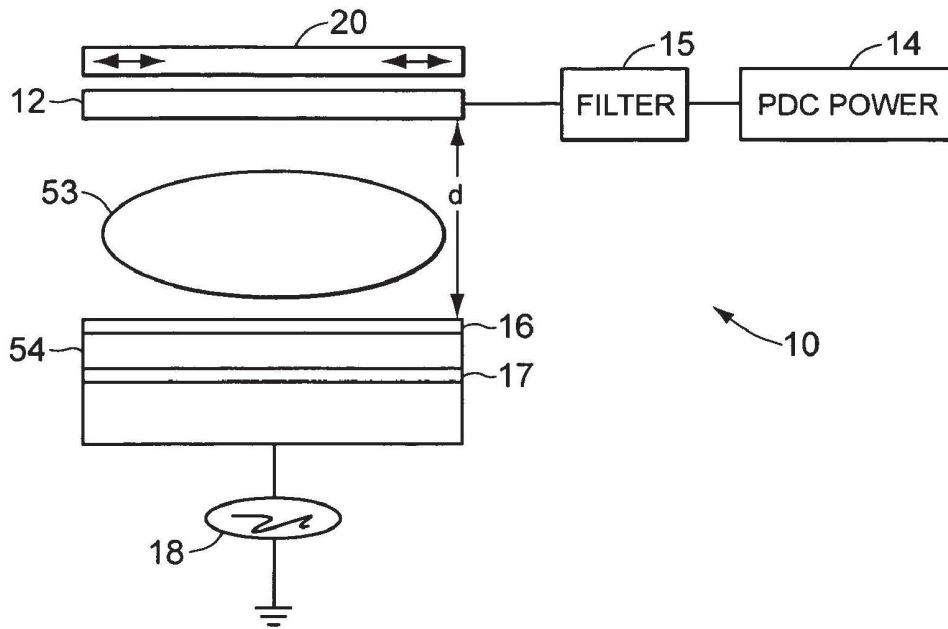
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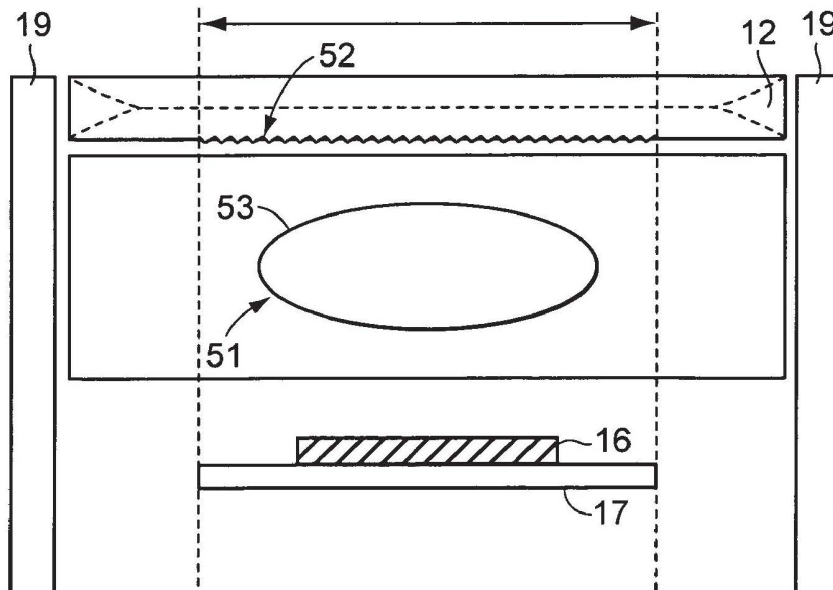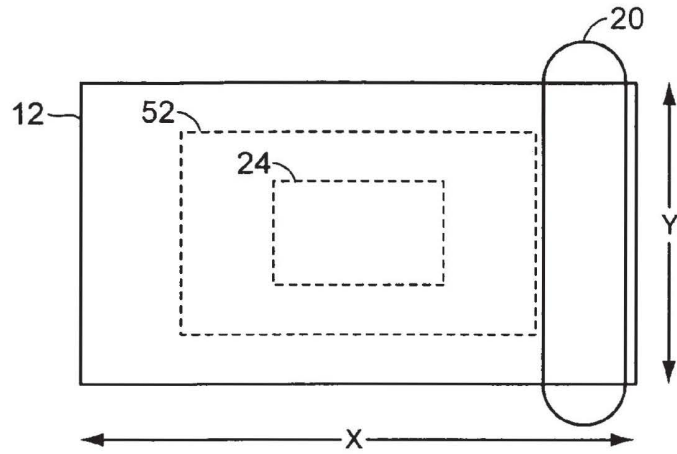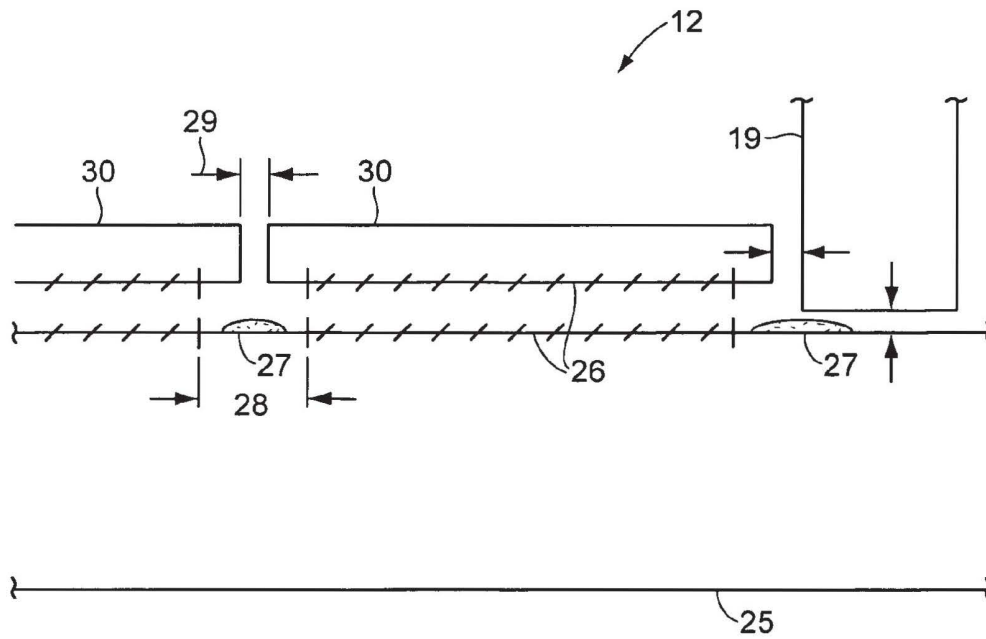
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FIG. 1A

FIG. 1B

FIG. 2

FIG. 3

400

| TARGET PREPARATION | — 401 |

↓

| SUBSTRATE PREPARATION | — 402 |

↓

| SET GAS FLOW | — 403 |

↓

| SET SUBSTRATE TEMPERATURE | — 404 |

↓

| SET MAGNET SCAN | — 405 |

↓

| SET BIAS | — 406 |

↓

| SET PULSED POWER PARAMETERS | — 407 |

↓

| DEPOSIT FILM | — 408 |

FIG. 4

TARGET VOLTAGE vs. OXYGEN FLOW

FIG. 5

LIFE TIME AND PL vs. ANNEAL TEMPERATURE

FIG. 6

FIG. 7

Refractive Index as a Function of Al% in Aluminosilicates

$$y=1E\text{-}10x^5\text{-}3E\text{-}08x^4+3E\text{-}06x^3\text{-}8E\text{-}05x^2+0.002x+1.4583$$
$$R^2=0.9499$$

70-30 Hipped Target
(Fe contamination)

◆ R.I (As Dep)
□ R.I Post Anneal
— Poly. (R.I(As Dep))

FIG. 8

Material (Er/Yb/Al/Si)	Expected index (Post anneal)	Actual index (Post anneal)	Process Conditions (Annealed 725c 30min)
0.8/0.8/41.4/57	1.506	1.510	6KW, Ar-60,02-28sccm, 120Khz, 2.2us, 60mm T-W Space, 4-5mm T-M Space, 0-400W Bias
1.6/0.5/49/48.9	1.526	1.528	6KW, Ar-60,02-28sccm, 120Khz, 2.2us, 60mm T-W Space, 4-5mm T-M Space, 0-400W Bias
0/0/8/92	1.452	1.456-1.459	4.5KW, Ar-30-60,02-28-44, 120-200Khz, 2.2us, 60-85mm T-W Space, 4-5mm T-M Space, 0-400W Bias
0/0/40/60	1.504	1.486-1.501	3.0-4.5KW, Ar-30,02-44, 75-200Khz, 2.2-3.0us, 60-85mm T-W Space, 4-5mm T-M Space, 0-400W Bias
0/0/50/50	1.520	1.491-1.503	4.0-5KW, Ar-30,02-44, 75-200Khz, 2.2-3.0us, 60-85mm T-W Space, 4-5mm T-M Space, 0-400W Bias
0/0/65/35	1.544	1.545-1.560	4.5-5.5KW, Ar-75-90,0285-100, 200Khz, 2.2us, 85mm T-W Space, 4-5mm T-M Space, 0-400W Bias
0/0/30/70	1.490	1.562 (high Fe content)	5.0KW, Ar-75,02-100, 200Khz, 2.2us, 85mm T-W Space, 4-5mm T-M Space, 0-400W Bias
1.5/0/48.5/50	1.523	1.509-1.513	6KW, Ar-60,02-28sccm, 120Khz, 2.2us, 60mm T-W Space, 4-5mm T-M Space, 0-400W Bias

FIG. 9

Bias Power (Watts)	DC Power (KW)	Pulsing Freq (KHz)	Reverse Time (μs)	Ar Flow (sccms)	O2 Flow (sccms)	Wafer Position	Target To Wafer Spacing (mm)	Refractive Index Avg (@1550nm)	Refractive Index STD (@1550nm)	Dep Rate (um/Hr)
150	4.5	200	2.2	100	100	1	55	1.461508	0.000535	0.957654
150	4.5	200	2.2	100	100	2	55	1.462329	0.000376	0.962581
400	4.5	200	2.2	100	100	1	55	1.462774	0.000103	0.814007
400	4.5	200	2.2	100	100	2	55	1.463583	0.000095	0.824566

FIG. 10

FIG. 11

FIG. 12

**Index and Dep Pate Drifts Pre & Post Anneal
Target 92-8 AKT 4300 Based Reactor**

FIG. 13

FIG. 14

Index Drift Control
83-17 Target/AKT 1600 Based Reactor
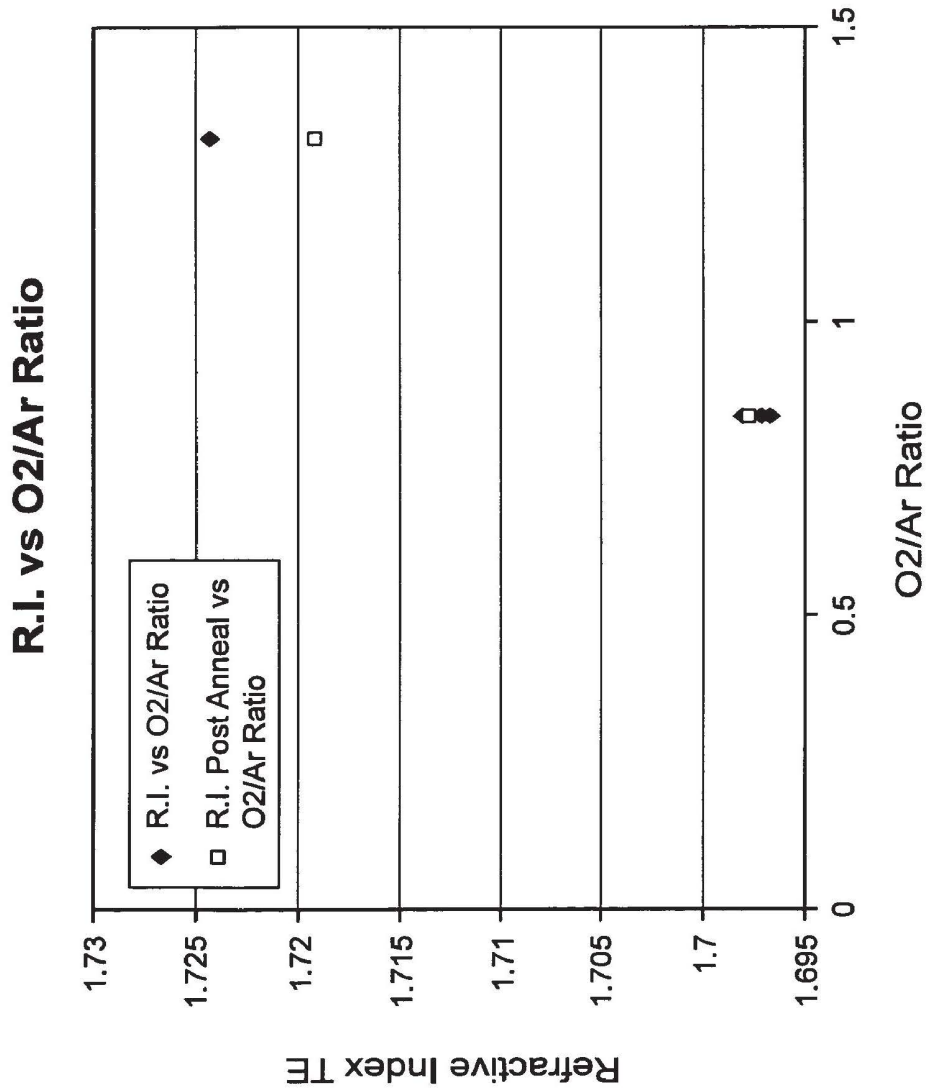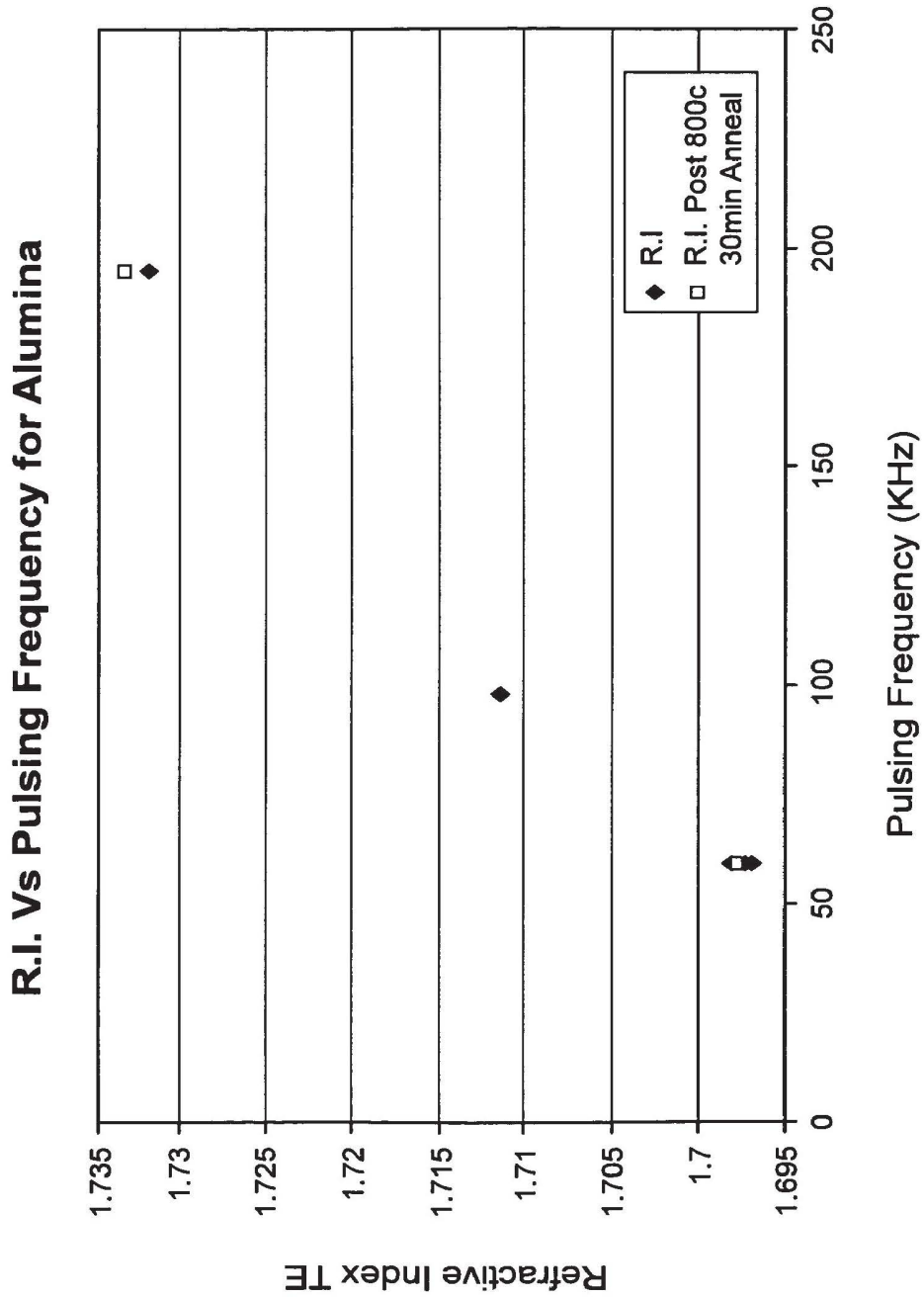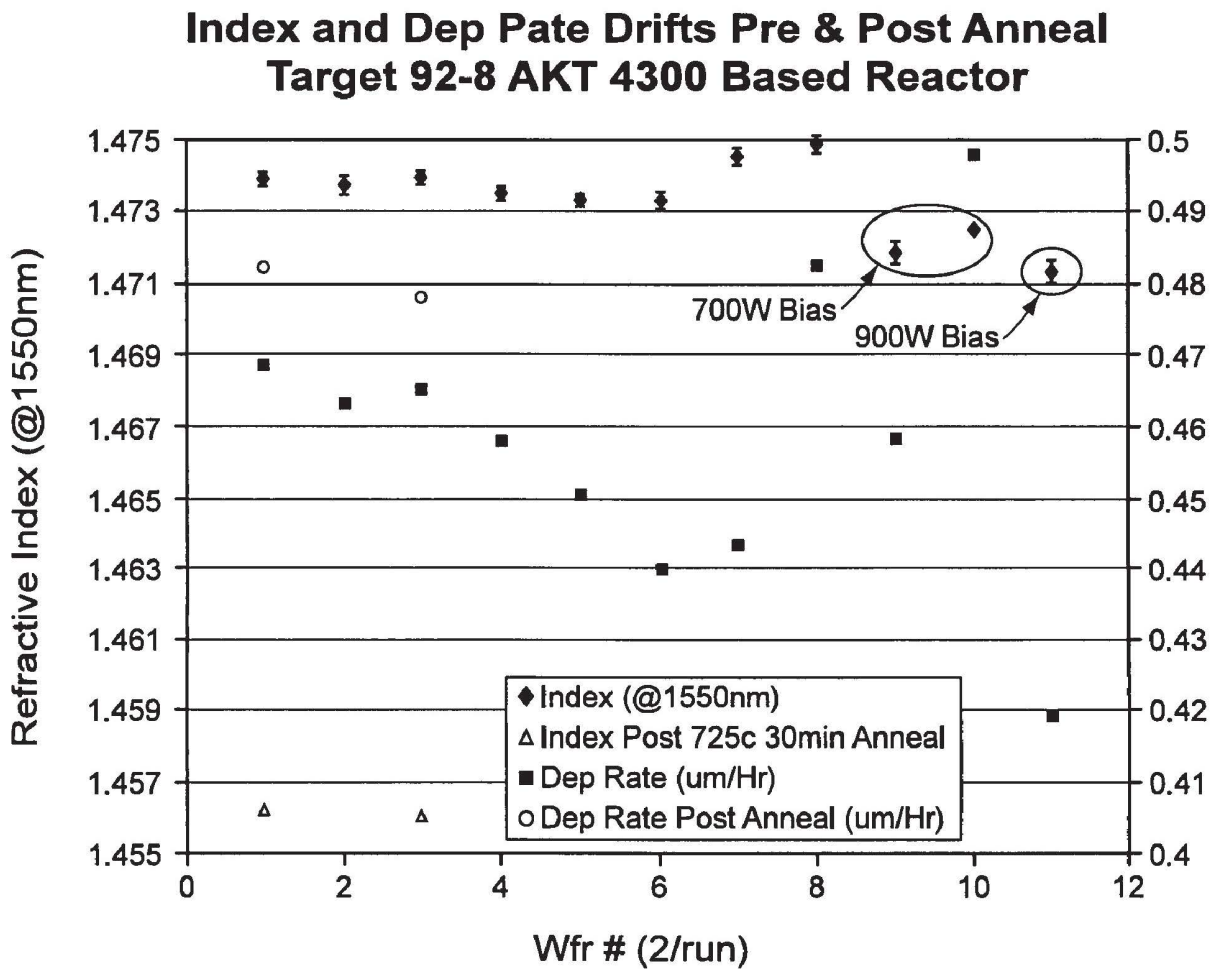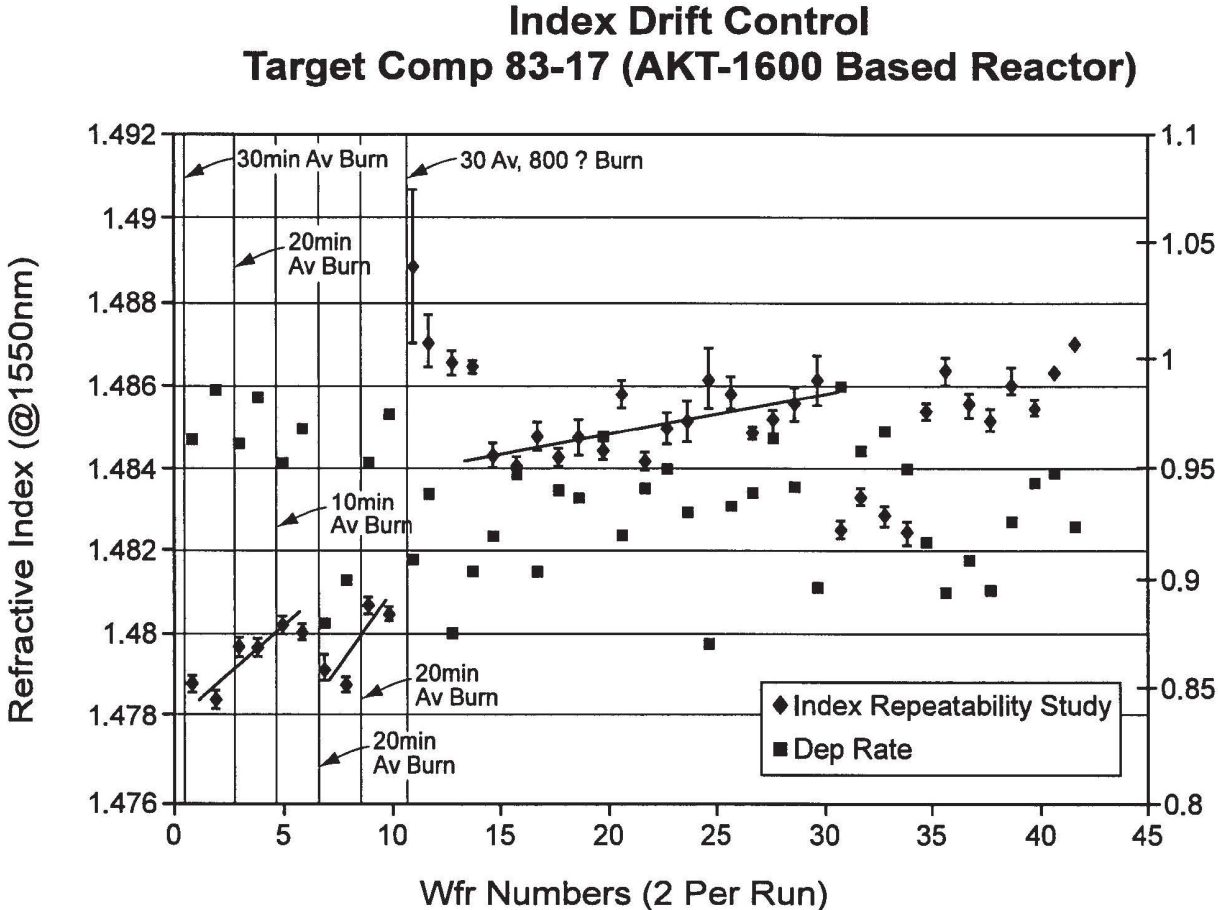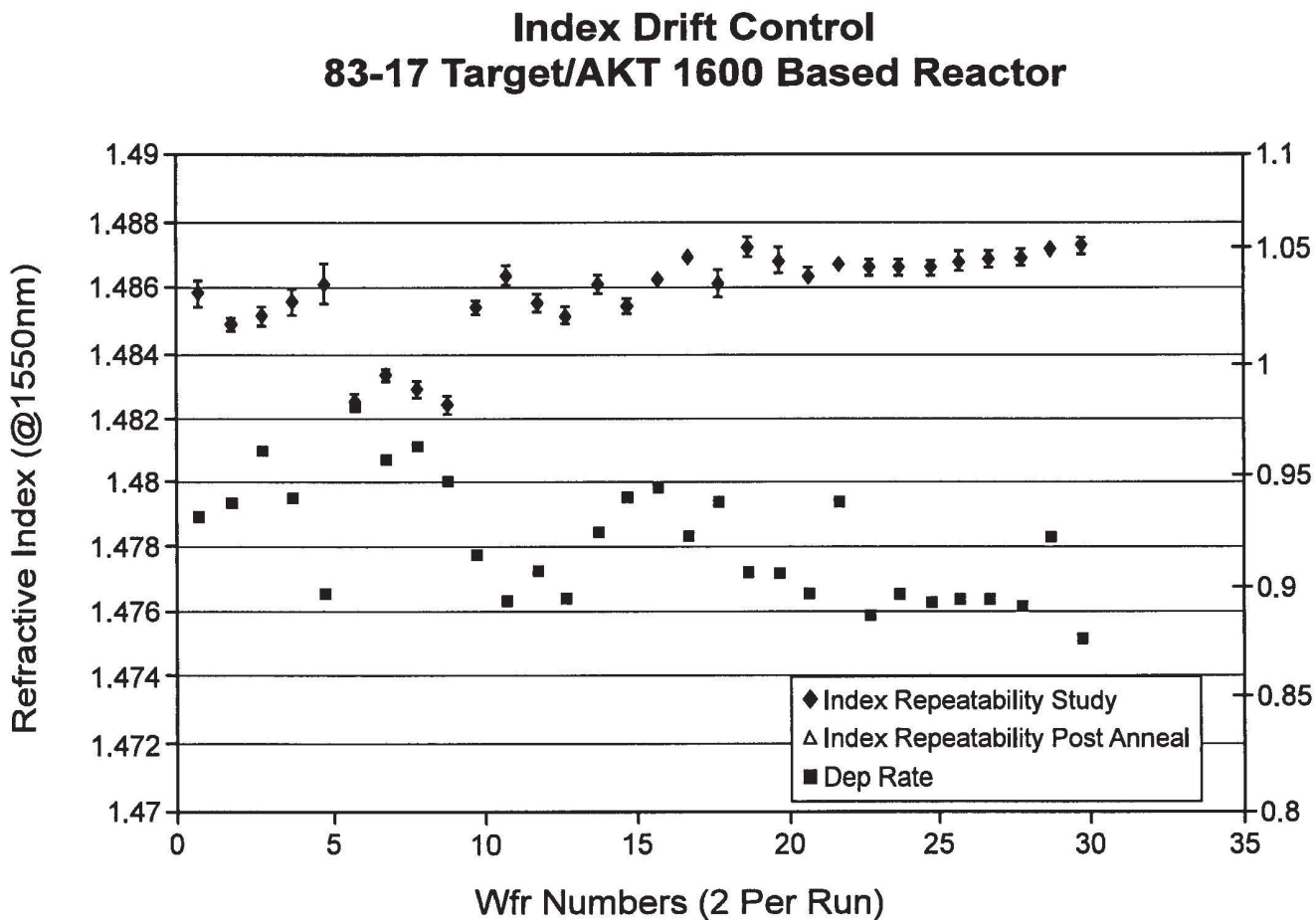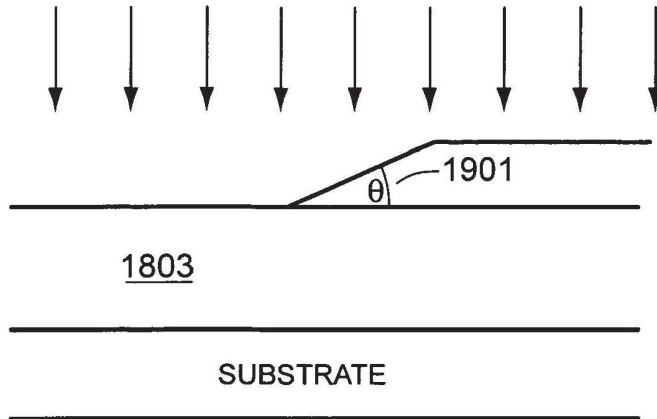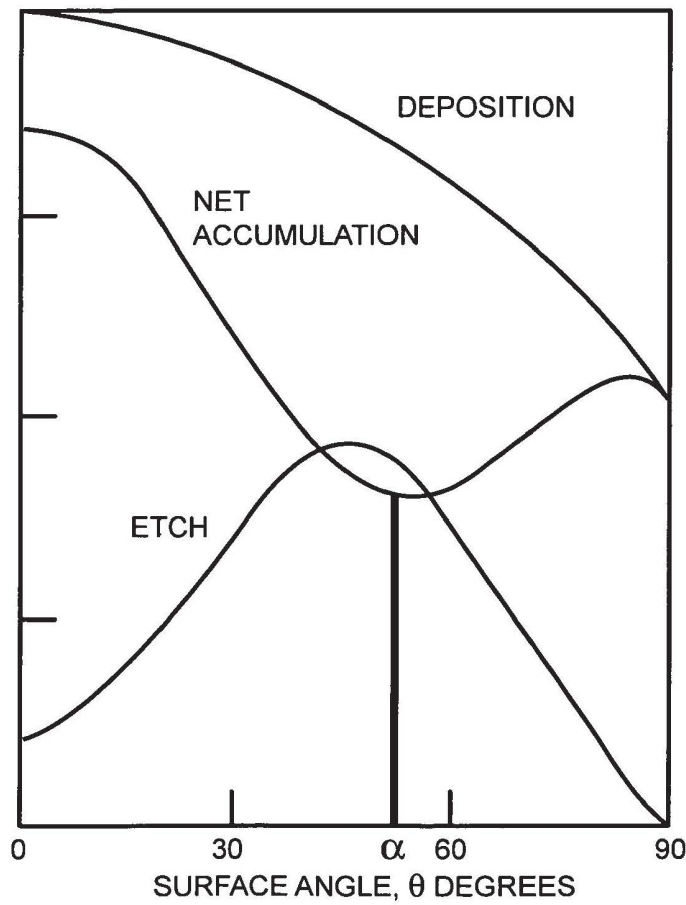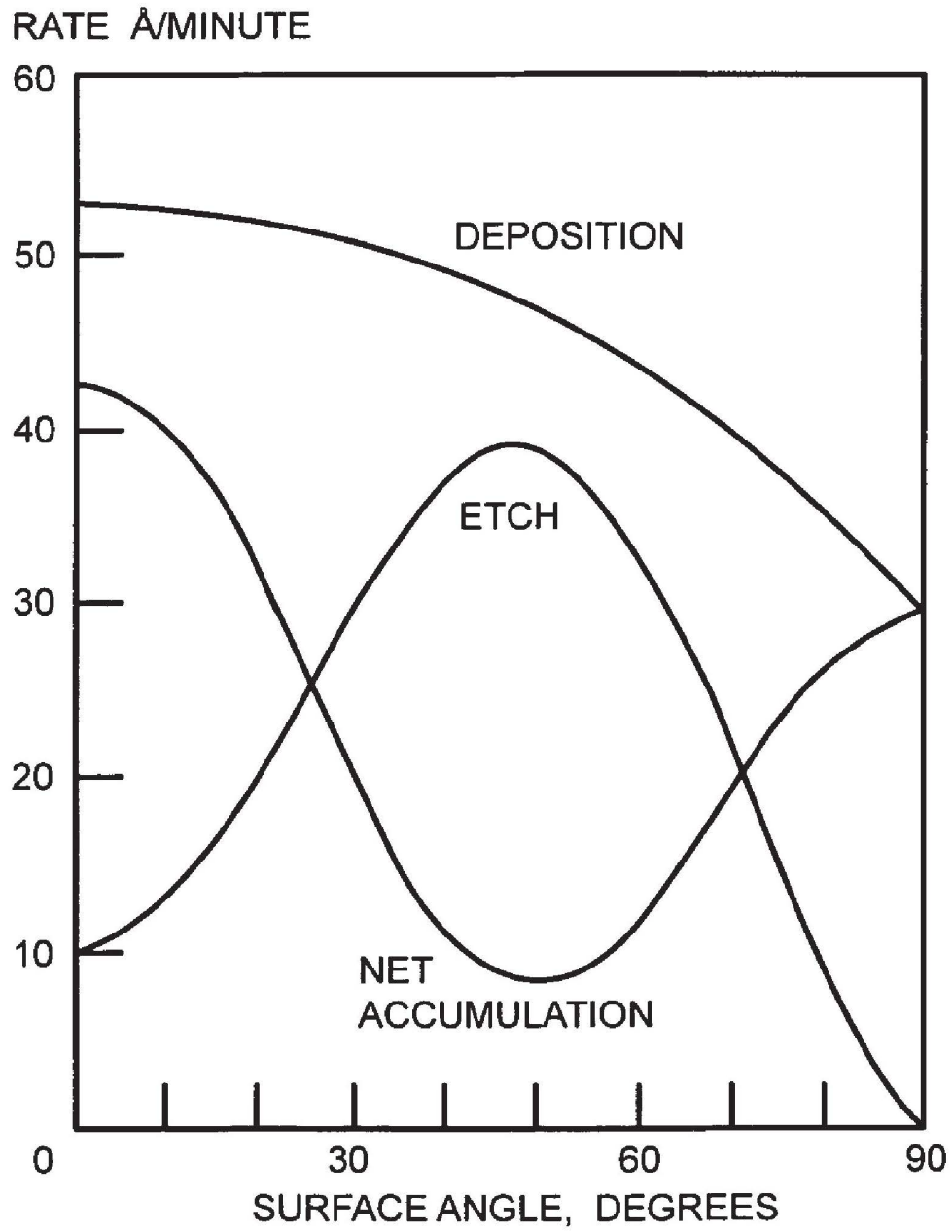
FIG. 15
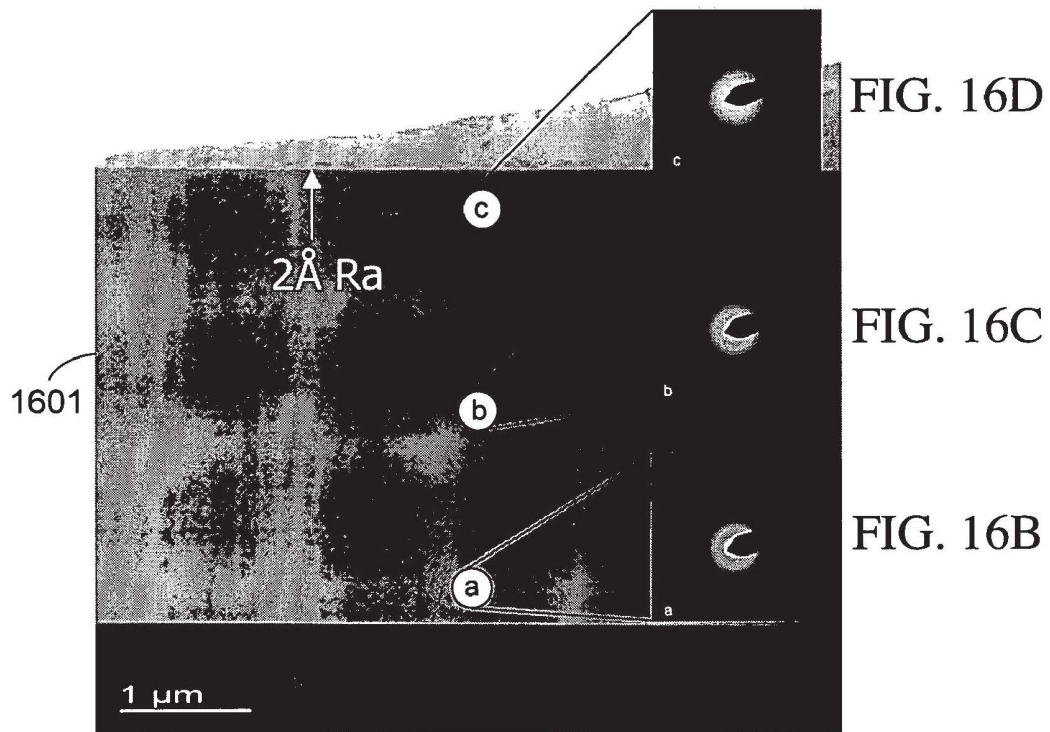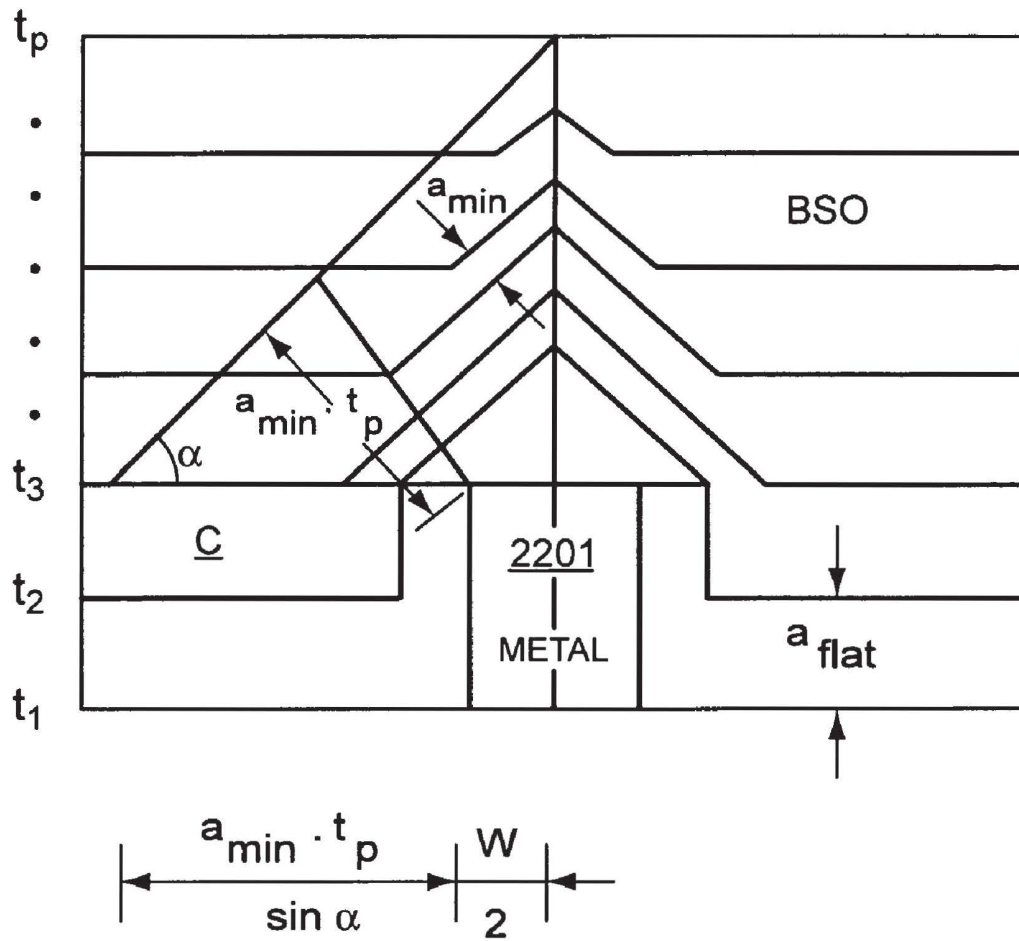
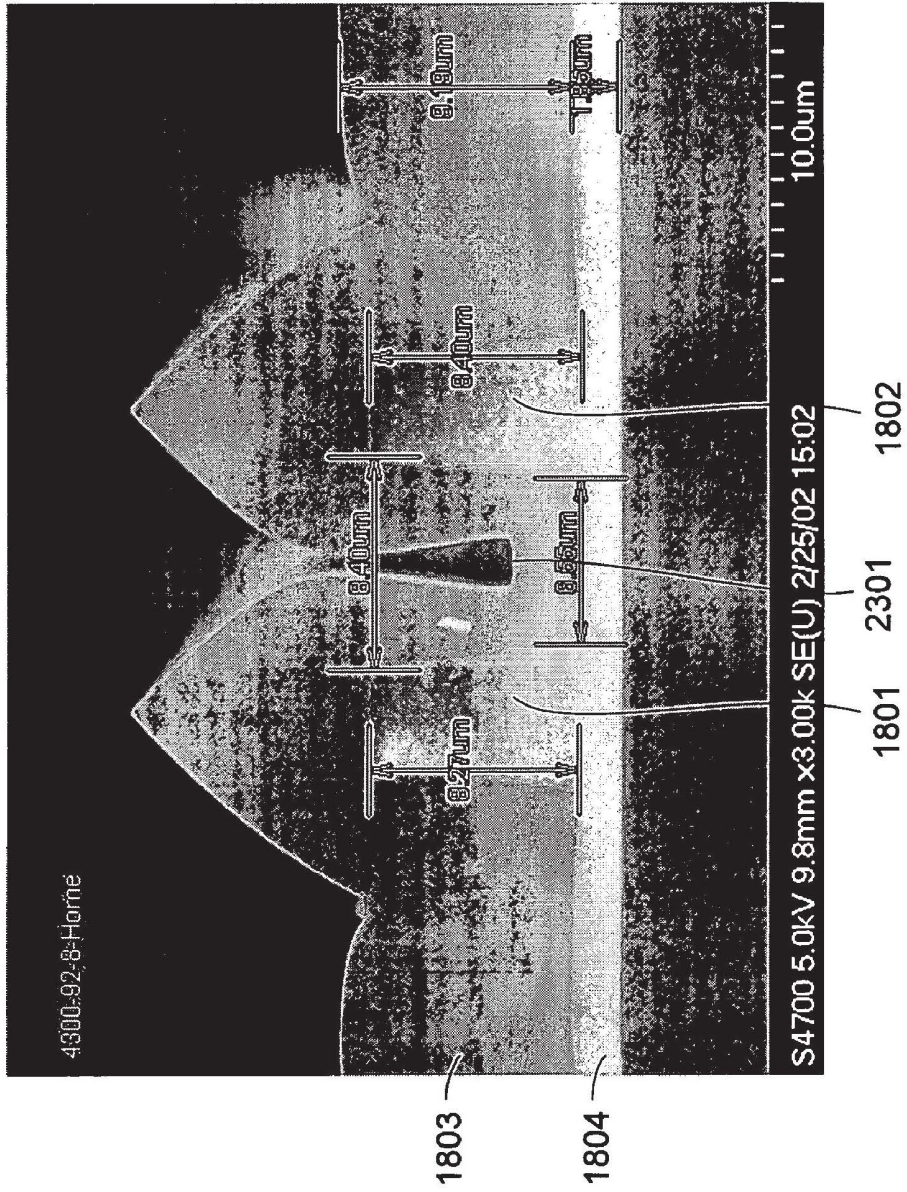FIG. 16A

SYMMORPHIX PVD ALUMINOSILICATE
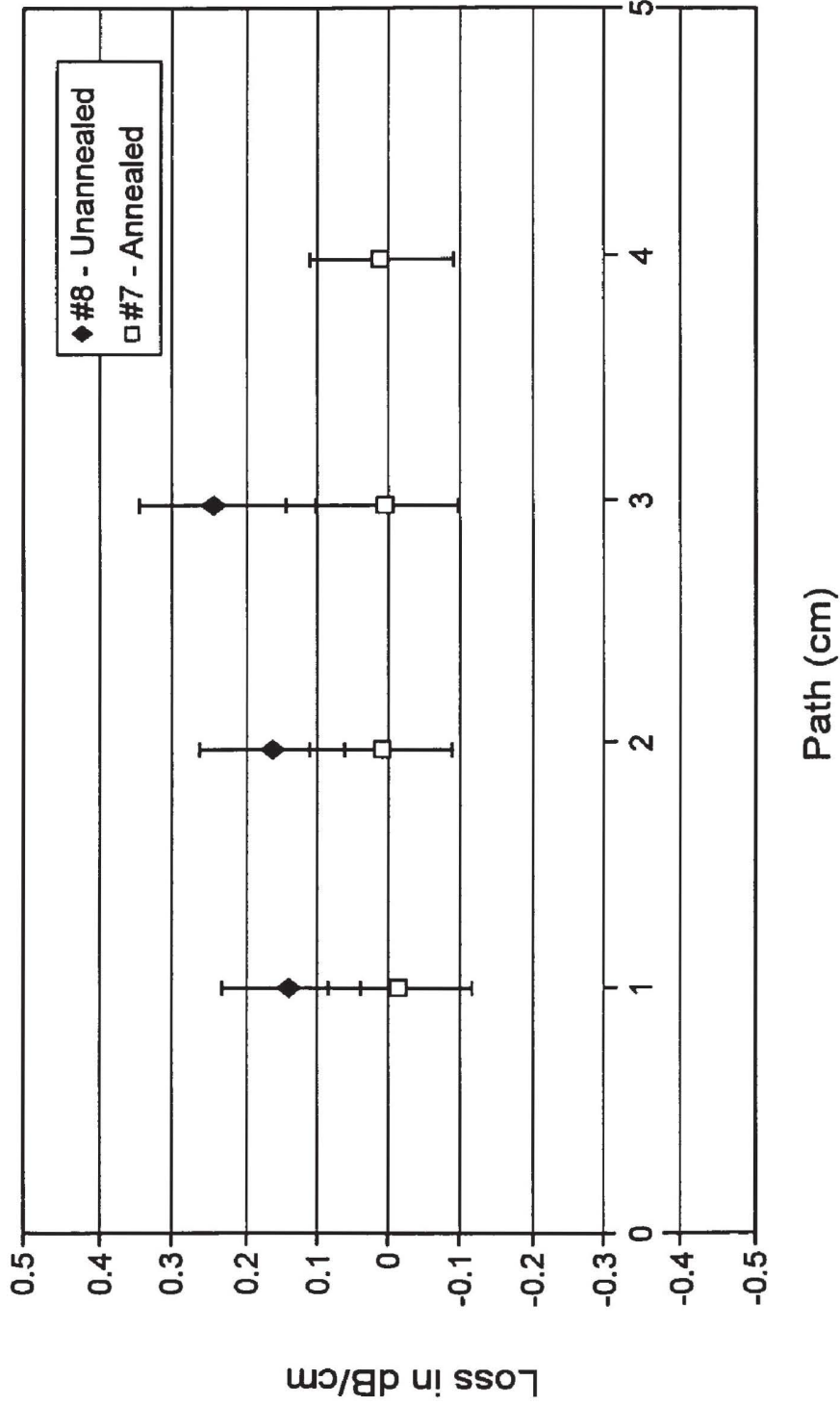
FIG. 17
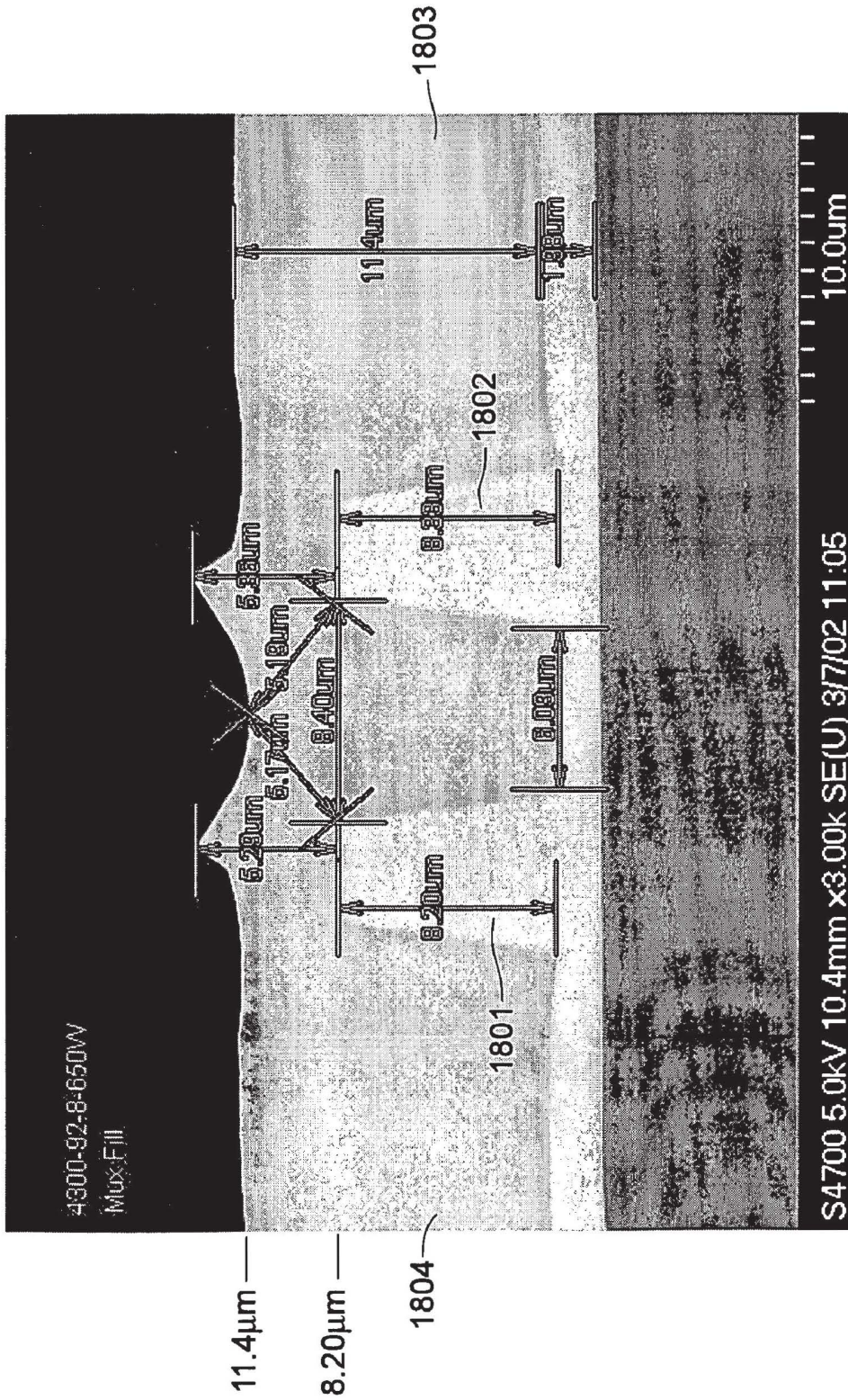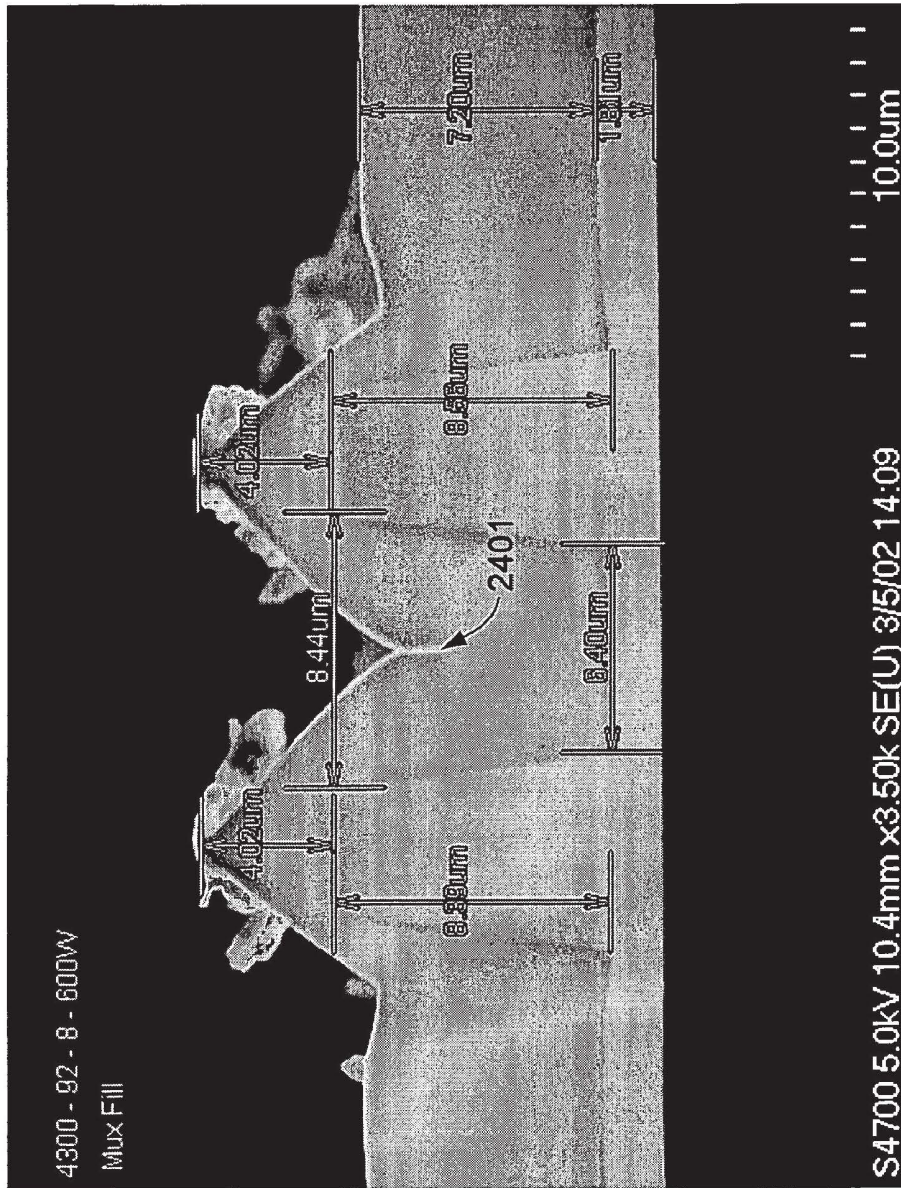
FIG. 18

FIG. 19

FIG. 20
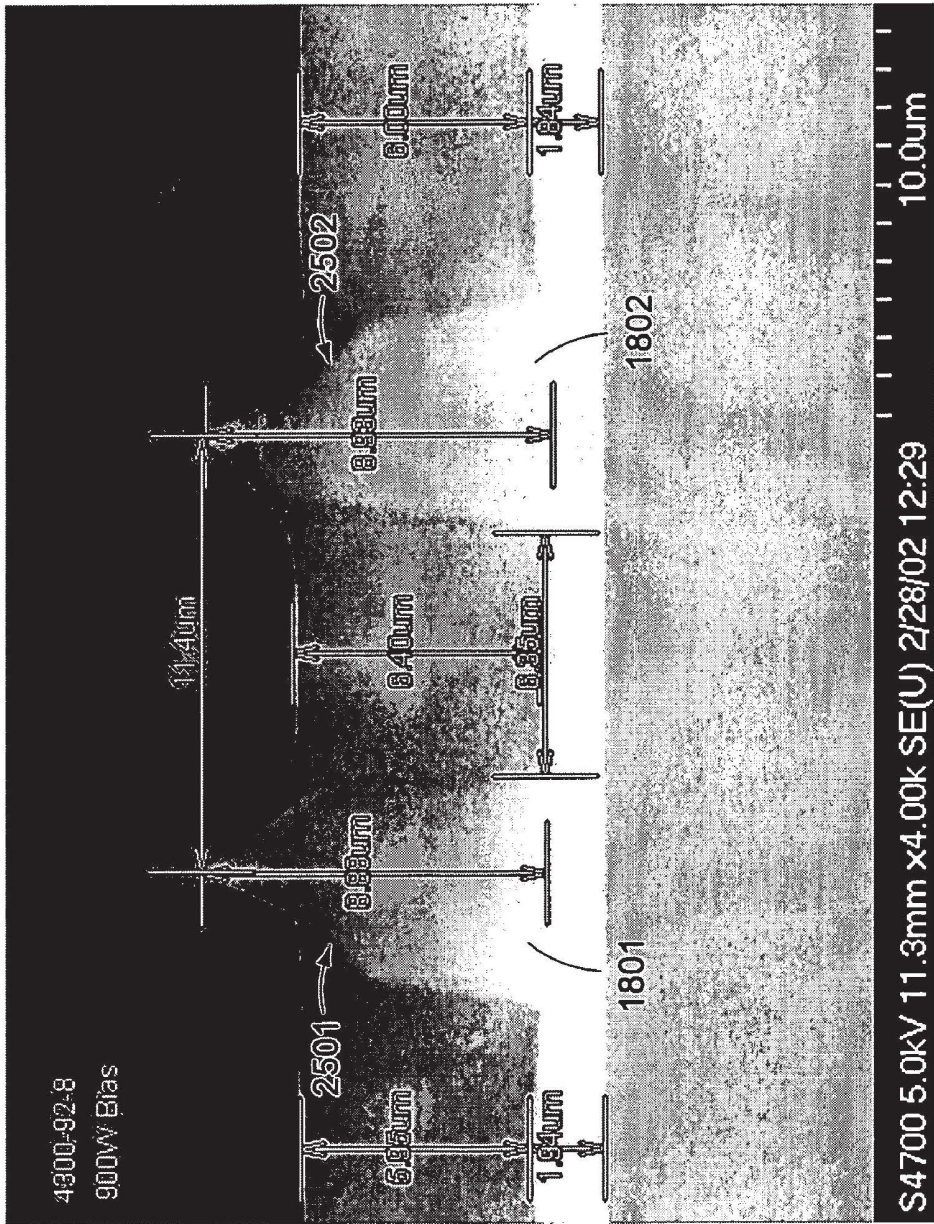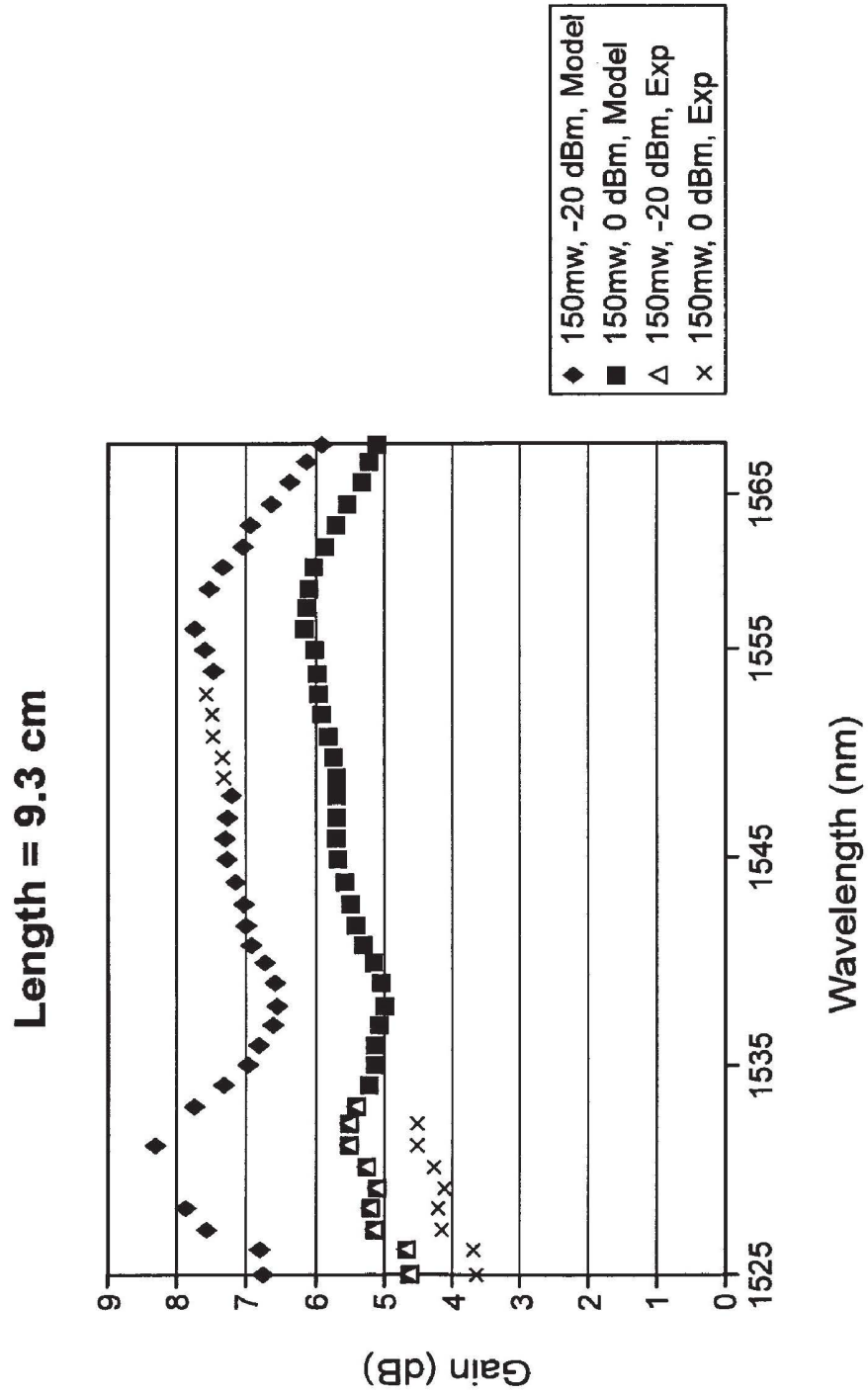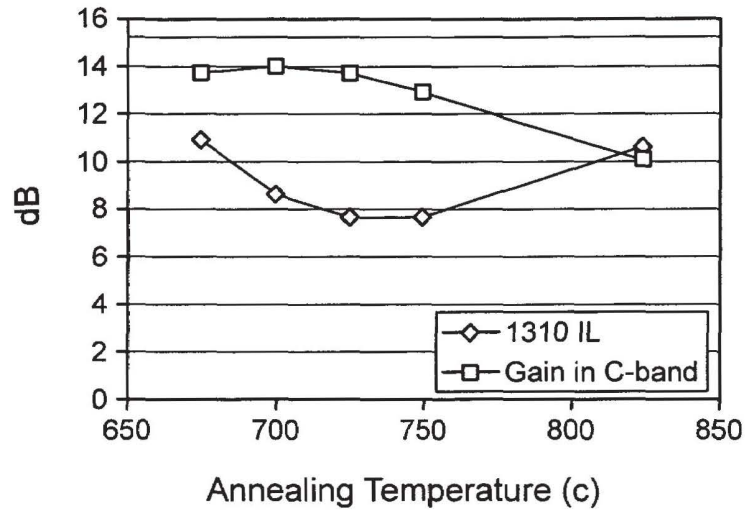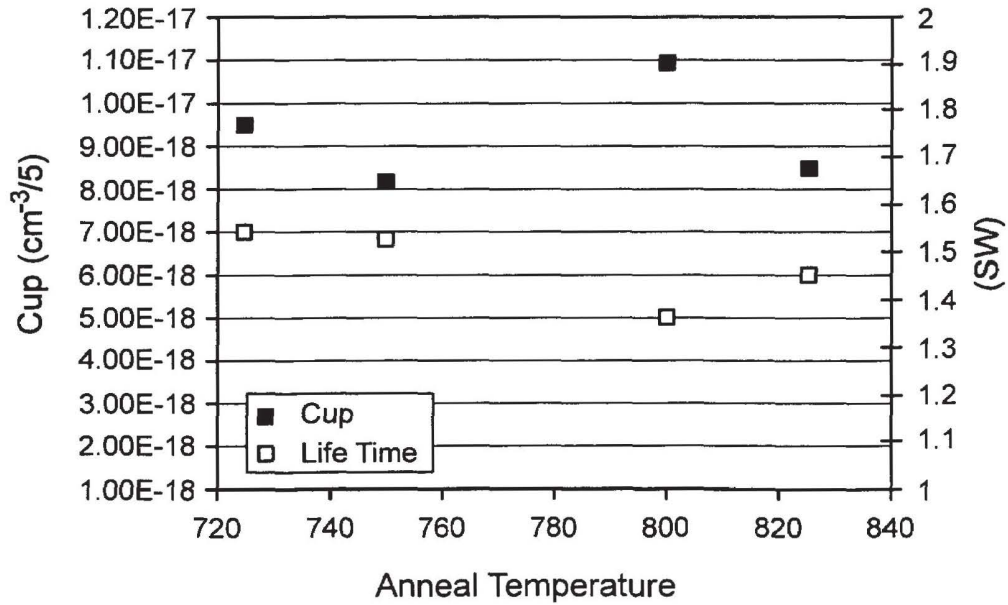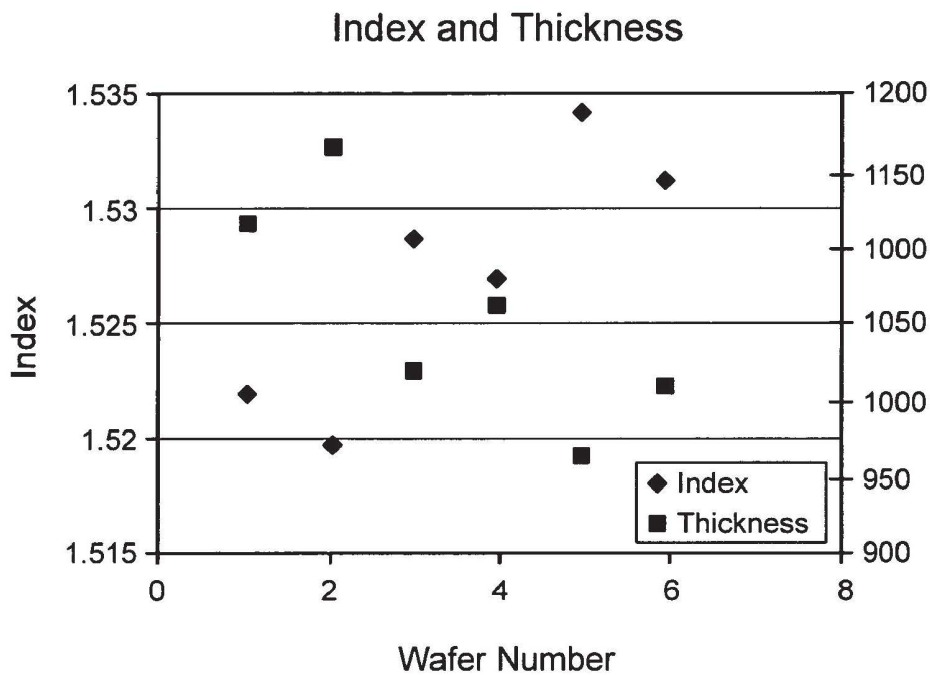
FIG. 21

FIG. 22

FIG. 23

FIG. 24

FIG. 25

FIG. 26

FIG. 27

Life Time and Up-conversion vs. Annealing
Temperature

FIG. 28

Index and Thickness

Wafer Number

FIG. 29
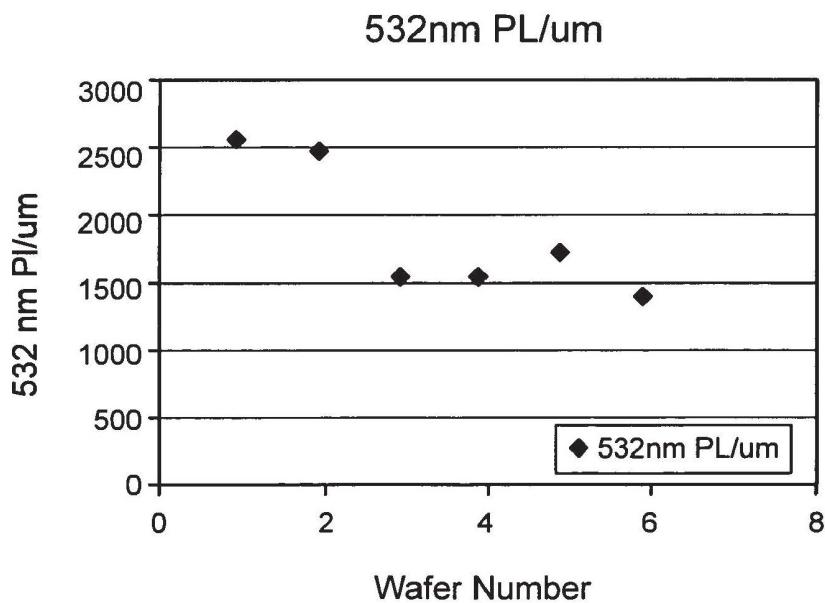
532nm PL/um

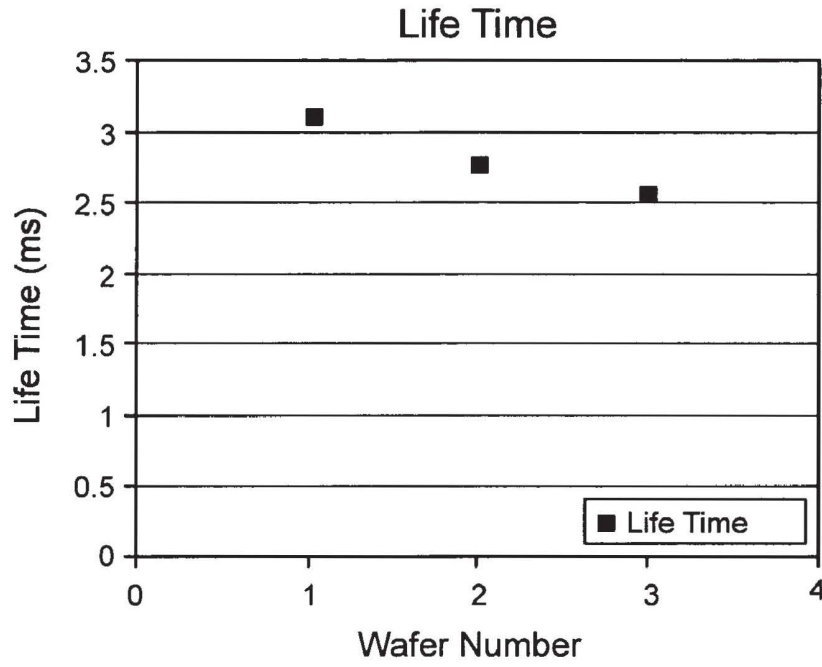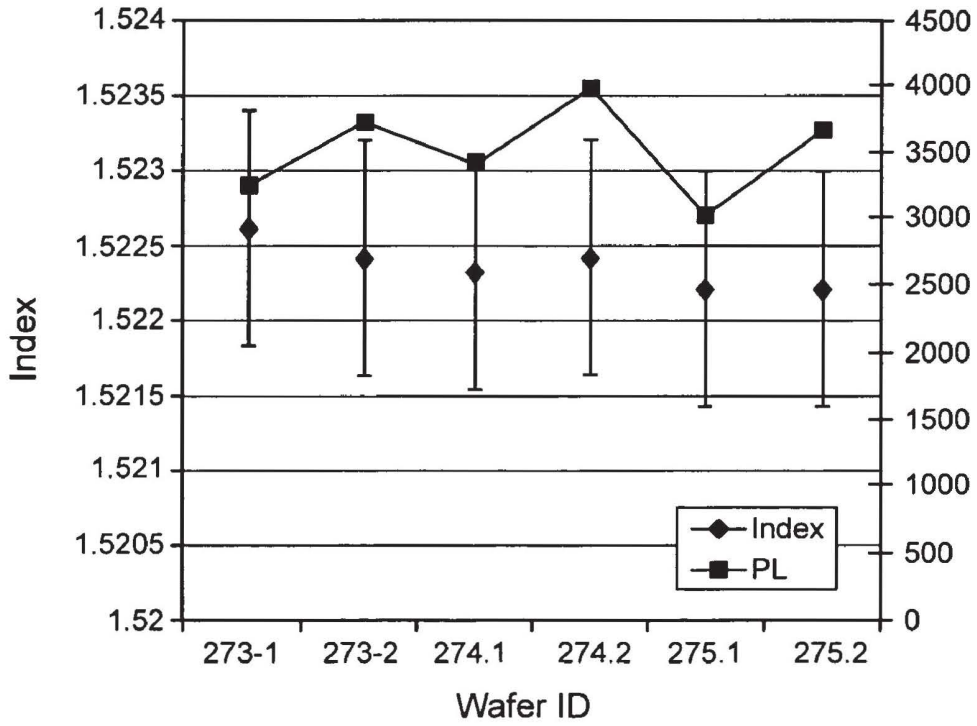**Wafer Number**

FIG. 30

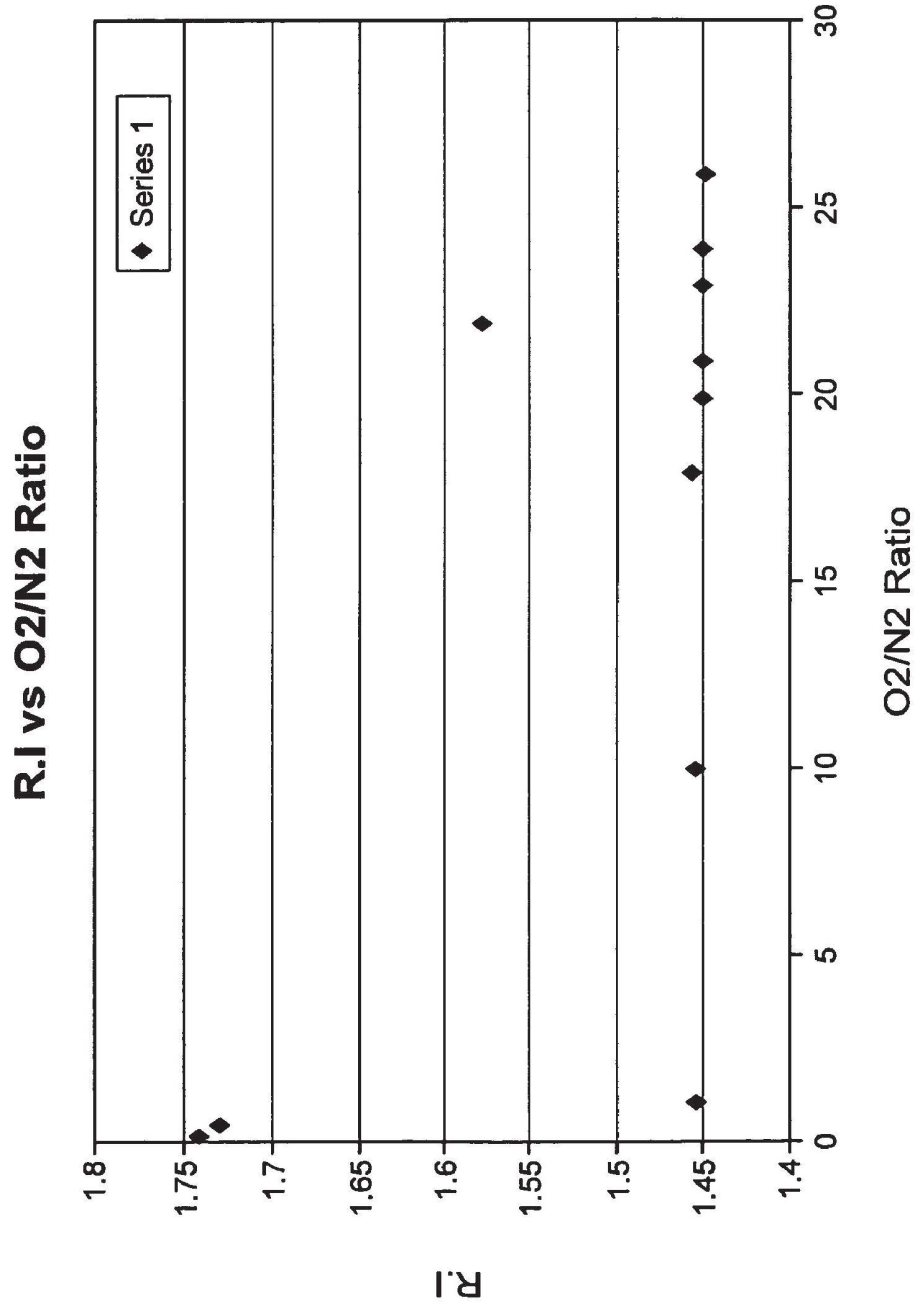## Life Time

FIG. 31

FIG. 32

FIG. 33

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BIASED PULSE DC REACTIVE SPUTTERING OF OXIDE FILMS

This is a continuation of application Ser. No. 10/101,863, filed Mar. 16, 2002 which is incorporated herein by reference.

BACKGROUND

1. Field of the Invention

The present invention relates to deposition of oxide and oxynitride films and, in particular, to deposition of oxide and oxynitride films by pulsed DC reactive sputtering.

2. Discussion of Related Art

Deposition of insulating materials and especially optical materials is technologically important in several areas including production of optical devices and production of semiconductor devices. In semiconductor devices, doped alumina silicates can be utilized as high dielectric insulators.

The increasing prevalence of fiber optic communications systems has created an unprecedented demand for devices for processing optical signals. Planar devices such as optical waveguides, couplers, splitters, and amplifiers, fabricated on planar substrates, like those commonly used for integrated circuits, and configured to receive and process signals from optical fibers are highly desirable. Such devices hold promise for integrated optical and electronic signal processing on a single semiconductor-like substance.

The basic design of planar optical waveguides and amplifiers is well known, as described, for example, in U.S. Pat. Nos. 5,119,460 and 5,563,979 to Bruce et al., 5,613,995 to Bhandarkar et al., 5,900,057 to Buchal et al., and 5,107,538 to Benton et al., to cite only a few. These devices, very generally, include a core region, typically bar shaped, of a certain refractive index surrounded by a cladding region of a lower refractive index. In the case of an optical amplifier, the core region includes a certain concentration of a dopant, typically a rare earth ion such as an erbium or praseodymium ion which, when pumped by a laser, fluoresces, for example, in the 1550 nm and 1300 nm wavelength ranges used for optical communication, to amplify the optical signal passing through the core.

As described, for example in the patents by Bruce et al., Bhandarkar et al, and Buchal et al., planar optical devices may be fabricated by process sequences including forming a layer of cladding material on a substrate; forming a layer of core material on the layer of cladding mater; patterning the core layer using a photolighotgraphic mask and an etching process to form a core ridge; and covering the core ridge with an upper cladding layer.

The performance of these planar optical devices depends sensitively on the value and uniformity of the refractive index of the core region and of the cladding region, and particularly on the difference in refractive index, Δn, between the regions. Particularly for passive devices such as waveguides, couplers, and splitters, Δn should be carefully controlled, for example to values within about 1%, and the refractive index of both core and cladding need to be highly uniform, for some applications at the fewer than parts per thousand level. In the case of doped materials forming the core region of planar optical amplifiers, it is important that the dopant be uniformly distributed so as to avoid non-radiative quenching or radiative quenching, for example by upconversion. The refractive index and other desirable properties of the core and cladding regions, such as physical and chemical uniformity, low stress, and high density, depend, of

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course, on the choice of materials for the devices and on the processes by which they are fabricated.

Because of their optical properties, silica and refractory oxides such as Al_2O_3, are good candidate materials for planar optical devices. Further, these oxides serve as suitable hosts for rare earth dopants used in optical amplifiers. A common material choice is so-called low temperature glasses, doped with alkali metals, boron, or phosphorous, which have the advantage of requiring lower processing temperatures. In addition, dopants are used to modify the refractive index. Methods such as flame hydrolysis, ion exchange for introducing alkali ions in glasses, sputtering, and various chemical vapor deposition processes (CVD) have been used to form films of doped glasses. However, dopants such as phosphorous and boron are hygroscopic, and alkalis are undesirable for integration with electronic devices. Control of uniformity of doping in CVD processes can be difficult and CVD deposited films can have structural defects leading to scattering losses when used to guide light. In addition, doped low temperature glasses may require further processing after deposition. A method for eliminating bubbles in thin films of sodium-boro-silicate glass by high temperature sintering is described, for example, in the '995 patent to Bhandarkar et al.

Typically, RF sputtering has been utilized for deposition of oxide dielectric films. However, RF sputtering utilizes ceramic targets which are typically formed of multiple smaller tiles. Since the tiles can not be made very large, there may be a large problem of arcing between tiles and therefore contamination of the deposited film due to this arcing. Further, the reactors required for RF sputtering tend to be rather complicated. In particular, the engineering of low capacitance efficient RF power distribution to the cathode is difficult in RF systems. Routing of low capacitance forward and return power into a vacuum vessel of the reaction chamber often exposes the power path in such a way that diffuse plasma discharge is allowed under some conditions of impedance tuning of the matching networks.

Therefore, there is a need for new methods of depositing oxide and oxynitride films and for forming planar optical devices.

SUMMARY

In accordance with the present invention, a sputtering reactor apparatus for depositing oxide and oxynitride films is presented. Further, methods for depositing oxide and oxynitride films for optical waveguide devices are also presented. A sputtering reactor according to the present invention includes a pulsed DC power supply coupled through a filter to a target and a substrate electrode coupled to an RF power supply. A substrate mounted on the substrate electrode is therefore supplied with a bias from the RF power supply.

The target can be a metallic target made of a material to be deposited on the substrate. In some embodiments, the metallic target is formed from Al, Si and various rare-earth ions. A target with an erbium concentration, for example, can be utilized to deposit a film that can be formed into a waveguide optical amplifier.

A substrate can be any material and, in some embodiments, is a silicon wafer. In some embodiments, RF power can be supplied to the wafer. In some embodiments, the wafer and the electrode can be separated by an insulating glass.

In some embodiments, up to about 10 kW of pulsed DC power at a frequency of between about 40 kHz and 350 kHz

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and a reverse pulse time of up to about 5 µs is supplied to the target. The wafer can be biased with up to about several hundred watts of RF power. The temperature of the substrate can be controlled to within about 10° C. and can vary from about −50° C. to several hundred degrees C. Process gasses can be fed into the reaction chamber of the reactor apparatus. In some embodiments, the process gasses can include combinations of Ar, N_2, O_2, C_2F_6, CO_2, CO and other process gasses.

Several material properties of the deposited layer can be modified by adjusting the composition of the target, the composition and flow rate of the process gasses, the power supplied to the target and the substrate, and the temperature of the substrate. For example, the index of refraction of the deposited layer depends on deposition parameters. Further, in some embodiments stress can be relieved on the substrate by depositing a thin film of material on a back side of the wafer. Films deposited according to the present invention can be utilized to form optical waveguide devices such as multiplexers and rare-earth doped amplifiers.

These and other embodiments, along with examples of material layers deposited according to the present invention, are further described below with respect to the following figures.

BRIEF DESCRIPTION OF THE FIGURES

FIGS. 1A and 1B show a pulsed DC sputtering reactor according to the present invention.

FIG. 2 shows a planar view of target utilized in a reactor as shown in FIGS. 1A and 1B.

FIG. 3 shows a cross-section view of an example target utilized in a reactor as shown in FIGS. 1A and 1B.

FIG. 4 shows a flow chart of an embodiment of a process for depositing a film on a substrate according to the present invention.

FIG. 5 shows a hysterises curve of target voltage versus oxygen flow rates for an example target in an embodiment of a reactor according to the present invention.

FIG. 6 shows a photo-luminescence and lifetimes of a film deposited in a process according to the present invention as a function of after deposition anneal temperature.

FIG. 7 shows the relationship between the index of refraction of a film as a function of deposited oxide layers according to the present invention and due to oxide build-up on the target.

FIG. 8 shows a graph of the index of refraction of a film deposited according to the present invention as a function of the aluminum content in a composite Al/Si target.

FIG. 9 shows a graph of typical indices of refraction of material layers deposited according to the present invention.

FIG. 10 shows a table of indices of refraction for a silica layer deposited according to the present invention as a function of different process parameters.

FIG. 11 shows the refractive indices as a function of O_2/Ar ratio utilized in an Alumina process according to the present invention.

FIG. 12 shows the refractive indices as a function of DC pulsed power frequency for an Alumina layer deposited according to the present invention.

FIG. 13 shows variation in the refractive index over time during repeated depositions from a single target.

FIG. 14 shows variation in refractive index over time for repeated depositions from a target of another material layer according to the present invention.

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FIG. 15 shows the variation refractive index over time for repeated depositions from a target of another material layer according to the present invention.

FIG. 16A through 16D shows a TEM film deposited according to the present invention.

FIG. 17 shows the transparency of a film deposited according to the present invention.

FIG. 18 shows an uppercladding layer deposited according to the present invention over a multiple-waveguide structure such that the deposited layer is substantially planarized.

FIG. 19 illustrates the deposition of a film over a waveguide structure.

FIGS. 20 and 21 illustrate different etch and deposition rates for deposition of films as a function of the surface angle of the film.

FIG. 22 illustrates calculation of the planarization time for a particular deposition process.

FIGS. 23 through 25 through illustrate adjustment of process parameters in order to achieve planarization of a film deposited over a waveguide structure according to the present invention.

FIG. 26 shows the gain characteristics of an erbium doped waveguide amplifier formed of films depositions according to the present invention.

FIG. 27 shows gain, insertion loss of a waveguide with an active core deposited according to the present invention.

FIG. 28 shows up-conversion constants, and lifetimes of the active core layer of FIG. 27 deposited according to the present invention.

FIG. 29 shows drift in the index of refraction with subsequent depositions for films deposited from a target according to the present invention.

FIG. 30 shows drift in the photoluminescence with subsequent depositions according to the present invention.

FIG. 31 shows drift in the excited state lifetime with subsequent depositions according to the present invention.

FIG. 32 shows stabilization of the index of refraction in subsequent depositions.

FIG. 33 shows the index of refraction of a film formed from a pure silicon target as a function of the ratio of O_2/N_2 in the process gas.

In the figures, elements having the same designation have the same or similar function.

DETAILED DESCRIPTION

Reactive DC magnetron sputtering of nitrides and carbides is a widely practiced technique, but the reactive dc magnetron sputtering of nonconducting oxides is done rarely. Films such as aluminum oxide are almost impossible to deposit by conventional reactive DC magnetron sputtering due to rapid formation of insulating oxide layers on the target surface. The insulating surfaces charges up and result in arcing during process. This arcing can damage the power supply, produce particles and degrade the properties of deposited oxide films.

RF sputtering of oxide films is discussed in application Ser. No. 09/903,050 (the '050 application) (now U.S. Pat. No. 6,506,289) by Demaray et al., entitled "Planar Optical Devices and Methods for Their Manufacture," assigned to the same assignee as is the present invention, herein incorporated by reference in its entirety. Further, targets that can be utilized in a reactor according to the present invention are discussed in U.S. application Ser. No. 10/101,341 (the '341 application), filed concurrently with the present disclosure, assigned to the same assignee as is the present invention,

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herein incorporated by reference in its entirety. A gain-flattened amplifier formed of films deposited according to the present invention are described in U.S. application Ser. No. 10/101,493 (the '493 application), filed concurrently with the present disclosure, assigned to the same assignee as is the present invention, herein incorporated by reference in its entirety. Further, a mode size converter formed with films deposited according to the present invention is described in U.S. application Ser. No. 10/101,492 (the '492 application), filed concurrently with the present disclosure, assigned to the same assignee as is the present invention, herein incorporated by reference in its entirety.

FIG. **1**A 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 (400×500 mm substrate size) system from Applied Komatsu or an AKT-4300 (600×720 mm substrate size) system from Applied Komatsu, Santa Clara, Calif. The AKT-1600 reactor, for example, has three deposition chambers connected by a vacuum transport chamber. These Komatsu reactors can be modified such that pulsed DC power is supplied to the target and RF power is supplied to the substrate during deposition of a material film.

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**.

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 depend on target material, cathode current and reverse time. High quality oxide films can be made using reactive pulse DC magnetron sputtering in apparatus **10**.

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. 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 between 0 and 5 μs.

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

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

However, both RF and pulsed DC deposited films are not fully dense and most likely have columnar structures. These

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columnar structures are detrimental for optical wave guide applications due to the scattering loss caused by the structure. By applying a RF bias on wafer **16** during deposition, the deposited film can be dandified by energetic ion bombardment and the columnar structure can be substantially eliminated.

In the AKT-1600 based system, for example, target **12** can have an active size of about 675.70×582.48 by 4 mm in order to deposit films on substrate **16** that have dimension about 400×500 mm. The temperature of substrate **16** can be held at between −50C and 500C. 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 0.7 and 6 millitorr. Magnet **20** provides a magnetic field of strength between about 400 and about 600 Gauss directed in the plane of target **12** and is moved across target **12** at a rate of less than about 20-30 sec/scan. In some embodiments utilizing the AKT 1600 reactor, magnet **20** can be a race-track shaped magnet with dimension about 150 mm by 600 mm.

A top down view of magnet **20** and wide area target **12** is shown in FIG. **2**. 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 FIG. **1**B that is exposed to a uniform plasma condition. In some implementations, carrier **17** can be coextensive with region **52**. Region **24** shown in FIG. **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. FIG. **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.

In some embodiments, magnet **20** extends beyond area **52** in one direction, the Y direction in FIG. **2**, so that scanning is necessary in only one direction, the X direction, to provide a time averaged uniform magnetic field. As shown in FIGS. **1**A and **1**B, 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**.

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

Target **12** can be formed of any materials, but is typically metallic materials such as, for example, combinations of Al and Si. 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,

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the rare earth ions can be pre-alloyed with the metallic host components to form intermetallics. See the '341 application.

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. FIG. 3A shows an embodiment of target 12 formed with individual tiles 30 mounted on a cooled backplate 25. In order to form a wide area target of an alloy target material, the consolidated material of individual tiles 30 should first be uniform to the grain size of the powder from which it is formed. It also should be formed into a structural material capable of forming and finishing to a tile shape having a surface roughness on the order of the powder size from which it is consolidated. 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 30, for example between 2 to 20 individual tiles 30. Tiles 30 are finished to a size so as to provide a margin of non-contact, tile to tile, 29 in FIG. 3A, less than about 0.010" to about 0.020" or less than half a millimeter so as to eliminate plasma processes between adjacent ones of tiles 30. The distance between tiles 30 of target 12 and the dark space anode or ground shield 19, in FIG. 1B can be somewhat larger so as to provide non contact assembly or provide for thermal expansion tolerance during process chamber conditioning or operation.

Several useful examples of target 12 that can be utilized in apparatus 10 according to the present invention include the following targets compositions: (Si/Al/Er/Yb) being about (57.0/41.4/0.8/0.8), (48.9/49/1.6/0.5), (92/8/0/0), (60/40/0/0), (50/50/0/0), (65/35/0/0), (70/30/0,0), and (50,48.5/1.5/0) cat. %, to list only a few. These targets can be referred to as the 0.8/0.8 target, the 1.6/0.5 target, the 92-8 target, the 60-40 target, the 50-50 target, the target, the 70-30 target, and the 1.5/0 target, respectively. The 0.8/0.8, 1.6/0.5, and 1.5/0 targets can be made by pre-alloyed targets formed from an atomization and hot-isostatic pressing (HIPing) process as described in the '341 application. The remaining targets can be formed, for example, by HIPing. Targets formed from Si, Al, Er and Yb can have any composition. In some embodiments, the rare earth content can be up to 10 cat. % of the total ion content in the target. Rare earth ions are added to form active layers for amplification. Targets utilized in apparatus 10 can have any composition and can include ions other than Si, Al, Er and Yb, including: Zn, Ga, Ge, P, As, Sn, Sb, Pb, Ag, Au, and rare earths: Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy Ho, Er, Tm Yb and Lu.

Optically useful materials to be deposited onto substrate 16 include oxides, fluorides, sulfides, nitrides, phosphates, sulfates, and carbonates, as well as other wide band gap semiconductor materials. To achieve uniform deposition, target 12, itself can be chemically uniform and of uniform thickness over an extended area.

Target 12 can be a composite target fabricated from individual tiles, precisely bonded together on a backing plate with minimal separation, as is discussed further with respect to FIG. 3. In some embodiments, the mixed intermetallics can be plasma sprayed directly onto a backing plate to form target 12. The complete target assembly can also includes structures for cooling the target, embodiments of which have been described in U.S. Pat. No. 5,565,071 to Demaray et al, and incorporated herein by reference.

Substrate 16 can be a solid, smooth surface. Typically, substrate 16 can be a silicon wafer or a silicon wafer coated with a layer of silicon oxide formed by a chemical vapor deposition process or by a thermal oxidation process. Alternatively, substrate 16 can be a glass, such as Corning 1737 (Corning Inc., Elmira, N.Y.), a glass-like material, quartz, a

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metal, a metal oxide, or a plastic material. Substrate 16 can be supported on a holder or carrier sheet that may be larger than substrate 16. Substrate 16 can be electrically biased by power supply 18.

In some embodiments, the area of wide area target 12 can be greater than the area on the carrier sheet on which physically and chemically uniform deposition is accomplished. Secondly, in some embodiments a central region on target 12, overlying 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 uniformly deposited film can be 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% or 10%. 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 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.

As shown in FIG. 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.

Other approaches to providing a uniform condition of sputter erosion rely on creating a large uniform magnetic field or a scanning magnetic field that produces a time-averaged, uniform magnetic field. For example, rotating magnets or electromagnets can be utilized to provide wide areas of substantially uniform target erosion. For magnetically enhanced sputter deposition, a scanning magnet magnetron source can be used to provide a uniform, wide area condition of target erosion.

As illustrated in FIG. 1A, apparatus 10 can include a scanning magnet magnetron source 20 positioned above target 12. An embodiment of a scanning magnetron source used for dc sputtering of metallic films is described in U.S. Pat. No. 5,855,744 to Halsey, et. al., (hereafter '744), which is incorporated herein by reference in its entirety. The '744 patent demonstrates the improvement in thickness uniformity that is achieved by reducing local target erosion due to magnetic effects in the sputtering of a wide area rectangular target. As described in the '744 patent, by reducing the magnetic field intensity at these positions, the local target

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erosion was decreased and the resulting film thickness nonuniformity was improved from 8%, to 4%, over a rectangular substrate of 400×500 mm.

The process gas utilized in reactor 10 includes an inert gas, typically argon, used as the background sputtering gas. Additionally, with some embodiments of target 12, reactive components such as, for example, oxygen may be added to the sputtering gas. Other gasses such as N_2, NH_3, CO, NO, CO_2, halide containing gasses other gas-phase reactants can also be utilized. The deposition chamber can be operated at low pressure, often between about 0.5 millitorr and 8-10 millitorr. Typical process pressure is below about 3-5 millitorr where there are very few collisions in the gas phase, resulting in a condition of uniform "free molecular" flow. This ensures that the gas phase concentration of a gaseous component is uniform throughout the process chamber. For example, background gas flow rates in the range of up to about 200 sccm, used with a pump operated at a fixed pumping speed of about 50 liters/second, result in free molecular flow conditions.

The distance d, in FIG. 1A, between target 12 and substrate 16 can, in some embodiments, be varied between about 4 cm and about 9 cm. A typical target to substrate distance d is about 6 cm. The target to substrate distance can be chosen to optimize the thickness uniformity of the film. At large source to substrate distances the film thickness distribution is dome shaped with the thickest region of the film at the center of the substrate. At close source to substrate distance the film thickness is dish shaped with the thickest film formed at the edge of the substrate. The substrate temperature can be held constant in the range of about −40° C. to about 550° C. and can be maintained at a chosen temperature to within about 10° C. by means of preheating substrate 16 and the substrate holder prior to deposition. During the course of deposition, the heat energy impressed upon the substrate by the process can be conducted away from substrate 16 by cooling the table on which substrate 16 is positioned during the process, as known to those skilled in the art. The process is performed under conditions of uniform gas introduction, uniform pumping speed, and uniform application of power to the periphery of the target as known to skilled practitioners.

The speed at which a scanning magnet 20 can be swept over the entire target can be determined such that a layer thickness less than about 5 to 10 Å, corresponding roughly to two to four monolayers of material, is deposited on each scan. Magnet 20 can be moved at rates up to about 30 sec/one-way scan and typically is moved at a rate of about 4 sec/one-way scan. The rate at which material is deposited depends on the applied power and on the distance d, in FIG. 1A, between the target 12 and the substrate 16. For deposition of optical oxide materials, for example scanning speeds between about 2 sec/one-way scan across the target to 20-30 sec/scan provide a beneficial layer thickness. Limiting the amount of material deposited in each pass promotes chemical and physical uniformity of the deposited layer.

Substrate bias has been used previously to planarize RF sputtered deposited quartz films. A theoretical model of the mechanism by which substrate bias operates, has been put forward by Ting et al. (J. Vac. Sci. Technol. 15, 1105 (1978)). When power is applied to the substrate, a so-called plasma sheath is formed about the substrate and ions are coupled from the plasma. The sheath serves to accelerate ions from the plasma so that they bombard the film as it is deposited, sputtering the film, and forward scattering surface atoms, densifying the film and eliminating columnar structure. The effects of adding substrate bias are akin to, but

more dramatic than, the effects of adding the low frequency RF component to the sputter source.

Biasing substrate 16 results in the deposited film being simultaneously deposited and etched. The net accumulation of film at any point on a surface depends on the relative rates of deposition and etching, which depend respectively, on the power applied to the target and to the substrate, and to the angle that the surface makes with the horizontal. The rate of etching is greatest for intermediate angles, on the order of 45 degrees, that is between about 30 and 60 degrees.

Powers to target 12 and substrate 16 can be adjusted such that the rates of deposition and etching are approximately the same for a range of intermediate angles. In this case, films deposited with bias sputtering have the following characteristics. At a step where a horizontal surface meets a vertical surface, the deposited film makes an intermediate angle with the horizontal. On a surface at an intermediate angle, there will be no net deposition since the deposition rate and etch rate are approximately equal. There is net deposition on a vertical surface.

Target 12 can have an active size of about 675.70×582.48 by 4 mm, for example, in a AKT-1600 based system in order to deposit films on a substrate 16 that is about 400×500 mm. The temperature of substrate 16 can be held at between −50C and 500C. The distance between target 12 and substrate 16 can be between 3 and 9 cm. Process gas can be inserted into the chamber of apparatus 10 at a rate of between about 30 to about 100 sccm while the pressure in the chamber of apparatus 10 can be held at below about 2 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.

Therefore, any given process utilizing apparatus 10 can be characterized by providing the power supplied to target 12, the power supplied to substrate 16, the temperature of substrate 16, the characteristics and constituents of the reactive gasses, the speed of the magnet, and the spacing between substrate 16 and target 12.

Sputtered oxide films according to some embodiments of the present invention can be deposited onto a Si wafer or thermal oxide wafers at pressure of between about 3 and about 6 mTorr. The ratio of O_2/Ar gas flow can be set at a value to ensure that target 12 is operating within a poison mode. The poison mode is defined as the ratio where the oxide is etched from the surface of target 12 as fast as the oxide layer is formed. Operating in the poison mode results in the stoichiometric film. Sub-stoichiometric oxides may not be optically transparent. The pulsing frequency range for power supply 14 can be from about up to about 250 KHz. The frequency 40 KHz is approximately the lowest frequency at which no arcing will occur during deposition in, for example, the AKT 1600 based system. The reverse pulsing time is determined by the amount of arcing generated during the process. Longer reverse time means longer discharge time and thus less arcs. However, if the reverse time is too long, the deposition rate will decrease. Power supply 18 is a 2 MHz RF power supply operated at powers up to several hundred Watts.

FIG. 4 shows an embodiment of a process procedure 400 performed on apparatus 10. In step 401, the target is prepared for the deposition. In some embodiments, target 12 can be cleaned by pure Ar sputtering. In other words, apparatus 10 is operated with pure Ar gas only (referred to as the metal mode) in order to sputter away a surface layer of target 12.

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FIG. **7** shows the typical drift in the index of refraction with deposition of oxide layers for several different targets over different runs for each target. In FIG. **7**, the compositions of the target materials utilized in target **12** for the depositions shown are as follows: Si: 60 cat. % and Al: 40 cat. %; Si: 50 cat. % and Al: 50 cat. %; Si: 85 cat. % and Al: 15 cat. %; Si: 35 cat. % and Al: 65 cat. %; and Si: 92 cat. % and 8 cat. %. Each deposition was operated under the same process parameters: 4.5 kW of pulsed DC power at 200 kHz with a reverse time of 2.3 μs applied to target **12**, O_2 flow at 44 sccm, Ar flow at 30 sccm introduced to apparatus **10**, **100** W of bias power at 2 MHz applied to substrate **16**, the temperature of substrate **16** held at 200° C., and the distance between substrate **16** and target **12** being set at 6 cm. For each target measured, the index drifted up during repeated utilization.

FIG. **8** shows the relationship between the index of refraction of a film deposited according to the present invention and the amount of aluminum in the composite target. As can be seen from FIG. **8**, the index of refraction of the deposited film depends strongly on the aluminum content. Therefore, as the aluminum in a metal target is depleted, the index of refraction drifts. In some embodiments, the ratio of Ar and O_2 utilized in the process can be maintained to provide films of uniform index over a large number of depositions on the target.

Reactive sputtering from a metal or metallic alloy target **12** can be characterized by two modes of operation. In the first mode, which is sometimes referred to as the 'metallic mode' the surface of target **12** is substantially metallic. This mode is characterized by a small addition of reactive gas to the inert gas flow of apparatus **10** as well as a higher impedance magnetron discharge. It is also characterized by incomplete oxidation of film deposited on substrate **16** and therefore higher index films. As the proportion of reactive to inert gas is increased, the sputter voltage at target **12** begins to fall at constant power.

FIG. **5** shows the voltage on target **12** of an embodiment of apparatus **10** according to the present invention as a function of process gas constitution. In the example illustrated in FIG. **5**, for example, a metallic target with composition 0.8 cat. % Er, 0.8 cat. % Yb, 57.4 cat. % Si and 41 cat. % Si, which can be formed as described in the '341 application, was sputtered in an embodiment of apparatus **10** based on the AKT-1600 PVD system with 6 kW of pulsed DC power at a frequency of 120 kHz and a reverse time of 2.3 micro seconds. The Argon gas flow was set at 60 sccm and the Oxygen gas flow was varied from zero up to 40 sccm. For more details regarding this deposition, see Example 1 below.

As shown in FIG. **5**, the voltage on target **12** during deposition (the "target voltage") was constant at about 420 Volts for oxygen flow rates up to about 20 sccm. This is clearly the metallic mode of operation for this embodiment of target **12**. Films deposited in this range of oxygen flow are characterized as metallic with an oxygen content that increases with oxygen flow rate during deposition. As the oxygen flow is increased up to about 26 sccm, the voltage on target **12** begins to decrease, indicating that the surface of target **12** is beginning to form an oxide layer. The oxide layer on the surface of target **12** has a higher secondary electron yield under the influence of the Argon ion flux. The additional electron flux to the magnetron electron trap increases the ion production in the plasma, which, in turn, decreases the impedance of the plasma discharge in apparatus **10**.

At slightly higher oxygen flow during deposition, the oxide layer on target **12** forms a continuous layer and the voltage of target **12** during deposition falls rapidly to the range of about 190 to about 270 Volts, indicating complete coverage of the surface of target **12** with an oxide that is at least as thick as the material removed during one scan of the magnetron. Under this condition, the rate of oxide formation on the surface of target **12** equals or exceeds the rate of sputter removal of the surface of target **12** by the moving magnetron **20**. This condition is sometimes referred to as the 'poisoned mode'.

Under steady state DC voltage conditions, the oxide layer on target **12** soon charges up, leading to reduced rate of sputtering and increased micro-arc discharging in apparatus **10**. This discharging leads to particulation of the oxide layer on target **12**, which degrades the quality of a film deposited on substrate **16**. In the example shown with FIG. **5**, the negative going DC Voltage is reduced at a frequency of 120 kHz to a positive value for a period of about 2.3 micro seconds per cycle, allowing charge neutralization of the surface of target **12**, increasing the steady state sputter and deposition rates as well as decreasing the rate of micro-arcing.

In the case of a magnetron configuration of magnet **20** having a significant deep local target erosion (rather than a configuration of magnet **20** described above which yields uniform target erosion), the change in the target voltage of target **12** is more gradual with increasing oxygen flow since it is more difficult to establish an oxide condition at the center of an intense region of local erosion. The resulting deposited film, however, will be rich in metallic sputtered flux to the substrate in the region of higher sputter erosion, leading to non uniform stoicheometry and non-uniform indices of refraction in a film deposited on substrate **16**. In the case of a scanning magnetron **20** with uniform target erosion, the change in the surface condition from metallic to poisoned is more abrupt, as the formation rate of the oxide increases to equal the sputter removal of the oxide over a wide area of the target. In this case, there is uniform distribution of sputtered oxide from the target. Uniform stoicheometry and uniform indices of refraction result for the film deposited on substrate **16**.

FIG. **8** shows the range of indices of refraction of films deposited for targets of differing silica and alumina compositions, as deposited and after a subsequent anneal step. In the case of a pure silicon target, the as-deposited index of refraction can be as high as 3.4 for pure amorphous silicon. In FIG. **8**, pure silica films (zero Al%) can be deposited with a reactive pulsed DC and substrate bias deposition according to the present invention with substantially complete oxygen stoicheometry, so as to approximate monolithic amorphous silica. The index of refraction of such films decreases with a subsequent heat treatment of between about 700-900° C., indicating somewhat more complete oxidation reaction of the material of the film together with some degree of stress relaxation of the film deposited on substrate **16**.

At the opposite extreme, a pure aluminum embodiment of target **12** (100% Al) can be utilized to deposit films on substrate **16** under similar process conditions as is utilized to deposit pure silica films on substrate **16**. In the case of the pure aluminum reactive deposition, the dependence of the index of refraction of the film deposited on substrate **16** on oxygen flow as well as on the frequency of the pulsed DC process can be examined. As a result, a larger range of effective index of refraction is achieved together with a reduced or zero dependence of the index on the subsequent anneal process. Six targets having differing aluminum composition were utilized to evaluate the index of refraction of sputtered films on substrate **16** of related composition. The

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largest change of index with the sputtering conditions is achieved for composition near the middle of the Al/Si composition range (about 50% Al and 50% Si).

FIG. **7** shows the change in film index for oxide films for several embodiments of target **12** and processes with an initial 30 minutes of Argon only sputtering, followed by continuous deposition with an oxygen flow rate sufficient for operation in the poisonous mode. Note that the rate of increase in the index of refraction of a resulting film deposited on substrate **16** with continuous poisoned mode deposition is proportional to the concentration of aluminum in the composition of target **12**. This result is due to the depletion of the aluminum from the target surface during the metallic sputtering or pre-condition process. The aluminum in target **12** is preferentially sputtered over the silicon in target **12**, leaving the surface of target **12** rich in silicon. At the onset of poisoned mode sputtering, the film deposited on substrate **16** is rich in silica and demonstrates a systematic and reproducible decrease in index of refraction. During continuous poisoned mode deposition, the silicon rich surface of target **12** can be sputtered away and the aluminum portion substantially returned to the bulk composition of target **12**. Consequently, a metallic pre-condition step can be utilized to achieve a subsequent process for the deposition of a film having an increasing index of refraction under conditions of oxide/metal stoicheometry.

In step **402** of FIG. **4**, substrate **16** is prepared. Substrate **16** can be mounted on carrier sheet **17** and placed in apparatus **10**. In step **403**, gas flow parameters are adjusted for the particular deposition to be performed. The constituency and flow rates of the process gas are fixed. In some embodiments, the ratio of Ar and O_2, for example, can be set and the flow rate of each gas set. Further, the combination of flow rate and vacuum system of apparatus **10** determines the pressure during deposition in apparatus **10**.

In step **404**, the substrate temperature is set. Substrate **16** may be brought to temperature over a period of time. In step **405**, the scan characteristics of magnet **20** are fixed. In step **406**, the power setting for power supply **18** is set. Finally, in step **407**, the parameters of pulsed DC power supply **14** is set, including the power, frequency, and reverse pulsing time. In step **408**, then, a film that depends on the parameters of reactor apparatus **10** is deposited on substrate **16**. In some embodiments, films deposited by procedure **400** are thermally annealed after deposition.

FIG. **4** illustrates an example deposition process only. Embodiments of deposition processes according to the present invention can be performed in various different orders.

FIG. **9** shows a chart of various deposition parameters according to the present invention for various embodiments of target **12** and the indices of refraction, both before and after an anneal step, for the resulting deposited film on substrate **16**. Each deposition was accomplished with an embodiment of apparatus **10** based on the AKT 1600 PVD reactor. Anneals were accomplished at 725° C. for 30 min. Specific examples of particular depositions and characteristics of the resulting films deposited on substrate **16** are further discussed below.

FIG. **10** shows the dependence of the index of refraction of silica layers deposited according to the present invention with process conditions. FIG. **11** shows the dependence of index of refraction on the O2/Ar flow ratio for the deposition of pure alumina according to the present invention. FIG. **12** shows the dependence of index for pure alumina films on the frequency of the pulsed DC power applied to target **12**. Both parameters can be utilized to reliably control the index of

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refraction of films deposited on substrate **16** over a range of index values without the use of an additional cationic species, a so called 'dopant'. A third process parameter that can be utilized to adjust the index of refraction of a film deposited on substrate **16** is the bias power applied to substrate **16**. Increasing the oxygen flow ratio, the frequency of the pulsed DC power applied to target **12** or the bias power applied to substrate **16** will systematically increase the index of refraction of the alumina film deposited on substrate **16**. In the case of pure alumina films, minor to no change in the index occurs due to a subsequent anneal process.

FIG. **13** shows the index of refraction of a film deposited on substrate **16** from an embodiment of target **12** with about 92 cat. % of Si and about 8 cat. % of Al for a series of sequential depositions in an embodiment of apparatus **10** based on the AKT 4300 PVD reactor, each following a metallic process condition. For constant high oxygen flow conditions, a small upward trend in the index of refraction is observed. As is generally true, the index of films deposited with higher substrate bias power is systematically lower than films deposited without substrate bias.

FIG. **14** shows the upward trend of the index of refraction after metallic mode precondition of an embodiment of target **12** having composition of about 83 cat. % Si and about 17 cat. % Al for a series of depositions in an embodiment of apparatus **10** based on the AKT 1600 PVD reaction. As is shown in FIG. **14**, longer metallic preconditioning of target **12** results in the index of refraction of the films deposited on substrate **16** having a higher rate of increase than for cases with less prolonged metallic preconditioning of target **12**. The vertical lines on FIG. **14** indicate places where target **12** was preconditioned with only Ar for the indicated periods of time. FIG. **15** shows a decrease in the change in index for sequential films with this embodiment of target **12** deposited with reduced oxygen flow rates at a constant total pressure. A flow rate for oxygen was determined so that the run to run variation for the index of refraction of the film deposited on substrate **16** from this target was about 0.0001 (see the circled data points on the graph of FIG. **15**) which is similar to the variance of the index over the entire wafer of substrate **16**, which is about 70 parts per million.

In some embodiments, films deposited by a pulsed DC biased method according to the present invention are uniformly amorphous throughout their thickness. As has been discussed above, biasing of substrate **16** leads to densification and uniformity in the deposited film. FIGS. **16**A through **16**D show a TEM photograph of a film **1601** deposited according to the present invention. Further, diffraction patterns shown in FIGS. **16**B, **16**C and **16**D at points a, b and c, respectively, in deposited film **1601** show that the film is ammorphous through the thickness of the film. The diffraction patterns of FIGS. **16**B, **16**C and **16**D show no effects of crystallization. Further, the smoothness of the surface of film **1601** indicates a defect free film. The film deposited in FIG. **16**A is deposited with an 0.8/0.8 target (i.e., a target having the composition 52.0 cat. % of Si, 41.0 cat. % of Al, 0.8 cat. % of Er and 0.8 cat. % of Yb). The film is deposited at 6 kW of 120 kHz pulsed DC power with a reverse time of 2.3 μs. The Argon and Oxygen flow rates are 60 sccm and 28 sccm, respectively. Substrate **16** is biased with 100 W of power.

FIG. **17** shows the optical loss per centimeter, measured at 1310 nm, using a three prism coupling to the so called slab mode of the film on a 10 micron oxide, silicon wafer. As deposited the biased, pulsed DC film from a 60 cat. % Si and 40 cat. % Al film demonstrated about 0.1 dB/cm loss. After an 800° C. anneal in air, the loss was less than the mea-

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surement sensitivity of the prism coupling method. This data clearly demonstrates that films deposited according to embodiments of the present invention can be used for the purpose of constructing low loss planar light wave circuits.

Deposition of films according to the present invention can be utilized to deposit cladding layers, active core layers, and passive core layers of an optical amplifier structure or optical waveguide structure. In some applications, for example multiplexer structures, the separation between adjacent waveguides can be small, for example about 8 μm. In some embodiments, the deposition parameters of the upper cladding layer can be adjusted to not only adjust the index of refraction of the layer, but also to insure that the spacing between adjacent waveguides is small.

FIG. **18** shows an example planarization deposition over a multiplexer structure. In the particular example of upper cladding layer **1803** shown in FIG. **18**, the deposition parameters from a 92 cat. % Si and 8 cat. % Al is: 5.5 Kw of Pulsed DC power applied at 200 KHz with 2.2 μs of reverse time, gas flow of 75 sccm Ar and 100 sccm O_2, a substrate bias of 650 W (at 2 MHz), and a substrate temperature of 200° C. Layer **1803** was deposited with an AKT 4300 based embodiment of apparatus **10**. As shown in FIG. **18**, the layer thickness in areas other than over waveguide structures **1801** and **1802** is 11.4 μm. Waveguide structures **1801** and **1802** are 8.20 μm high waveguides and separated by 6.09 μm at the base and by 8.40 μm at their top. In FIG. **18**, the undercladding layer **1804** is about 1.98 μm thick.

FIG. **19** illustrates deposition of material over a structure. Upper cladding layer **1803**, in region **1901**, will be angled from the horizontal by an angle θ. The deposition and etching rates of a deposited layer depends on the angle θ. FIGS. **20** and **21** illustrate different cases of deposition and etch rates as a function of the angle θ. The relationship between the rate of deposition and the etch rates can be adjusted by adjusting the deposition parameters. For example, the bias power to substrate **16** can be adjusted to control the relationship between the etch rates and deposition rates of material.

FIG. **22** illustrates deposition rates over a structure **2201** as a function of time. In FIG. **2201**, h is the thickness deposited over structure **2201**. The planarization when layer **1803** becomes flat.

The time for planarization can be estimated as

$$t_p = \frac{\frac{W}{2}\tan\alpha + H}{a_{flat} - \frac{a_{min}}{\cos\alpha}},$$

where W is the width of structure **2201**, H is the height of structure **2201**, a_{flat} refers to the accumulation rate on the flat surface, a_{min} refers to the accumulation rate on the minimum accumulation slope, and a is the surface angle from the horizontal plane of the minimum accumulation slope.

FIG. **23** shows a deposited film **1803** as shown in FIG. **18**, except that the bias power to substrate **16** is set to 400 W instead of 650 W. As can be seen in FIG. **23**, a keyhole **2301** is formed with an incomplete filling of uppercladding layer **1803** between structures **1801** and **1802**. Deposition of uppercladding layer **1803** substantially follows the trends illustrated in FIGS. **19** through **22**.

FIG. **24** shows deposition as shown in FIG. **18**, except that the bias power to substrate **16** is set to 600 W instead of 650

W. As can be seen in FIG. **24**, keyhole **2301** has closed leaving a small line defect **2401** in the fill.

FIG. **28** shows deposition as shown in FIG. **18**, except that the bias power to substrate **16** is set to 900 W instead of 650 W. As can be seen in FIG. **28**, the etch rate has been increased to such an extent that the corners of structures **1801** and **1802** have been etched to form slopes **2501** and **2502**, respectively.

Therefore, as illustrated in FIGS. **18** through **25**, an uppercladding layer can be deposited in accordance with the present invention such that it fills the space between adjacently placed waveguides. In general, the parameters can be optimized for index control and the bias power to substrate **16** can be adjusted for fill. In some embodiments, other parameters (e.g., the constituency of process gas, frequency and power of pulsed DC power source **14**, and other parameters) in order to adjust the deposition and etch rates and thereby effectively planarize the structure as described.

Therefore, depositions of various films in embodiments of apparatus **10** according to the present invention with several embodiments of target **12** and the effects on index of refraction, uniformity of films, and fill characteristics of varying several of the process parameters has been discussed above. In some embodiments, stress effects due to wafer bowing of substrate **16** can also be reduced. Wafer bowing of substrate **16** can be reduced, reducing the stress in a film deposited on substrate **16**, by, for example, depositing a film on the backside of substrate **16** before deposition of a film on substrate **16**. In some embodiments, a film having a similar thickness of a similar layer of material can be deposited on backside of substrate **16** prior to deposition of the film on substrate **16** according to the present invention. The wafer bowing resulting from differing thermal expansions of the film and substrate **16** is therefore countered by a similar stress from another film deposited on the backside of substrate **16**.

Several specific examples film depositions utilizing apparatus **10** are discussed below. Further, examples of optical amplifiers produced utilizing the ceramic tiles according to the present invention are presented. These examples are provided for illustrative purposes only and are not intended to be limiting. Unless otherwise specified, apparatus **10** utilized in the following examples was based on the AKT 1600 reactor. Further, unless otherwise specified, the temperature of substrate **16** was held at about 200° C. and the distance between substrate **16** and target **12** was 4 s/scan. The separation between substrate **16** and target **12** is about 6 cm.

EXAMPLE 1

An AKT 1600 based reactor can be utilized to deposit a film. In this example, a wide area metallic target of dimension 550×650 mm with composition (Si/Al/Er/Yb) being about 57.0 cat. % Si, 41.4 cat. % Al, 0.8 cat. % Er, and 0.8 cat. % Yb (a "0.8/0.8" target) was fabricated as described in the '341 patent.

In step **402**, a 150 mm P-type silicon wafer substrate was placed in the center of a 400×500 mm glass carrier sheet **17**. Power supply **14** was set to supply 6000 watts of pulse DC power at a frequency of 120 KHz with a reverse pulsing time of about 2.3 us. Magnet **20**, which is a race-track shaped magnet of approximate dimension 150 mm×600 mm, was swept over the backside of the target at a rate of about 4 seconds per one-way scan. The temperature of substrate **16** was held at 200C and 100 W of 2 MHz RF power was applied to substrate **16**. The target **12** to substrate **16** distance

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was about 6.5 cm. The sputtering gas was a mixture of Argon and Oxygen. Substrate **16** and carrier **17** was preheated to 350° C. for at least 30 min prior to deposition. The active film was deposited in the poison mode. Deposition efficiency was approximately 1 um/hr.

FIG. **5** shows the hysteresis curve of this particular embodiment of target **12**. When target **12** under goes the transition from metallic to poison mode, the target voltage drops from an average of about 420V to an average of about 260V. Before each film deposition, in step **401**, target **12** is cleaned by pure Argon sputtering in the metallic mode. Then target is then conditioned in poison mode with the oxygen flow much higher than the flow required at the transition region.

Tables 1A through 1C shows some effects on the deposited films of depositions with the 0.8/0.8 target under different operating conditions. Table 1A includes photoluminescence (pumped at 532 nm) and index of refraction for films deposited on substrate **16** with different Ar/O$_2$ gas flow ratios with no bias power applied to substrate **16**.

TABLE 1A

Target Power (KW)	Ar/ O2	Frequency (KHz)	Reverse Pulsing Time (us)	Bias (W)	PL/um (532 nm)	Index
6	30/42	200	2.3	0	1973	1.5142
6	30/36	200	2.3	0	2358	1.5215
6	60/30	200	2.3	0	3157	1.5229
6	60/28	200	2.3	0	3421	1.5229

Table 1B shows the variation in photoluminescence (pumped at 532 nm) and index of refraction of the film deposited on substrate **16** with deposition processes having with the same Ar/O$_2$ ratios but different pulsed DC power frequencies from power supply **14**.

TABLE 1B

Target Power (KW)	Ar/ O2	Frequency (KHz)	Reverse Pulsing Time (us)	Bias (W)	PL/um (532 nm)	Index
3	60/28	100	2.3	100	1472	1.5146
4	60/28	75	3.5	100	2340	1.5189
6	60/28	120	2.3	100	5178	1.5220

Table 1C shows the photoluinescence and index as deposited where the bias power to substrate **16** is varied.

TABLE 1C

Target Power (KW)	Ar/ O2	Frequency (KHz)	Reverse Pulsing Time (us)	Bias (W)	PL/um (532 nm)	Index
6	60/28	200	2.3	0	3657	1.5230
6	60/28	200	2.3	100	2187	1.5244
6	60/28	200	2.3	200	3952	1.5229
6	60/28	200	2.3	300	5000	1.5280

The photoluminescence values can be measured with a Phillips PL-100. The deposited film can be pumped with a 532 nm laser and the luminescence at 980 is measured. The index is the index of refraction. Typically, films deposited are annealed in order to activate the erbium. FIG. **6** shows the photoluminescence and lifetime versus anneal temperature for a typical film deposited as described in this example.

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EXAMPLE 2

A waveguide amplifier can be deposited according to the present invention. An embodiment of target **12** having composition 57.4 cat. % Si, 41.0 cat. % Al, 0.8 cat. % Er 0.8 cat. % Yb (the "0.8/0.8 target") can be formed as disclosed in the '341 application. The Er—Yb (0.8/0.8) co-doped Alumino-Silicate film was deposited onto a 6 inch wafer of substrate **16** which includes a 10 μm thick thermal oxide substrate, which can be purchased from companies such as Silicon Quest International, Santa Clara, CA. Target **12** was first cleaned by sputtering with Ar (80 sccm) only in the metallic mode. Target **12** was then conditioned in poison mode by flowing 60 sccm of Argon and 40 sccm of oxygen respectively. The power supplied to target **12** during conditioning was kept at about 6 kW.

An active core film was then deposited on substrate **16**. The thickness of the deposited film is approximately 1.2 μm. The deposition parameters are shown in Table 2.

TABLE 2

Target Power (KW)	Ar/O2 (sccm)	Pulsing Frequency (KHz)	Bias (W)	Reverse pulsing time (us)
6	60/28	120	100	2.3

A straight waveguide pattern can then formed by standard photolithography techniques. The active core was etched using reactive ion etch followed by striping and cleaning. Next, a 10 μm top cladding layer is deposited using a similar deposition process according to the present invention. An embodiment of target **12** with composition 92 cat. % Si and 8 cat. % Al as shown in FIG. **9** to form the top cladding layer. The index difference between the top cladding layer and the active layer is about 3.7%. The amplifier is then annealed at 725° C. for about 30 min (see FIG. **6**, for example).

The erbium excited-state lifetime and the up-conversion coefficient were measured to be 3 ms and 4.5×10^{-18} cm^3/s, respectively. A net gain of about 4 dB for small signal (about −20 dBm) with fiber to waveguide and to fiber coupling was obtained. Waveguide length was 10 cm and the width was about 1.5 to 8 μm. The coupling loss between the fiber and the waveguide is 3-4 dB/facet, and passive excess loss is 0.1-0.2 dB/cm for 3 um waveguide. The waveguide was both co- and counter pumped with 150 mW 980 nm laser per facet.

EXAMPLE 3

This example describes production of a dual core Erbium/Yttrbium co-doped amplifier according to the present invention. In one example, substrate **16** is a silicon substrate with an undercladding layer of thermally oxidized SiO$_2$ of about 15 μm thick. Substrate **16** with the thermal oxide layer can be purchased from companies such as Silicon Quest International, Santa Clara, Calif. A layer of active core material is then deposited on substrate **16** with a Shadow Mask as described in the '492 application. Use of a shadow mask results in a vertical taper on each side of a finished waveguide which greatly enhances the coupling of light into and out of the waveguide.

Active core layer is deposited from a 0.8/0.8 target as described in the '247 application having composition 57.4 cat. % Si, 41.0 cat. % Al, 0.8 cat. % Er, and 0.8 cat. % Yb.

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The deposition parameters are identical to that of Example 2 described above. The active layer is deposited to a thickness of about 1.2 μm.

A passive layer of aluminasilicate is then deposited over the active layer. A passive layer of about 4.25 μm thickness can be deposited with an embodiment of target **12** having composition of Si/Al of about 87 cat. % Si and about 13 cat. % Al. The passive layer and active layer are then patterned by standard lithography techniques to form a core that has a width of about 5.0 μm for the active core and tapering to about 3.5 μm at the top of the passive core with an effective length of about 9.3 cm.

Upper cladding layer is then deposited from a Si/Al target of 92 cat. % Si and 8 cat. % Al. Deposition of the upper cladding layer is shown in FIG. **9**. In some embodiments, the upper cladding layer can be deposited with a non-biased process. The thickness of the upper cladding layer can be about 10 μm. The amplifier formed by this process is then annealed at 725° C. for about 30 min.

The as-deposited Erbium and Ytterbium concentrations in the active layer of core **303** is 2.3×10^{20} cm^{-3} Erbium concentration and 2.3×10^{20} cm^{-1} Ytterbium concentration. The index of the core is 1.508 and the index of cladding layers are 1.4458 for undercladding layer **302** and 1.452 for uppercladding layer **304**. The parameter Δn/n is therefore about 5.0%.

A reverse taper mode size converter, see the '492 application, is utilized for coupling light into waveguide amplifier **300**. The insertion loss at 1310 nm is about 2 dB. FIG. **26** shows the amplifier performance of this example. In FIG. **26**, amplifier **300** is pumped with 150 mW from one side pumping with 984 nm light. Gain flattening is achieved within about 1 dB in the range 1528 nm to 1562 nm for small input signals (−20 dBm). For large input signals (0 dBm), gain flattening is also achieved within about 1 dB.

EXAMPLE 4

Another example of production of a waveguide amplifier is described here. Again, substrate **16** can be a Si wafer with about a 15 μm thick thermal oxide as can be purchased from Silicon Quest International, Santa Clara, Calif. The embodiment of target **12** for the deposition of the active core can have a composition of about 50 cat. % Si, 48.5 cat. % Al, 1.5 cat. % Er (the "1.5/0" target), which can be fabricated as discussed in the '138 application. Target **12** was first cleaned by sputtering with Ar (80 sccm) only in the metallic mode. Target **12** was then conditioned in poison mode by flowing 60 sccm of Argon and 40 sccm of oxygen respectively.

The pulsed DC power supplied to target **12** was about 6 kW. Whenever a brand new target was used or when the target has been expose to atmosphere, a long time of condition (for example more than 30 hrs of conditioning) may be necessary to ensure films with the best active core property (longest life time and highest photoluminescence) are deposited. Substrate **16** is then preheat at about 350° C. for about 30 min before deposition.

The active core film was deposited onto a 6 inch thermal oxide wafer, which has been previously discussed, from the 1.5/0 target. The thermal oxide thickness was about 10 μm as described in previous examples. The active core is deposited to a thickness of about 1.2 μm with a deposition time of approximately 1 hr. The process condition are as listed in Table 4 below.

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TABLE 3

Target Power (KW)	Ar/O2 (sccm)	Pulsing Frequency (KHz)	Bias (W)	Reverse pulsing time (us)
6	60/28	120	100	2.3

A straight waveguide pattern can then be formed by a standard photolithography procedure. The active core was etched using reactive ion etch followed by striping and cleaning. Finally, a 10 μm top cladding layer is deposited using a similar process. A target having composition 92 cat. % Si and 8 cat. % Al with deposition parameters as described in FIG. **9** was used to deposit the top cladding. The difference between the index of refraction between the core and the cladding is then about 3.7%.

In this example, annealing of the amplifier structure was performed at various anneal temperatures. The results of the various anneals are shown graphically in FIGS. **27** and **28**. FIG. **27** shows both internal gain in the C-band and insertion loss at 1310 nm of a 2.5 μm wide, 10.1 cm long waveguide as deposited in this example as a function of annealing temperature. The life time in ms and up-conversion constants in cm^{-3}/s measurements for the deposited active core film at different annealing temperature are shown in FIG. **28**.

EXAMPLE 5

One of the problems encountered during the reactive sputtering from an alloy metallic target is that the film composition drifts from run to run due to the difference in sputtering yields from the elements that forms the target alloy. For example, with Ar as a sputtering gas, the sputtering yield of Aluminum is about 3-4 times that of Silicon, while sputtering yield of Alumina is only about 50% that of Silica. Therefore, during the metal burn in, more Aluminum is sputtered from the target, resulting in a Si rich target surface. When sputtering in the poison mode, more Silica will be removed from target. Thus, as deposition goes on, the composition of the film deposited on substrate **16** will drift from lower Alumina concentration to higher Alumina concentration. This results in the index of refraction of a film drifting up with subsequent depositions from a target **12**, as is shown for the deposition described in Example 4 in FIG. **29**. FIG. **30** shows the drift in photoluminescence pumped at 532 nm with subsequent depositions. FIG. **31** shows drift in the excited state lifetime with subsequent depositions from a target. The embodiment of target **12** utilized in FIGS. **29** through **31** is the 1.5/0 target and the deposition parameters are as described above in Example 4.

The drift can be stabilized by recondition target **12** prior to deposition. The recondition process (or burn in) consists of both sputtering in metallic mode and then sputtering in poison mode to condition target **12**. The burn in time in metallic mode needs to be as short as possible and at the same time insure no arcing during the poison mode deposition. FIG. **32** shows the much improved drift in the index of refraction and the photoluminescence when target **12** is reconditioned between subsequent depositions.

EXAMPLE 6

This example describes the fabrication of another Er-Yb codoped waveguide amplifier according to the present invention. The active core is deposited with an embodiment of target **12** with composition about 49 cat. % Si, 48 cat. %

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Al, 1.6 cat. % Er and 0.5 cat. % Yb, which can be fabricated as described in the '341 application. Target **12** was first cleaned by sputtering with Ar (80 sccm) only in the metallic mode. Target **12** was then conditioned in poison mode by flowing 60 sccm of Argon and 40 sccm of oxygen respectively. The pulsed DC power supplied to target **12** was kept at 5 kW. Table 4 shows photoluminescence and index of refraction of as-deposited films from this example at some typical process conditions. The units for photoluminescence are the number of counts per micron. Lifetime and photoluminescence measured after annealing at various different temperatures are shown in Table 5.

				Target 4		
Target Power (KW)	Ar/O2 (sccm)	Pulsing Frequency (KHz)	Bias (W)	Reverse Pulsing Time (us)	532 nm PL/um	Index
5	60/34	120	100	2.3	3367	1.5333
5	60/30	120	100	2.3	3719	1.5334

TABLE 5

Anneal Temperature ° C.	Life Time (ms)	PL (532 nm)/um
725	3	7000
775	3	7000
800	4	7500
825	4.7	8560
850	5.8	10000
900	6.9	17000

A waveguide amplifier was fabricated using this material in the similar fashion as described in examples 2-4. The active core was first deposited on substrate **16**, which includes a 10 um thermal oxide layer, using the following deposition parameters: target power 5 KW, pulsing frequency 120 KHz, bias 100 W, reverse time 2.3 us, Argon and Oxygen flow are 60 sccm and 30 sccm respectively. The active core thickness is deposited to a thickness about 1.2 μm, which takes approximately 1 hr. All wafers are preheated at about 350° C. for 30 min before deposition. A straight waveguide pattern is then formed by standard photolithography procedure. The active core was etched using reactive ion etch following by striping and cleaning. Next, a 10 μm top cladding layer is deposited using similar process. The "92/8" (92 cat. % Si and 8 cat. % Al) metallic target was used to deposit top clad according to deposition parameters shown in FIG. **9**, resulting in a 4% index difference between active core and cladding. The wave guide was then annealed at 800° C. for about 30 min.

This waveguide was tested for gain using the method described in previous examples. However no net gain was observed from this waveguide since the passive loss was too high.

EXAMPLE 7

In addition to active material layers (i.e., layers having rare-earth ion concentrations), passive layers can also be deposited. FIG. **9** shows deposition parameters for several target compositions, including some targets for deposition of passive (i.e., alloys of Al and Si with no rare earth ion

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concentration) layers. In this example, an embodiment of target **12** with a material composition of pure silicon is utilized.

Apparatus **10** can be based on an AKT 1600 reactor and deposited with about 1 to 3 kW of pulsed DC target power supplied to target **12**. Particular depositions have been accomplished at 2.5 kW and 1.5 kW. The frequency of the pulsed DC power is between about 100 and 200 Khz. Some depositions were performed at 200 kHz while others were performed at 100 kHz. The reverse time was varied between about 2 μs and about 4 μs with particular depositions performed at 2.3 μs and 3.5 μs. The bias power to substrate **16** was set to zero.

Index variation of SiO2 films with bias to substrate **16** and deposition rates as a function of bias power to substrate **16** is shown in FIG. **10**.

The process gas included a mixture of Ar, N_2 and O_2. The Ar flow rates was set at 20 sccm while the O_2 flow rate was varied between about 5 and about 20 sccm and the N_2 flow rate was varied from about 2 to about 35 sccm. FIG. **33** shows the variation in the index of refraction of a film deposition on substrate **16** as the O_2/N_2 ratio is varied.

EXAMPLE 8

Alternatively, films can be deposited on substrate **16** from a pure alumina target. In an example deposition with an embodiment of target **12** of alumina in an embodiment of apparatus **10** based on the AKT 1600 reactor, the pulsed DC target power was set at 3 kW and the frequency was varied between about 60 kHz and 200 kHz. The reverse time was set at 2.5 μs. Again, no bias power was supplied to substrate **16**. The O_2 flow rate was varied from about 20 to about 35 sccm, with particular depositions performed at 22 and 35 sccm. The Ar flow rate was set at 26 sccm. A post deposition anneal of substrate **16** at 800° C. for 30 min. was performed.

FIG. **12** shows the variation of refractive index of the film deposited on substrate **16** with varying frequency of the pulsed DC power supplied to target **12**. FIG. **11** shows the variation in refractive index of a film deposited on substrate **16** with varying O_2/Ar ratio. As can be seen from FIGS. **33**, **34** and **35**, the index of refraction of films deposited from alumina can be adjusted by adjusting the process gas constituents or by adjusting the frequency of the pulsed DC power supplied to target **12** during deposition.

EXAMPLE 9

Additionally, passive films can be deposited from targets having a composition of Si and Al. For example, layers have been deposited from embodiments of target **12** with composition 83% Si and 17% Al. About 4.5 kW of pulsed DC power at about 200 kHz frequency was supplied to target **12**. The reverse time was about 2.2 μs. A bias power of about 150 W was supplied to substrate **16** during deposition. FIGS. **14** and **15** show variation of the index of refraction for subsequent runs from this target.

The examples and embodiments discussed above are exemplary only and are not intended to be limiting. One skilled in the art can vary the processes specifically described here in various ways. Further, the theories and discussions of mechanisms presented above are for discussion only. The invention disclosed herein is not intended to be bound by any particular theory set forth by the inventors to explain the results obtained. As such, the invention is limited only by the following claims.

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We claim:

1. A method of depositing a film on an insulating substrate, comprising:

providing a process gas between a conductive target and the substrate;

providing pulsed DC power to the target through a narrow band rejection filter such that the target alternates between positive and negative voltages;

providing an RF bias at a frequency that corresponds to the narrow band rejection filter to the substrate;

providing a magnetic field to the target; and

reconditioning the target;

wherein reconditioning the target includes reactive sputtering in the metallic mode and then reactive sputtering in the poison mode.

2. A method of depositing an insulating film on a substrate, comprising:

providing a process gas between a target and a substrate;

providing pulsed DC power to the target through a narrow band rejection filter such that the voltage on the target alternates between positive and negative voltages;

providing an RF bias that corresponds to the narrow band rejection filter to the substrate; and

providing a magnetic field to the target;

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.

3. The method of claim **2** wherein the target is a metallic target and the process gas includes oxygen.

4. The method of claim **2** 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.

5. The method of claim **2** wherein the target is a ceramic target.

6. The method of claim **2** wherein the magnetic field is provided by a moving magnetron.

7. The method of claim **2** further including holding the temperature of the substrate substantially constant.

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8. The method of claim **2** wherein the process gas includes a mixture of Oxygen and Argon.

9. The method of claim **2** wherein the Oxygen flow is adjusted to adjust the index of refraction of the film.

10. The method of claim **2** wherein the process gas further includes nitrogen.

11. The method of claim **2** 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.

12. The method of claim **2**, further including uniformly sweeping the target with a magnetic field.

13. The method of claim **12** 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.

14. The method of claim **2** wherein the target is an alloyed target.

15. The method of claim **14** wherein the alloyed target includes one or more rare-earth ions.

16. The method of claim **14** wherein the alloyed target includes Si and Al.

17. The method of claim **14** 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, Th, Dy Ho, Tm, and Lu.

18. The method of claim **14** wherein the alloyed target is a tiled target.

19. The method of claim **18** wherein each tile of the tiled target is formed by prealloy atomization and hot isostatic pressing of a powder.

20. The method according to claim **2**, wherein the narrow band-rejection filter has a bandwidth of about 100 kHz.

21. The method according to claim **2**, wherein the RF frequency is about 2 MHz.

*   *   *   *   *

UNITED STATES PATENT AND TRADEMARK OFFICE
CERTIFICATE OF CORRECTION

PATENT NO. : 7,381,657 B2   Page 1 of 1
APPLICATION NO. : 10/954182
DATED   : June 3, 2008
INVENTOR(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