A Study on Laser Induced Damage Threshold of SiO\textsubscript{2} and TiO\textsubscript{2} Thin Films

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ABSTRACT

Laser induced damage threshold of SiO\textsubscript{2} and TiO\textsubscript{2} thin films prepared by e-beam evaporation technique on BK7 glass substrate at normal without heating. TiO\textsubscript{2} and SiO\textsubscript{2} deposited by such method were annealed in air for an hour at temperature of 250°C. X-ray diffraction of these samples shows the amorphous behavior of the SiO\textsubscript{2} and TiO\textsubscript{2} films. Using spectrophotometer standardization for quarter wave optical thickness (QWOT) were calculated reflectivity of TiO\textsubscript{2}, at quarter wave optical thickness confirms its reflective behavior while that of SiO\textsubscript{2} film confirms anti-reflective behaviors. Simulation of above film design performed with OpenFilter software. Electric field intensity and reflectivity are characterized with this software. Simulation results show that standing wave electric field is higher in TiO\textsubscript{2} as compare to SiO\textsubscript{2} film and thus due to this higher electric field damage thresholds of TiO\textsubscript{2} will be lower than the SiO\textsubscript{2} films.

Keywords: Quarter wave optical thickness (QWOT), Reflectivity, E-beam evaporation, Simulation, Amorphous.

INTRODUCTION

In the field of laser and its applications, high reflective coatings (HRCs) have been possessed a lot of challenges with the significant decrement in laser induced damage thresholds as the absorption edge of film specimen is concerned and are affected significantly due to presence of high electric field at interface of the high and low index layers or inside a low damage resistant material of the stack. The electric field distribution has a significant importance which can be understood by considering the laser damage threshold of film specimen, which can be used by depositing with low defect density as well as low absorption and a high value of resistance corresponding to laser damage.\textsuperscript{1}

The damage threshold of the material depends on the wavelength of laser, its pulse width, number of pulses, surface and bulk qualities of the material. The laser induced damage studies in terms of these parameters allow a complete characterization of the damage process in terms of the intrinsic or extrinsic thermo-physical and metallurgical properties of the material.\textsuperscript{2}

Thermal induced stress within the film has been proposed as a mechanism of failure which requires relatively lower temperatures than melting. It is suggested that material damage by short pulse lasers may be independent of absorption, at least the type of absorption normally associated with optical materials. It is propsoed that the local electric field intensity created by lasers is sufficient to induce a self-absorption phenomenon which rapidly extracts radiant energy from the laser pulse and deposits it in the form of heat in the material.\textsuperscript{3}

The electrons in case of wide band gap dielectrics, few tens of picoseconds pulse get excited electrons into conduction band (CB) from its valance band by the incident light, which further resulting into creation of phonons by transfer their energy to the lattice. As a result, if the depositing amount of heat is in sufficient amount to alter materials, then the wide band gap dielectrics damage occur. The critical energy density at which damage occurs corresponds to a critical electron density in the conduction band of \(N_{cr} = 10^{16} - 10^{17}\) cm\textsuperscript{-3}.\textsuperscript{4}

The dielectric materials absorbs the laser energy whenever the energy density reaches up to a desired cutoff limit which further resulted into ablation and structural changes in the dielectric specimen. The electric field distribution within the specimen has a significant effect over the rate of various non-linear phenomenon. The more general case showing this effect is of multilayer coatings, where a lot of attempts have been taken into considering for the modification of electric field distribution in dielectric specimen in order to enhance the LIDTs. Because the interface of high and low refractive index within the dielectric materials can be considered as a weakest region,

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hence a lot of investigations have been done and shown that by shifting the peak electric field value to a region of low resistance refractive index materials, one can reach a limit which is in accordance with the LIDTs enhancement as determined by dielectric properties of the concerned materials.\textsuperscript{5,6}

**EXPERIMENTS**

We used BK7 glasses to standardize QWOT of these films and got four glass sample pieces prepared for this experiment are on which thin films were deposited. High-quality BK7 substrates are super polished and cleaned ultrasonically in alcohol solution having identical preparation before deposition. All films are electronic beams deposited with the similar process parameters like the substrate temperature were kept at normal cooled nearly 30\(^\circ\)C during deposition, the base chamber vacuum were 8.6x10\(^{-6}\) mbar, the deposition rates of TiO\(_2\) and SiO\(_2\) are 0.25 and 0.15\AA/s, respectively. The two samples (one of each) are annealed at 250\(^\circ\)C in air for an hour after deposition. The reflectance spectra of films are measured with a Varian CARY 5000 spectrometer. The surface structure of samples was investigated by X-ray diffractometer (Rigaku Miniflex II). The results are compared with standard powder diffraction PDF card files. LIDTs of coatings at 532nm are measured using the Nd:YAG laser system.\textsuperscript{7,8} The effective spot diameter is around 43\(\mu\)m. For getting idea about LIDT of these coatings at 532nm, 10ns laser pulse is tested in the one-on-one mode with the ISO standard\textsuperscript{11254-1}.\textsuperscript{7,9} The damage morphology after laser radiation is observed with microscope.

**RESULTS AND DISCUSSION**

**MICROSTRUCTURE**

The samples were measured by XRD with 20 angle in the range of 10\(^\circ\) to 80\(^\circ\) using filtered Cu -K\(_x\) radiation in steps of 4\(^\circ\). The inter-planar distance d was calculated by the equation of 2d sin \(\theta = n\lambda\), where \(\theta\) is the Bragg diffraction angle, \(\lambda\) is 0.154 nm of the Cu- K\(_x\) radiation.\textsuperscript{7,11} X-ray diffraction pattern has been used to investigate the phase of the prepared TiO\(_2\) thin films. The X-ray diffraction pattern displays the existence of amorphous TiO\(_2\) regions by showing the presence of the broad hump in the low 2\(\theta\) region. In case of SiO\(_2\) also the absence of any peak confirms the amorphous nature of film. The amorphous phase of SiO\(_2\) depicts the absence of grain in the grown film. The presence of grain boundaries usually provides a path for the transport of charge carriers which is not seen here.

This demonstrating short range order and amorphicity.\textsuperscript{12} XRD pattern of TiO\(_2\) and SiO\(_2\) both shows the amorphous behavior as shown in the figure 1 and 2.

**OPTICAL PROPERTIES**

The reflectance spectra of single layer TiO\(_2\) film and SiO\(_2\) film shown in the figure 3-4. TiO\(_2\) shows reflective peak at quarter wave optical thickness and we obtain about 35% reflectivity in single quarter wave thin layer at 532 nm. SiO\(_2\) is anti-reflective at quarter wave optical thickness. Valley is obtained at 532nm which is confirmation of the quarter wave optical thickness of the antireflective coating at 532nm. Similarly peak is obtained at 532nm in TiO\(_2\) quarter wave thick layer.
**Figure 4** Reflectivity of single layer of SiO$_2$ thin film.

**ELECTRIC FIELD**

Standing wave electric field of single layer of TiO$_2$ and SiO$_2$ of quarter wave optical thickness were measured with open filter software. The electric field graph shows that peak of the field lies on the air film interface in both TiO$_2$ and SiO$_2$ film. The standing wave electric field intensity of TiO$_2$ and SiO$_2$ film decreases along the direction of the depth of the film. This decreases more rapidly in TiO$_2$ than SiO$_2$ but laser induced damage threshold is higher value in SiO$_2$ because of large band gap in SiO$_2$ than TiO$_2$. So electron from valence band to conduction band easily reached in TiO$_2$ and more energy is required in SiO$_2$, which is responsible for the damage. The result of both is shown in the figure 5-6.

**Figure 5** Electric field measured in TiO$_2$ thin film.

**Figure 6** Electric field measured in SiO$_2$ thin film

**LASER DAMAGE THRESHOLD OF SINGLE LAYER OF TiO$_2$ AND SiO$_2$**

LIDT of coatings at 532nm, 10ns laser pulse is tested in the one-on-one mode with the ISO standard 11254-1.$^9$ It is found that LIDT of TiO$_2$ single layer is lower than the SiO$_2$ single layer. Damage mechanism which is responsible for damage is different in both films. TiO$_2$ films prepared by conventional electron beam evaporation may exhibit considerable absorption if proper oxidation is not maintained during evaporation. Optical absorption in TiO$_2$ films depends not only preparation techniques but also on the deposition conditions and absorption is main region for damage.$^{13,14}$ The laser radiation incident on TiO$_2$ film has very high probability of absorption if TiO$_2$ film are not in proper oxidation state and thus this heat generated is directly given to the lattice and thermal induced damage occurs in the film. Damage site of TiO$_2$ shows that very large area is damaged and damage threshold value is small. On the other hand SiO$_2$ films prepared by electron beam evaporation are highly transparent and very small absorption and possess laser damage threshold comparable to bulk silica.$^{13,15}$ In SiO$_2$ films primary damage mechanism is avalanche ionization. In avalanche ionization the damage occur due to electron density increases in the conduction band. Electrons in valence band absorb energy and excited into the conduction band, when electron density $N_{cr}$ reaches $10^{16}$ - $10^{18}$cm$^{-3}$ damage occurs.$^5$ Damage site of SiO$_2$ shows that it is deep well type. Damage morphology of TiO$_2$ and SiO$_2$ thin films are shown in the figure 7-8 respectively.

**Figure 7** Damaged site of TiO$_2$ film radiated by laser radiation and measured LIDT is 4J/cm$^2$.

**Figure 8** Damaged site of SiO$_2$ film radiated by laser radiation and measured LIDT is 9J/cm$^2$. 

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CONCLUSION

Investigations of LIDT of SiO₂ and TiO₂ thin films samples prepared by e-beam evaporation technique under similar evaporation conditions confirms that due to higher field conditions in TiO₂ films it was expected lower than that of SiO₂ and same has been confirmed 4J/cm² for TiO₂ and 9J/cm² for SiO₂ with damage results. Hence, damage thresholds TiO₂ is lower than the SiO₂ films.

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