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A Review of 1064-nm Damage Tests of Electron-Beam Deposited Ta_2O_5/SiO_2 Antireflection Coatings

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ABSTRACT

Damage tests of Ta_2O_5/SiO_2 antireflection films deposited under a variety of conditions showed that thresholds of films deposited at 175 C were greater than thresholds of films deposited at either 250 C or 325 C. Deposition at high rate and low oxygen pressure produced highly absorptive films with low thresholds. Thresholds did not correlate with film reflectivity or net stress in the films, and correlated with film absorption only when the film absorption was greater than 10^4 ppm. Baking the films for four hours at 400 C reduced film absorption, altered net film stress, and produced an increase in the average damage threshold.

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1. Introduction

This study was conducted to determine the influence of deposition parameters on laser damage thresholds of silica/tantala antireflection (AR) coatings, and to determine what, if any, correlation exists between damage threshold and 1) the substrate material and the method of substrate polishing, 2) the net film stress, 3) the average absorption of the film, and 4) the reflectivity of the film.

2. Samples

The samples were four-layer silica/tantala AR films deposited by electron-beam evaporaton. An undercoat[1] layer of silica with an optical thickness of a halfwave at 1064-nm was deposited between the AR film and the substrate.

Three sets of coatings were produced. The first set (A) of coatings was deposited in separate runs under each of eighteen different deposition conditions corresponding to the unique combinations of three deposition temperatures (175 C, 250 C and 325 C), three values of oxygen pressure during the deposition of the tantala layers (0.5×10^{-4}, 1.0×10^{-4} and 2.0×10^{-4} Torr) and two rates of deposition (1.5 Å/sec and 5 Å/sec). In each run, coatings were deposited on two fused silica substrates to be used for damage samples, and on thin fused silica substrates to be used in measurements of coating stress and absorption. These eighteen runs were then repeated to provide information on the reproducibility of the results. These repeat samples constitute the second set (B) of coatings.

The third set (C) of samples was fabricated after damage tests of coatings in sets A and B indicated that the optimum deposition parameters were a substrate temperature of 175 C, an oxygen pressure of 1 x 10^{-4} Torr, and a rate of 1.5 Å/sec. Using these deposition parameters, three additional coating runs were made. In each of these runs, coatings were deposited onto two conventionally polished fused silica substrates, two conventionally polished BK-7 glass substrates, and two bowl-feed polished BK-7 substrates.

Substrates were made of Suprasil II fused silica (a product of Amersil, Inc.) or PH-3 quality BK-7 glass. Bowl-feed polishing was done by Optical Coating Laboratory, Inc. In the bowl-feed process, the slurry is recirculated and breaks into successively finer particles.[2] Details of the process are proprietary and may vary among vendors. Conventional polishing was done by Zygo, Inc., using standard fresh-feed procedures specified by LLNL for high power laser components.

3. Data and Discussion

Laser-damage thresholds were measured[3] with 1-ns, 1064-nm pulses focused to provide an approximately Gaussian beam which was 2.5 mm in diameter at the sample surface. Each test site on a sample was irradiated once. The sites were examined and photographed before and after irradiation, using a Nomarski microscope at a magnification of 100 X. Damage was defined to be any permanent alteration of the surface that was detectable by the Nomarski inspection.

For each shot, we recorded the beam profile and the pulse energy, and computed the peak on-axis fluence. Threshold was defined to be midway between the lowest fluence that caused damage and the highest fluence that caused no damage.

Damage thresholds for samples in sets A and B are shown in Table 1. Films deposited at 175 C had thresholds that were generally greater than thresholds of films deposited at 250 C or 325 C. The films with the lowest thresholds were those deposited at the greatest rate (5 Å/sec) and lowest oxygen pressure (0.5×10^{-4} Torr).

Thresholds for samples in set C are given in Table 2 and summarized in Table 3. For both fused silica and BK-7 substrates, thresholds were greatest for films deposited on bowl-feed-polished surfaces.[4]

The absorption and net stress of the films in sets A and B were determined by measurements made on thin witness samples that were coated in each run. Film absorption was measured by laser calorimetry.[5] Net stress was determined by using a Fizeau interferometer to measure stress-induced deformation[6] of the witness. Measured values of stress and absorption are given in Table 4. Absorption was largest for films deposited at the higher rate and at low oxygen pressure, which suggests that the absorption was due to incomplete oxidation of the film materials. Net stress was greater in films deposited at 175 C than in films deposited at either 250 C or 325 C, but not strongly dependent on either deposition rate or oxygen pressure.

After these initial tests, we baked a subset of the films in air for four hours at 400 C and remeasured the damage threshold, absorption, and stress. Values measured before and after baking are shown in Table 5.

As a result of the bake, stress was reduced in magnitude and converted from compressive to tensile. Absorption was greatly reduced in some films and thresholds were, on the average, slightly increased. However, inspecton of the data in Table 5 indicates slight, if any, correlation between thresholds and either net stress or film absorption in either baked or unbaked films.

Finally, a Beckman DK2A two-beam spectrophotometer was used to measure the reflectance of each film in sets A and B. Reflectance values ranged from 0.01 to 0.18 percent except for one film with large absorption whose reflectance was 0.33 percent. This demonstrates that, low reflectance can be obtained in films deposited under a variety of deposition conditions; however, measurement of reflectance does not identify films with high damage thresholds.

4. Conclusions

Thresholds of silica/tantala antireflection films deposited by electron-beam evaporation on bowl-feed polished silica substrates were greatest when the films were deposited at low temperature (175 C) at low rate (1.5 Å/sec), and in the presence of adequate oxygen (1×10^{-4} Torr). Thresholds did not correlate with the measured net stress or the film reflectance, and correlated with film absorption only when the film absorption was greater than 10^4 ppm. Baking films for four hours at 400 C reduced film absorption, altered film stress, and produced some increase in the average damage threshold.

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Table 1.

Damage Thresholds (1-ns, 1064-nm) of Ta_2O_5/SiO_2 AR coatings with $\lambda/2$ SiO_2 undercoats deposited onto bowl-feed polished fused silica at three substrate temperatures, three oxygen pressures, and two deposition rates.

Deposition Rate (Å/s)	O_2 Pressure $\times 10^{-4}$ Torr	Substrate Temperature of 175 C: Sample*	Substrate Temperature of 175 C: Threshold (J/cm^2)	Substrate Temperature of 250 C: Sample	Substrate Temperature of 250 C: Threshold (J/cm^2)	Substrate Temperature of 325 C: Sample	Substrate Temperature of 325 C: Threshold (J/cm^2)
1.5	0.5	A-1a	13.0 ± 3.0	A-4a	7.1 ± 0.7	A-7a	6.6 ± 0.7
	"	A-1b	9.6 ± 1.0	A-4b	6.6 ± 1.0	A-7b	6.8 ± 1.0
	"	B-1	9.0 ± 1.4	B-4	5.7 ± 1.3	B-7	3.5 ± 0.4
"	1.0	A-2a	18.7 ± 1.9	A-5a	6.9 ± 0.7	A-8a	4.7 ± 0.5
	"	A-2b	14.9 ± 1.5	A-5b	8.0 ± 0.8	A-8b	8.1 ± 0.8
	"	B-2	10.3 ± 1.0	B-5	6.1 ± 1.4	B-8	6.3 ± 0.7
"	2.0	A-3a	12.9 ± 1.4	A-6a	6.7 ± 0.7	A-9a	6.6 ± 0.7
	"	A-3b	9.5 ± 1.5	A-6b	11.0 ± 1.1	A-9b	- -
	"	B-3	11.1 ± 1.2	B-6	7.3 ± 0.9	B-9	5.8 ± 0.9
5.0	0.5	A-10a	7.1 ± 0.8	A-13a	2.2 ± 0.3	A-16a	1.8 ± 0.6
	"	A-10b	6.8 ± 0.9	A-13b	3.0 ± 0.4	A-16b	- -
	"	B-10	4.9 ± 1.3	B-13	6.0 ± 0.6	B-16	3.9 ± 0.5
"	1.0	A-11a	6.7 ± 1.0	A-14a	5.3 ± 0.5	A-17a	5.4 ± 0.7
	"	A-11b	6.9 ± 0.7	A-14a	8.0 ± 0.8	A-17b	5.5 ± 0.6
	"	B-11	9.0 ± 1.0	B-13	6.9 ± 0.9	B-17	5.9 ± 0.8
"	2.0	A-12a	11.3 ± 1.1	A-15a	9.5 ± 1.4	A-18a	6.5 ± 0.6
	"	A-12b	9.3 ± 1.2	A-15b	8.5 ± 0.8	A-18b	6.4 ± 0.6
	"	B-12	5.5 ± 1.0	B-15	5.1 ± 0.5	B-18	5.5 ± 0.8

*Samples are designated by a symbol which indicates the coating set (A or B), the particular coating run (1 through 18), and an additional designator (a or b) to distinguish two parts made in a single coating run.

Table 2

Damage thresholds (1-ns, 1064-nm) of Ta_2O_5/SiO_2 AR coatings with $\lambda/2$ SiO_2 undercoats deposited on three types of substrates at T = 175 C, Rate = 1.5 Å/sec, and O_2 pressure = 1.0 x 10^{-4} torr. Eighteen coatings were made in three separate runs.

Sample*	Substrate Material	Substrate Polish	Damage Threshold (J/cm^2)
C1-a	fused silica	conventional	9.2 ± 0.9
C1-b	"	"	6.8 ± 1.3
C1-c	BK-7	"	5.5 ± 0.9
C1-d	"	"	8.5 ± 1.2
C1-e	"	bowl feed	6.3 ± 0.6
C1-f	"	"	9.0 ± 1.0
C2-a	fused silica	conventional	5.8 ± 1.0
C2-b	"	"	3.7 ± 0.6
C2-c	BK-7	"	7.0 ± 0.8
C2-d	"	"	5.8 ± 0.6
C2-e	"	bowl feed	10.2 ± 1.0
C2-f	"	"	13.1 ± 1.3
C3-a	fused silica	conventional	9.4 ± 1.0
C3-b	"	"	11.4 ± 1.2
C3-c	BK-7	"	6.0 ± 0.8
C3-d	"	"	7.7 ± 0.8
C3-e	"	bowl feed	7.7 ± 0.8
C3-f	"	"	11.3 ± 1.2

*Sample designation indicates the coating run (C1, C2 or C3) and the particular substrate (a-f).

Table 3.

Summary of damage threshold data (1-ns, 1064-nm) for Ta_2O_5/SiO_2 AR coatings with λ/2 SiO_2 undercoats deposited on four types of surfaces. (T = 175 C, rate = 1.5 Å/sec, O_2 pressure = 1 x 10^{-4} Torr)

Material	Polish	Number of Samples	Median Threshold (J/cm^2)	Range of Observed Thresholds (J/cm^2)
fused silica	bowl-feed	3	14.9	10.3 - 18.7
BK-7	"	6	9.6	6.3 - 13.1
fused silica	conventional	6	8.0	3.7 - 11.4
BK-7	"	6	6.5	5.5 - 8.5

Table 4.

Coating absorption and net coating stress measured from witness samples coated in the eighteen coating runs comprising set A.

Deposition Rate (Å/sec)	Substrate Temperature (C)	O_2 Pressure ($x10^{-4}$) Torr	Absorption (ppm) Set A	Absorption (ppm) Set B	Net Stress (KPSI) Set A	Net Stress (KPSI) Set B
1.5	175	0.5	100	67	50	46
"	"	1.0	365	166	49	48
"	"	2.0	91	79	34	44
"	250	0.5	573	88	48	45
"	"	1.0	54	41	48	45
"	"	2.0	27	46	37	41
"	325	0.5	308	1770	37	47
"	"	1.0	38	22	38	40
"	"	2.0	26	29	-	46
5.0	175	0.5	2770	547	53	61
"	"	1.0	1730	1030	53	62
"	"	2.0	2180	1390	53	57
"	250	0.5	9450	4620	-	52
"	"	1.0	294	898	47	47
"	"	2.0	2240	46	49	48
"	325	0.5	23000	2300	26	25
"	"	1.0	39	44	39	-
"	"	2.0	27	692	49	44

Table 5.

Damage thresholds (1-ns, 1064-nm), net stress and absorption for fourteen Ta_2O_5/SiO_2 AR coatings measured before and after the coatings were baked in air for four hours at 400 C.

Sample*	Rate (Å/sec)	Deposition Temp (C)	O_2 Pressure ($\times 10^{-4}$ Torr)	Thresholds (J/cm^2) pre-bake	Thresholds (J/cm^2) baked	Net Stress* (KPSI) pre-bake	Net Stress* (KPSI) baked	Absorption (ppm) pre-bake	Absorption (ppm) baked
A-1a	1.5	175	0.5	13.0±3.0	17.4±2.0	50	-12	100	16
A-3a	"	"	2.0	12.9±1.4	20.1±2.0	34	- 1	91	20
A-4a	"	250	0.5	7.1±0.7	4.7±0.5	48	-16	573	22
A-5a	"	"	1.0	6.9±0.7	7.6±0.8	48	-13	54	22
A-6a	"	"	2.0	6.7±0.7	12.7±1.8	37	- 1	27	25
A-7a	"	325	0.5	6.6±0.7	6.8±0.7	37	-14	308	23
A-8a	"	"	1.0	4.7±0.5	5.0±0.8	38	-15	38	30
A-10a	5.0	175	0.5	7.1±0.8	8.4±0.8	53	-17	2770	43
A-11a	"	"	1.0	6.7±1.0	9.0±1.0	53	-16	1730	31
A-12a	"	"	2.0	11.3±1.1	13.0±1.3	53	-10	2180	12
A-14a	"	250	1.0	5.3±0.5	3.9±0.7	47	-20	294	41
A-15a	"	"	2.0	9.5±1.4	4.9±1.2	49	-18	2240	30
A-17a	"	325	1.0	5.4±0.7	5.6±0.6	39	-14	39	26
A-18a	"	"	2.0	6.5±0.6	6.1±0.8	49	-13	27	11

* By convention, a positive value indicates compressive stress and a negative value indicates tensile stress.