HfO₂/SiO₂ enhanced diamond turned aluminum mirrors for IR laser optics

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ABSTRACT

HfO₂/SiO₂ multilayers were deposited on single point diamond turned aluminum substrates via modified reactive plasma ion assisted deposition to form a laser durable and environmentally stable dielectric enhanced IR mirror at a wavelength of 1064nm. The effect of the surface quality of the diamond turned aluminum on the optical performance of the dielectric enhanced mirror was assessed. A laser-induced damage threshold up to 11 J/cm² was obtained from the enhanced aluminum mirror tested in pulse mode at 1064nm with a pulse length of 20ns and a repetition rate of 20Hz. Laser damage morphology was revealed by a scanning electron microscopy. The damage mechanism was attributed to nodule defects generated by particle embedded on the aluminum substrate surface.

Keywords: IR laser optics, diamond turned aluminum, HfO₂/SiO₂ multilayer coating, laserinduced damage threshold, laser damage morphology

1. Introduction

Aluminum is an important technical material, because of its high reflectance over a broad spectrum, light weight and suitability for diamond turning to enable precise and cost effective surface finishing. Single point diamond-turned aluminum (SPDT-Al) has been extensively used for IR optical systems, including reflective optics. The environmental stability of aluminum based reflective optics can be improved by adding a single layer of dielectric protective coating. For high fluence application, laser-induced damage threshold (LIDT) of the reflective optics is a concern. The LIDT of optical materials especially optical glass and crystal has been extensively investigated. It is known that laser-induced damage is a thermal-driving process where absorption and defects play dominant roles in the nanosecond pulse regime from IR down to

Laser-Induced Damage in Optical Materials: 2011, edited by Gregory J. Exarhos, Vitaly E. Gruzdev, Joseph A. Menapace, Detlev Ristau, M. J. Soileau, Proc. of SPIE Vol. 8190 819005 · © 2011 SPIE · CCC code: 0277-786X/11/\$18 · doi: 10.1117/12.900226 DUV.^{1,2} Dielectric multilayer coating may improve both the LIDT and the environmental stability of aluminum based reflective optics. In this contribution, SPDT-Al witnesses are used as coating substrates to assess the laser damage resistance of dielectric multilayer enhanced aluminum mirrors. The LIDT of the aluminum based high reflective optics is presented in order to benchmark existing surface finishing and coating processes. The laser-induced damage mechanism is discussed and the LIDT-limiting factor is proposed.

2. Dielectric multilayer enhanced aluminum mirror

Previous studies have investigated numerous coating materials for high laser damage resistance in the NIR spectral region. Among these, HfO₂/SiO₂ has been recognized as the best coating material combination, due to its high LIDT.³ For dielectric multilayer enhanced coating, HfO₂/SiO₂ films were deposited on a 1" size SPDT-Al 6061-T6 witness via a modified reactive plasma ion assisted deposition.⁴ This coating process has been described in previous publications.⁴⁻⁶ Standard quarter-wave design was selected for the HfO₂/SiO₂ multilayer. Fig. 1 plots the calculated total scatter loss of the dielectric enhanced aluminum mirror at 1064nm as a function of surface and interfacial roughness RMS (root-mean-square). To simplify the calculation, a constant surface and interface RMS was assumed. In reality, due to the in-situ plasma ion smoothing effect, the RMS of the coated surface decreases as the deposited layer number increases on a substrate with a relatively high surface roughness. The estimation shown in Fig. 1 suggests that the surface roughness must be less than 6nm in order to ensure a reflectance greater than 99.5% at a normal angle of incidence in the NIR spectral region.



Fig. 1 Total scatter loss of a dielectric enhanced aluminum mirror at 1064nm as a function of surface and interface roughness.

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Fig. 2 plots the calculated reflectance (R_x) and absorptance (Abs) of a dielectric enhanced aluminum mirror as a function of the HfO₂/SiO₂ stack period. The calculation indicates that a maximum reflectance of 94.5% can be realized for a bare aluminum mirror with a surface roughness of 6nm. Of the 5.5% loss, 5.0% is due to absorptance and 0.5% is caused by scatter loss. With dielectric multilayer enhancement, the absorption loss can be significantly reduced. After 13 periods of HfO₂/SiO₂ coating on the SPID-Al, the substrate absorptance can be reduced from 5.0% to 10 ppm. As a result a reflectance of 99.5% can be realized on a RMS of a 5nm substrate. The extremely low absorptance ensures the high laser resistance of the enhanced mirror.

A scanning electron microscopy (SEM) with focus ion beam (FIB) cutting capability was employed to reveal the SPDT-Al substrate as well as the multilayer coating structure. Fig. 3 shows SEM cross-sectional image of the HfO_2/SiO_2 multilayer enhanced diamond turned aluminum mirror. A periodic structure of HfO_2/SiO_2 is revealed. The total coating thickness is 4.6 μ . Dense and homogeneous film structure is achieved through modified reactive plasma ion assisted deposition.



Fig. 2 Calculated reflectance (Rx) and absorption (Abs) of a HfO₂/SiO₂ enhanced aluminum mirror as a function of dielectric stack period.



Fig. 3 SEM images of HfO₂/SiO₂ multilayer enhanced Al mirror.

3. Laser induced damage threshold and damage mechanism

3.1. LIDT test

An LIDT test was performed using an Nd:YAG laser at a wavelength of 1064nm, a repetition rate of 20Hz, and a pulse length of 20ns. The output laser beam was in TEM_{00} mode with a 1.03mm spot size at $1/e^2$. Damage was determined by observing plasma formation, increased He-Ne laser scatter, and visible structural change with a 150x Nomarski brightfield microscope. Table 1 lists the main LIDT test parameters.

Wavelength (nm)	1064	Polarization	Linear
Repetition rate (Hz)	20	Test beam profile	TEM ₀₀
Pulse length (ns)	20	Number of sites	80
Spot diameter at 1/e ² (mm)	1.03	Shot per site	200
Incident angle (°)	0	Test method	Least fluence failure

Table 1 LIDT test parameters

Fig.4 plots the exposure histogram of a laser-induced damage test. During the damage test, 10 exposure sites were tested for a given fluence and each site had 200 shots. Here the LIDT is defined as the highest irradiated fluence which does not generate any damage. As seen in Fig. 4, no damage is detected when the laser fluence is increased step by step up to 11 J/cm². 6 damage

sites are generated as the laser fluence reaches 11.5 J/cm². As a result, a LIDT of 11 J/cm² is determined for the tested dielectric enhanced SPDT-Al mirror.



■Damage ■No Damage

Fig. 4 Exposure histogram of a laser-induced damage test.

3.2. Laser-induced damage morphology and mechanism

Based on the exposure histogram of the laser-induced damage test plotted in Fig. 4, there are 16 damage sites on the test mirror. Fig. 5 shows an SEM cross-sectional image of the laser-induced damage site. The cross-sectional image reveals detail of the laser-induced damage. The damage site is filled with platinum, which was placed down over the damage region prior to FIB cutting in order to preserve the damage shape and to ensure a sharp cutting edge. The damage depth is deeper than the total thickness of the HfO₂/SiO₂ multilayer. Significant damage appears on the substrate.



Fig. 5 SEM cross-sectional image of the laser-induced damage site.

Previous studies of laser-induced damage on HfO_2/SiO_2 multilayer mirrors deposited on glass substrates indicated that deeply embedded particles lead to nodule defect formation, resulting in light intensification under laser irradiation.⁷⁻⁹ Fig. 6(a) illustrates the typical geometry of a nodule defect generated by a particle inclusion located on a substrate surface, in which the nodule defect diameter (D), inclusion diameter (d), and film thickness determine the maximum angle of incidence ($\Phi/2$). Fig. 6(b) illustrates the simulated light-intensification of the nodule defect under laser irradiation.⁷ Based on the damage geometry shown in Fig. 5, the calculated maximum light intensification at the defect location for the p-polarized component is 80x greater than that of the defect-free region at the interface of multilayer coating and the SPDT-Al substrate. As a result, the intensified light leads to the deep damage of the aluminum substrate as shown in Fig. 5.



Fig. 6 Schematics of a nodule defect formation and light-intensification effect. (a) Nodule defect generated by a particle inclusion on substrate surface and (b) light-intensification of a nodule defect through HfO₂/SiO₂ multilayers down to the substrate as simulated at normal angle of incidence.⁷

3.3. Laser-induced damage on SPDT-Al and glass substrates

It is worthwhile to compare the laser-induced damage on SPDT-Al and the laser-induced damage on glass substrates. Fig. 7 shows SEM cross-sectional images of a nodule defect before and after laser irradiation.⁹ The HfO₂/SiO₂ high reflector was deposited by traditional electron beam evaporation on a BK7 glass substrate. As shown in Fig. 7(a), it is clear that the nodule defect is caused by a particle seed located on the glass substrate. Due to a reduced mechanical strength at the defect site, a pit is generated in region where the nodule effect is located by the laser irradiation. Fig. 8 illustrates an SEM cross-sectional view of 2 nodule defects on the HfO₂/SiO₂ multilayer enhanced SPDT-Al mirror. The defects are caused by 2 particle seeds located on the SPDT-Al substrate. It is known that the shape and size of a nodule defect depends on the angular range of the depositions as well as the deposition process. The modified reactive plasma ion assisted deposition tends to eliminate large diameter nodule defect formation when compared with the nodule defect derived from the tradition electron beam evaporation as shown in Fig. 7(a). It is clear that the nodule defect shown in Fig. 7(a) is initiated by a circular particle seed, leading to a smooth cone shaped defect structure. The 2 nodule defects shown in Fig. 8, however, are generated by random shaped particles. The surface structure of the random shaped seeds is duplicated as the layer number and thickness of the HfO₂/SiO₂ multilayer increases. As a result, ripples form around the nodule defects.



Fig. 7 SEM cross-sectional images of (a) a nodule defect initiated from a particle seed located on the glass substrate, and (b) a pit generated after laser irradiation where a nodule effect located.⁹

In addition to the nodule defect formation, in the case of HR on glass substrate, laser-induced damage leads to the ejection of the nodule defect. No damage occurs to the glass substrate. In the case of the SPDT-Al substrate, however, the low melting point and high absorption of aluminum

bulk result in severe laser-induced damage to the substrate. As soon as substrate damage begins, the entire nodule defect can be ejected, due to relatively weak boundaries when compared to those of a defect-free region. Because the dielectric multilayer significantly reduces light intensity, which can penetrate into the aluminum substrate, the surrounding nodule defect-free area survives under the same laser fluence irradiation.



Fig. 8 SEM cross-sectional view of 2 nodule defects on the HfO₂/SiO₂ multilayer enhanced SPDT-Al mirror. The defects are grown from 2 random shaped particles located on the SPDT-Al substrate surface.

4. Conclusions

HfO₂/SiO₂ multilayers were deposited on an SPDT-Al witness substrate to assess the optical performance and laser damage resistance of dielectric enhanced high reflective optics at a wavelength of 1064nm. A reflectance greater than 99.5% was realized on the aluminum substrate with 13 periods of HfO₂/SiO₂ multilayer enhancement via modified reactive plasma ion assisted deposition. An LIDT up to 11 J/cm² was achieved on the enhanced SPDT-Al 6061-T6 witness by a 1064nm Nd:YAG laser with a pulse length of 20ns and a repetition rate of 20Hz. Laser-induced damage sites were investigated by means of an SEM with FIB cutting capability. Deep substrate damage was attributed to the light-intensification of nodule defects generated by embedded particles located on the SPDT-Al substrate surface. The modified reactive plasma ion assisted deposition tends to eliminate large diameter nodule defect formation when compared with the nodule defect derived from tradition electron beam evaporation.

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References

1. M. J. Soileau, "40 year retrospective of fundamental mechanisms," SPIE 7132, 7132-01(2008).

2. Jue Wang, Steven VanKerkhove, Horst Schreiber, "Evaluation of coated and uncoated CaF₂ Optics by variable angle spectroscopic ellipsometry," *Thin Solid Films 519*, 2881(2011).

3. Christopher J. Stolz, Michael D. Thomas, and Andrew J. Griffin, "BDS thin film damage competition," *SPIE* 7132, 7132-17(2008).

4. Jue Wang, Horst Schreiber, "Low loss dual-wavelength laser optics coatings at 1060nm and 530nm," *SPIE 8016*, 80160Y(2011).

5. Jue Wang, Robert L. Maier, Horst Schreiber, "Elastic and plastic relaxation of densified SiO₂ films," *Applied Optics* 47(13), C189(2008).

6. Jue Wang, Robert L. Maier, Horst Schreiber, "Crystal phase transition of HfO_2 films evaporated by plasma ionassisted deposition," *Applied Optics* 47(13), C131(2008).

7. Christopher J. Stolz, Michael D. Feit, and Thomas V. Pistor, "Laser intensification by spherical inclusions embedded within multilayer coatings," *Applied Optics* 45(7), 1594(2006).

8. Christopher J. Stolz, Scott Hafeman, and Thomas V. Pistor, "Light intensification modeling of coating inclusions irradiated at 351nm and 1053nm," *Applied Optics* 47(13), C162(2008).

9. Xinbin Cheng, Zhengxiang Shen, Hongfei Jiao, Jinlong Zhang, Bin Ma, Tao Ding, Jiangtao Lu, Xiaodong Wang, and Zhanshan Wang, "Laser damage studey of nodules in electron-beam-evaporated HfO₂/SiO₂ high reflectors," *Applied Optics 50(9)*, C357(2011).

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Questions and Answers

Q. It seems to me it's worthwhile to include for consideration the thermal coefficient, thermal conductivity coefficient for aluminum which is largely different. That's the whole idea, but you didn't even mention it. Is there any reason not to mention it at all? It seems to me,... you showed nice slides when you increased the angle, the fields started penetrating into the aluminum. How low would the threshold be if you go with 95 % reflection versus 100%? You are decreasing it to 1 J/cm² or a fraction of that. Certainly it would be good to know. An aluminum mirror could potentially be better in the long run. It seems, intuitive to me that the substrate is highly conductive if you make the field not penetrate as far, then you would have a relaxing effect. But another thing you did not mention is CTE mismatch. Especially when you spoke about nsec pulses where you have thermal damage not multiphoton ionization. This is not also mentioned. Just to calibrate us, various things in aluminum versus hafnia, in silicon versus glass is also a better mismatch. These two parameters seem to fall out of your consideration completely. Maybe you could comment why and how you would include them?

A. Yes, that's a good question. We do need to consider the substrate and CTE and other properties. But basically, we try to put enough of a stack on the top and also I'm assuming that if it's thermal expansion related damage we should see tit on a larger scale since the laser beam spot is 1 mm in diameter. But we observe the damage on only a micrometer scale. That's why I did not include the CTE. Also, it's more difficult for me to explain what happens in the surrounding area of this thin film that survived. That's the reason.

Q. I understand that you checked the enhancement only at a wavelength of 1064 nm. What do you suppose would happen at other wavelengths, for example, 1054 nm, 1069 nm? Is it better, or worse, or would you get the same result?

A. Yes. That is a good question. If we design properly, we can move the spectral stack to other wavelengths. That's one way to make sure you wavelength has the highest reflectance. Another way is that we can design a broader wavelength mirror to cover all of those wavelengths.

Q. You think the damage you observed is caused by the low spatial frequency due to your diamond turning process or is it due to high spatial frequency?

A. The first damage is triggered by nodule defects of 3 micrometer size. This is pretty clear. The larger size, my assumption is that it is related to the high frequency structure with the high peak to valley.

Q. Since you have the high frequency single diamond turning process, can you generate some patterns on purpose?

A. Yes, that's something we are going to do. In the meantime, there is another process. We can further smooth out, but we haven't done that yet. We are doing that work but this is the first work we have done in this area. We have studied here the effect of the laser on the bare diamond turned aluminum surface without extra surface improvement. This is just benchmarking our existing process.

Q. Have you done any work on pure aluminum instead of 60/61 or on oxygen free copper?

A. Yes. We are exploring different aluminum alloys. We are currently working on that. Results will be out next year.