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A Study on Laser Induced Damage Threshold of SiO₂ and TiO₂ Thin Films

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ABSTRACT

Laser induced damage threshold of SiO₂ and TiO₂ thin films prepared by e-beam evaporation technique on BK7 glass substrate at normal without heating. TiO₂ and SiO₂ 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 SiO₂ and TiO₂ films. Using spectrophotometer standardization for quarter wave optical thickness (QWOT) were calculated reflectivity of TiO₂, at quarter wave optical thickness confirms its reflective behavior while that of SiO₂ 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 TiO₂ as compare to SiO₂ film and thus due to this higher electric field damage thresholds of TiO₂will be lower than the SiO₂ films.

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

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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.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.²

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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 propose 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.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 Ncr = $10^{16} - 10^{18}$ cm⁻³.⁴

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, 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.^{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. Highquality 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°C during deposition, the base chamber vacuum were 8.6×10^{-6} mbar, the deposition rates of TiO₂ and SiO₂ are 0.25 and 0.15Å/s, respectively. The two samples (one of each) are annealed at 250°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.^{7, 8} The effective spot diameter is around 43µm. For getting idea about LIDT of these coatings at 532nm, 10ns laser pulse is tested in the one-on-one mode with the ISO standard11254-1.7-9 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 10^0 to 80^0 using filtered Cu $-K_{\alpha}$ radiation in steps of 4^0 . The inter-planar distance d was calculated by the equation of 2d sin $\theta = n\lambda$ where θ is the Bragg diffraction angle, λ is 0.154 nm of the Cu- K_{α} radiation.^{10, 11} X-ray diffraction pattern has been used to investigate the phase of the prepared TiO2 thin films. The X-ray diffraction pattern displays the existence of amorphous TiO2 regions by showing the presence of the broad hump in the low 20 region. In case of SiO₂ also the absence of any peak confirms the amorphous nature of film. The amorphous phase of SiO2 depicts the absence of grain in the grown film. The presence of charge carriers which is not seen here.

This demonstrating short range order and amorphicity.¹² XRD pattern of TiO_2 and SiO_2 both shows the amorphous behavior as shown in the figure 1 and 2.



Figure 1 XRD of single layer of TiO₂ thin film.



Figure 2 XRD of single layer of SiO₂ thin film.

OPTICAL PROPERTIES

The reflectance spectra of single layer TiO_2 film and SiO_2 film shown in the figure 3-4.TiO₂ shows reflective peak at quarter wave optical thickness and we obtain about 35% reflectivity in single quarter wave thin layer at 532 nm. SiO₂ 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 TiO₂ quarter wave thick layer.



Figure 3 Reflectivity of single layer of TiO₂ thin film.



Figure 4 Reflectivity of single layer of SiO₂ 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 valance 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 6 Electric field measured in SiO₂ thin film

LASER DAMAGE THRESHOLD OF SINGLE LAYER OF TiO₂ AND SiO₂

LIDT of coatings at 532nm, 10ns laser pulse is tested in the one-on-one mode with the ISO standard11254-1.9 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. TiO₂ films prepared by conventional electron beam evaporation may exhibit considerable absorption if proper oxidation is not maintained during evaporation. Optical absorption in TiO₂ 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 TiO₂ film hasve high probability of absorption if TiO₂ 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 TiO₂ 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 SiO₂ 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 Ncr reaches 10¹⁶ - 10¹⁸ cm⁻³ damage occurs.⁴ Damage site of SiO₂ shows that it is deep well type. Damage morphology of TiO₂ and SiO₂ 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 $9J/cm^2$.

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 $4J/cm^2$ for TiO₂ and $9J/cm^2$ for SiO₂ with damage results..Hence, damage thresholdsTiO₂ is lower than the SiO₂ films.

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