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ii / SPIE Vol. 1047 Mirrors and Windows for High Power/High Energy Laser Systems (1989)

MIRRORS AND WINDOWS FOR HIGH POWER/HIGH ENERGY LASER SYSTEMS

Volume 1047

CONTENTS

	Conference Committee
	Symposium Organizers
	Introduction
SESSION 1	MIRRORS
1047-01	Advances in the shaping of lightweight large mirror blanks of Zerodur®
	T. A. Marx, H. Olyschlaeger, Schott Glass Technologies, Inc.; R. W. Mueller, H. W. Hoeness,
	Schott Glaswerke (FRG)
1047-02	Performance of overcoated mirrors designed for glancing incidence operation
	W. D. Kimura, Spectra Technology, Inc 10
1047-03	High index of refraction films for dielectric mirrors prepared by metal-organic CVD
	R. M. Brusasco, Lawrence Livermore National Lab
1047-04	Advanced figure-of-merit evaluation for CO ₂ laser optics using finite-element analysis
	G. L. Herrit, H. E. Reedy, II-VI Inc
1047-05	Parametric study of mesh-enhanced forced convection heat transfer for the cooling of high power density mirrors
	W. W. Yuen, R. V. Fleishman, Univ. of California/Santa Barbara
SESSION 2	WINDOWS
1047-10	Optical distortion coefficients of laser windows: one more time
	C. A. Klein, Raytheon Research Div
1047-11	Calcium fluoride: new properties for an old material
	C. W. King, O. H. Nestor, Harshaw Crystals and Electronics
1047-15	Thermal shock: catastrophic damage to transmissive optical components in high power cw and
1011 15	repetitive pulsed laser environments
	J. R. Palmer, Ctr. for Applied Optics/Univ. of Alabama at Huntsville
1047-17	Spherical vacuum-interface window with axial-flow impingement cooling
	G. S. Holderbaum, R. J. Phillips, Lincoln Lab./MIT
1047-18	Large exit windows for high power beam directors
1000 U	D. E. Weidler, Lincoln Lab./MIT
SESSION 3	LARGE OPTICS, RESONATORS, ET CETERA
1047-19	Large optics testing capability at the Naval Weapons Ctr.
	H. E. Bennett, Naval Weapons Ctr
1047-21	Boundary-layer control for large optics
	D. C. Johnson, Lincoln Lab./MIT
1047-22	Unobscured unstable resonator design for large-bore lasers
	S. W. Townsend, J. P. Reilly, W. J. Schafer Associates, Inc
1047-23	Analysis of the Boeing FEL mirror measurements
	C. E. Knapp, V. K. Viswanathan, O. D. Appert, Los Alamos National Lab
1047-25	CVD replication for optics applications
	J. S. Goela, R. L. Taylor, Morton Thiokol Inc./CVD Inc
OF	
142	(continued)



SPIE Vol. 1047 Mirrors and Windows for High Power/High Energy Laser Systems (1989) / iii

MIRRORS AND WINDOWS FOR HIGH POWER/HIGH ENERGY LASER SYSTEMS

Volume 1047

1047-27	Ion beam mirror coating: status and outlooks
	D. T. Wei, Xerox Corp.; H. R. Kaufman, Front Range Research
1047-38	Acoustic damage to large-aperture optics
	J. R. Smith, J. R. Murray, D. T. Kyrazis, R. B. Wilcox, T. L. Weiland, R. B. Ehrlich, C. E. Thompson, R. B. Engle, A. E. Brown, Lawrence Livermore National Lab. 219
SESSION 4	MEASUREMENTS AND CHARACTERIZATION
1047-32	Analysis of diamond-turned optics using ellipsometry
	S. F. Nee, H. E. Bennett, D. L. Decker, S. D. Greene, A. A. Ogloza, Naval Weapons Ctr
1047-33	Distortion measurements at cryogenic temperatures on unpolished substrates using holographic interferometry
	F. A. Kinder, H. E. Bennett, Naval Weapons Ctr
1047-34	Measurements of optical loss in transparent solids using a novel spectrometer based on optical cavity decay
	F. P. Milanovich, J. T. Hunt, J. N. Roe, Lawrence Livermore National Lab
1047-35	Specification and testing facilities for optical components used in a high average power visible laser system
	J. R. Taylor, Lawrence Livermore National Lab
1047-36	Instrumentation for optical characterization of laser optics
	R. A. House II, R. D. Petty, D. J. Gallant, Rockwell International Power Systems
1047-37	Effects of x rays on optical materials and coatings
	J. A. Rathkopf, R. H. White, Lawrence Livermore National Lab 276
1047-39	Laser damage mechanism of dielectric material surface by absorbing inclusion initiated thermal explosion
	M. F. Koldunov, A. A. Manenkov, I. L. Pokotilo, USSR Academy of Sciences (USSR)
	Addendum
	Author Index

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High Index of Refraction Films for Dielectric Mirrors Prepared by Metal-Organic Chemical Vapor Deposition*

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ABSTRACT

A wide variety of metal oxides with high index of refraction can be prepared by Metal-Organic Chemical Vapor Deposition. We present some recent optical and laser damage results on oxide films prepared by MOCVD which could be used in a multilayer structure for highly reflecting (HR) dielectric mirror applications. The method of preparation affects both optical properties and laser damage threshold.

1. INTRODUCTION

Thin films play an important role in high peak power lasers as anti-reflection (AR) and high reflection (HR) coatings. The performance demands on these coatings, especially in laser power handling, grow rapidly as the power levels increase in response to the needs of fusion target physics. For example, the laser damage threshold goal for highly reflecting dielectric mirrors in next generation lasers (at 1064 nm and 10 nsec pulse duration) approaches 40 J/cm² while current HR thin film structures are performing at approximately half that value.¹ In addition, high peak power laser HR's require high reflectivity (>99%) and low absorptive and scatter losses. Meeting these new milestones in optical performance may require new technologies for optical film growth or a deeper understanding of laser damage mechanisms or both.

Chemical Vapor Deposition (CVD) is a thin film growth technique used primarily in the microelectronics industry but which is also capable of preparing oxide coatings of interest in the optics community, such as Al_2O_3 , HfO_2 , ZrO_2 , TiO_2 , Ta_2O_5 , etc.²⁻⁵ Most literature citations on a CVD oxide coating give only a very brief discussion of the optical performance of the film, limited usually to a statement about the index of refraction. It is important to investigate the broadband reflectivity to understand how the film may be used in a multilayer macrostructure such as an HR dielectric stack.

The goal of this study was to examine alumina, hafnia and zirconia thin films prepared by several CVD methods and determine how process variations affected the optical performance, index of refraction, rate of deposition and laser damage threshold at 1064 nm.

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SPIE Vol. 1047 Mirrors and Windows for High Power/High Energy Laser Systems (1989) / 23

2. EXPERIMENTAL

Chemical Vapor Deposition reactions were carried out in a fused silica reaction tube 140 mm diameter at the substrate position and approximately four feet in length. Figure 1 shows the reactor geometry in schematic form. The substrates were Corning fused silica (7940) which were internally heated by a gold lined retainer. The reactor wall exterior was exposed to ambient air, creating a cold wall reactor. The substrates were cleaned according to the method described in Table I, loaded into the reactor chamber in a clean bench environment and pumped to approximately 1 Torr under flowing Argon. Gas flows were established and the substrate brought up to reaction temperature before admitting alkoxide vapors and beginning the reaction.



Figure 1. Schematic of Chemical Vapor Deposition Reactor.

Provision was made for handling either liquid or solid alkoxide species. The tertiary-butoxides formed a convenient set of starting materials since all are volatile and are very reactive. Al(OBu^t)₃ was prepared in this laboratory starting with aluminum metal and iso-propanol to make Al(OPr¹)₃, followed by alcohol interchange with t-butanol in benzene. The reaction of (zirconium, hafnium) dialkylamide with t-butanol to make (Zr, Hf)(OBu^t)₄ was performed as described by Takahashi et al.⁶ The temperature and flow rate of each alkoxide were chosen to give a deposition rate close to but less than 100Å min⁻¹. Once these alkoxide temperature and flow parameters were determined, they were held constant while varying the substrate temperature and environmental conditions, such as the presence of plasma or additional chemical species. A summary of reaction conditions appears in Table II.

TABLE I

Method	<u>Time</u>
Immersed in $H_2SO_4/(NH_4)_2S_2O_8$ solution at 135°C Cooled in air	30 min.
Rinsed in distilled water Dip in 5% w/w aqueous HF Rinsed in distilled water	10 sec.
Rinsed in ethanol	
Drag wiped with ethanol in clean bench	

TABLE II

Material*		Carrier Flow (sccm)		Source <u>Flow (sccm)</u>		Source Temperatur	e (°C)
Alumina Hafnia Zirconia		100 100 100		100 100 100		80 26 (room 26	temp.)
*All were	deposited	at 500 mTorr	nressure	and angle of	deposition	of 45°.	

Five modes of growth conditions were investigated. One involved the simple thermal decomposition of the precursor to give an oxide film at 400°C. A second involved the deposition at a lower substrate temperature of 250°C. The third involved maintaining the 250°C substrate temperature and adding 20 sccm of oxygen saturated with tertiary-butanol vapor at 26°C as a deposition rate enhancer. This method is known as the Chemical Assisted Deposition (CAD) process. The fourth mode used a continuous application of RF plasma during deposition. Finally, the fifth deposition scheme used a pulsed plasma cleaning step interleaved with successive growth steps. In this process the CAD deposition method was used to grow approximately 70Å of material, after which growth was interrupted and a 100% oxygen flow was started to introduce an oxygen plasma as a cleaning step. Plasmas were generated using an RF Plasma Products RF-10 plasma generator operating at 13.56 MHz with 1 kW total available power. Fifty (50) watts of power was delivered to the plasma by a pair of clamshell electrodes placed outside the reactor tube. An AM-10 "pi" matching network was used to correct for impedence mismatch. The plasma electrodes were positioned approximately four inches downstream from the substrate holder for continuous plasma deposition, due to the fact that the plasma greatly enhanced the reaction rate and the reactants were fully depleted at the position of most intense discharge. For the plasma cleaning method, the electrodes were positioned at the location of the substrate for the most intense plasma to exist near the growing film.

Optical transmission spectra were recorded with a Perkin-Elmer spectrophotometer model 337 between 1200 nm and 200 nm. The optical data were used to determine the index of refraction of the films at or near 400 nm using equation 1.⁷

SPIE Vol. 1047 Mirrors and Windows for High Power/High Energy Laser Systems (1989) / 25

$$n = \left[n_{S} \left(\frac{1 + \sqrt{R}}{1 - \sqrt{R}} \right) \right]^{1/2}$$

 $R = 1 - T_m$

(1)

$$T_{m} = \frac{1}{0.965}$$

where: T = observed transmittance

- n = index of film
 - $n_s = index of substrate$
 - R = reflectance
 - $T_m = transmittance of thin film$

The thickness of the films was also determined using the familiar relationships between the first and higher order reflection peaks from single layer coatings. The effects of inhomogenous film deposition was modeled using an IBM PC-AT running FILM*CALC software.⁸ Index gradients were approximated as step increments of either slightly increasing or slightly decreasing index of refraction and the results compared with the observed transmission spectra.

Damage thresholds were measured using the Variable Pulse Laser at LLNL. Damage testing was performed as 1-on-1 tests using a beam at 1064 nm with 10 nsec pulse duration and 2-3 mm spot size. The criterion for laser damage was any detectable visual change in the surface of the substrate when comparing the selected areas before and after irradiation at 100X magnification in a Nomarski optical microscope. The damage data were collected and plotted as damage threshold versus layer thickness.

3. RESULTS AND DISCUSSION

Table III presents the index of refraction of films as a function of the material identity and the processing conditions. In general, lower deposition temperatures result in lower indicies of refraction. This indicates that higher temperatures are desirable in preparing multilayer dielectric stacks with the minimum number of layers. The index of refraction can be influenced by the crystal structure and/or the microstructure of the film. X-ray diffraction measurements on the films created at 400°C reveal them to be crystalline in the case of zirconia and hafnia, with the hafnia films existing as the monoclinic phase and the zirconia as the cubic phase. All alumina films in this study as well as all films deposited below 400°C are amorphous to X-ray diffraction. In addition to amorphous crystal structure, lower substrate temperatures also lead to more porous films, due to reduced surface migration of the deposition species during film growth.⁹ The level of analysis here does not allow one to determine the relative contributions of crystal structure and microstructure changes on the index of refraction, but note that all alumina films are amorphous, meaning index differences are driven by microstructure. This would indicate microstructure plays a dominant role in this material and perhaps in the others as well.

26 / SPIE Vol. 1047 Mirrors and Windows for High Power/High Energy Laser Systems (1989)

MICRON Ex.1055 p.8

TABLE III Alumina 1.48-1.66 400°C 1.47-1.52 250°C Zirconia 1.89-1.99 400°C 1.78-1.89 250°C Hafnia 1.8 400°C 1.62-1.78 250°C

The reflectance, or transmittance, spectrum allows one to calculate thickness and index of refraction as well as give clues regarding absorption or inhomogeneities in the film. Figure 2 compares the optical transmission spectra between alumina films grown at 400°C and 250°C. The most obvious differences are the decreased amplitude of modulation of the transmission signal and the loss in transmittance in the shorter wavelengths for the 400°C deposited films. The loss in modulation amplitude illustrates the decrease in index of refraction. The differences in short wavelength transmissivity could be explained by either an increase in scatter or the presence of absorbing species in the 400°C films.



Figure 2. Optical transmission spectra of alumina films deposited at 250°C and 400°C.

Qualitative off-axis viewing of an intense white light shining through the films show no difference in the scatter intensity between high and low temperature films. Therefore, the loss in transmissivity of the short wavelengths is attributed to absorbing centers in the films. Indirect evidence for this is derived by observing the decomposition products of the high temperature thermal process. Oils are trapped in the vacuum system over long deposition periods; these oils can be incorporated into the films during growth. The source of these degradation products are attributed to the chemistry of decomposition of alkoxides which involves formation of olefins.¹⁰ Hot surfaces can polymerize these olefins. The organic oils are not observed with lower deposition temperatures.

SPIE Vol. 1047 Mirrors and Windows for High Power/High Energy Laser Systems (1989) / 27

MICRON Ex.1055 p.9

TABLE IV

	Deposition Rate (A min [*])				
Source	A (400°C)	B (250°C)	C (CAD 250°C)		
ATB	60	12	20		
AIP	63	9	32		
НТВ	24	21	21		
ZTB	44	30	33		

Table IV, Columns A and B, show the rate of deposition for a given source material at high (400°C) and low (250°C) temperature. Alumina and zirconia rates are much more temperature dependent than hafnia, indicating a surface reaction kinetics limitation for those two materials. This table points out a fundamental contradiction between the necessity of low deposition temperatures, to render the organic by-products inert, and higher deposition temperatures which boost reaction rates. Equations 2 and 3,

 $HOC(CH_3)_3 \rightarrow H_2O + (CH_3)_2C:CH_2$ (2)

 $M(OC(CH_3)_3)_x + xH_2O \rightarrow MOH'' + xHOC(CH_3)_3$

illustrate the chemical reactions used. Tertiary butanol is completely dehydrated at 250°C to give water and isobutylene. The water is formed at the hot substrate and reacts with available metal alkoxide to form intermediate species either characterized as oxide or possibly hydrous oxide. The alcohol formed from liberation of the alkoxide ligands also undergoes deydration, providing more water. The tert-butoxides are extremely unstable hydrolytically, thus the additional t-butanol acts as a water transport agent and boosts the rate of reaction. Column C in Table IV shows the rate enhancement using CAD. Note that hafnia is not affected, consistent with a mass transport limited reaction.

Figure 3 shows the optical spectrum resulting from an alumina film prepared with a continuous RF plasma. This optical curve cannot be explained by simple absorption or scatter, as the peaks in the curve rise above and fall below the curve for the uncoated substrate as a function of wavelength. A film with a homogeneous index of refraction whould have the transmission maxima co-incident with the curve for the uncoated glass.

The FILM*CALC optical modeling code was used to examine the effect of index inhomogeneity on the calculated transmission spectrum. The single layer alumina film was modeled to be a composite of 10 individual layers with slightly different indicies. Figure 4a shows a linear ramp of index, each component being equivalent in optical thickness, which progresses from high index at the film-glass interface to low index at the film-air interface. Figure 4b shows a fair agreement with the

28 / SPIE Vol. 1047 Mirrors and Windows for High Power/High Energy Laser Systems (1989)

MICRON Ex.1055 p.10

(3)



Figure 3. Optical transmission spectra of alumina deposited with continuous 50 W RF plasma.

observed spectrum. Allowing the program to optimize the thicknesses of the components produces the results shown in Fig. 5. The index profile (Fig. 5a) had become non-linear while the fit to the observed spectrum improved (Fig. 5b). Other index profiles resulted in poorer fits. Qualitatively, one can conclude that a smoothly decreasing index profile versus depth contributes to the unusual features of the optical spectrum produced by plasma assisted deposition in this study.



used to model continuous plasma deposited alumina film.

optical spectrum using linearly ramped index profile of alumina film.

1200

SPIE Vol. 1047 Mirrors and Windows for High Power/High Energy Laser Systems (1989) / 29

Observed Ramped index





- 5a. Index profile result after optimization of calculated optical spectrum of alumina film.
- 5b. Calculated versus observed optical spectrum after optimization of index profile.

The laser damage threshold data as a function of layer thickness for alumina, zirconia, and hafnia are given in Figs. 6, 7, and 8, respectively. For the alumina case, high temperature (400°C) deposition results in the lowest laser damage thresholds, about 5 J/cm^{-2} . The low threshold is roughly constant, regardless of the layer thickness. Decreasing the temperature and applying the CAD method increases the damage threshold for thinner films to about $10-12 \text{ J/cm}^{-2}$, but the damage threshold decreases with increasing thickness until a level approximating the threshold for the high temperature films is reached. By intermittently cleaning the growing film with oxygen plasma, the laser damage threshold on layer thickness to an optical thickness of 1400nm corresponding to a physical thickness of approximately 2200Å. These results suggest that while the plasma cleaning step has not changed any of the bulk average optical properties such as index of refraction or scatter, it has greatly increased the "optical hardness" of the alumina film.



Figure 6. Laser damage threshold versus film thickness for alumina films.

30 / SPIE Vol. 1047 Mirrors and Windows for High Power/High Energy Laser Systems (1989)



 Laser damage threshold versus film thickness for zirconia films.



 Laser damage threshold versus film thickness for hafnia films.

The zirconia data present a similar trend in the damage performance as a function of preparation conditions. Values of damage threshold for high temperature coatings are the lowest at approximately 8 J/cm^{-2} . An increase to approximately 12 J/cm^{-2} can be achieved by using the CAD deposition method. Using the pulsed plasma method, damage thresholds of approximately 18 J/cm^{-2} can be reached. Zirconia displays an apparent anomalous increase in laser damage threshold with increasing film thickness. No further attempt was made to try to understand this phenomenon. It is clear from reviewing the laser damage data that hafnia films are the least sensitive to deposition conditions of all the materials tried using LTCVD in this laboratory. However, the general trends are still apparent. High temperature CVD produces films with the lowest damage threshold of the set, but the highest found for any of the materials at 400°C (10 J/cm^{-2}). Preparation using the CAD method causes an increase in the laser damage to approximately 18-20 J/cm^{-2} and employing pulsed plasma causes little or no improvement in laser damage.

4. ACKNOWLEDGEMENTS

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SPIE Vol. 1047 Mirrors and Windows for High Power/High Energy Laser Systems (1989) / 31

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MICRON Ex.1055 p.14