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Influence of thickness and wavelength on Laser Damage Threshold of SiO_2 and multilayer TiO_2/SiO_2 thin film

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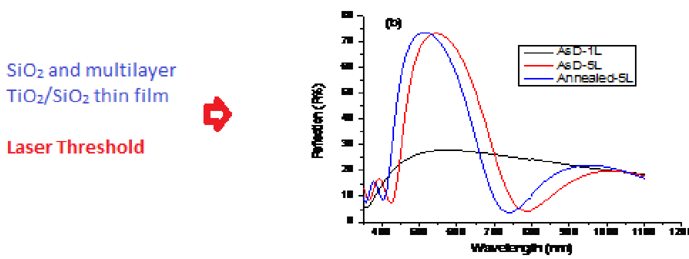
ABSTRACT



Laser induced damage threshold (LIDT) of optics is the key point since the time of invention of laser. In this study the LIDT of SiO_2 material which is used with TiO_2 in making higher reflective (HR) coating and antireflective (AR) coating, is explored on the bases of thickness of film and on two different laser wavelengths which lie in Infrared (IR) and visible regions. Samples are deposited by conventional e-beam deposition method having $\lambda/4$ and $3\lambda/4$ thickness, of SiO_2 single layer and five layer TiO_2/SiO_2 multilayers with combination of quarter wave optical thickness of each, on BK-7. Annealing of multilayers is done in air at a suitable temperature and its effect on LIDT is measured. LIDT is measured in Nano-second regime with Nd : YAG laser at 532nm and 1064nm, damage morphology is studied by scanning electron microscope (SEM) and suitable damage mechanism is found out. Optical properties of the films are investigated by spectrophotometer.

Keywords: Anti-reflective; Nano-second; E-beam; Transmission; Morphology.

INTRODUCTION

The maximum capacity of optical thin film to resist laser irradiation is called laser damage threshold. In the field of research of high power laser application, the main obstacle is laser induced damage threshold of optical components.[1-3] Laser damage threshold of thin films depends on many factors; selection of substrate whether substrate is metallic, transparent dielectric glass, or some polymers. Glass substrate is also further divided into BK-7, Borosilicate, Fused silica etc. Next important factor is substrate cleaning, before deposition freshly prepared substrate is cleaned properly otherwise impurity is embedded into the film and which reduces the LIDT.[4,5,7] Next factor is deposition of starting materials especially for dielectric coating as we use metal or metal oxide for preparation of thin film. The other factor is deposition techniques, there are a large number of thin film deposition technique thermal coating, thin film coating by sol-gel method, chemical coating, sputtering method, Ion assisted deposition, electron beam deposition methods etc., LIDT strongly depends on the type of these deposition method[5-7]. In case of e-beam deposition technique, LIDT depends upon process parameters, chamber pressure, substrate temperature, rate of evaporation.[1,8] On the other hand LIDT also depends on laser parameters, wavelength, pulse width, repetition rate, and spot size. Mechanism of LIDT is explained on the basis of pulse width, for long pulse and high repetition rate thermal damage is the main mechanism for the failure of optics and for short pulse avalanche ionization, multiphoton ionization are main mechanism.[9-11]

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In this study, the laser induced damage threshold (LIDT) of SiO_2 thin film having thickness odd multiples of quarter wave thickness and TiO_2/SiO_2 multilayers as deposited and post annealed is studied on two different laser wavelengths 1064nm and its second harmonics 532nm and effect of change in parameters and annealing on LIDT is explored.

EXPERIMENTAL

Samples of SiO_2 single layer of quarter wave optical thickness (QWOT), odd multiple of quarter wave optical thickness (3 QWOT) and five TiO_2/SiO_2 multilayers ($HL^{\wedge 2}H$) are prepared in Box type coating plant (Model BC-600 Hind High Vacuum), where H is quarter wave optical thickness of TiO_2 as high index material and L is quarter wave optical thickness of low index material. Thicknesses of samples are monitored by quartz crystal monitor and basic thickness of samples are calculated by

$$d = \frac{m\lambda}{4n} \quad (1)$$

where'd' is required thickness, 'λ' reference wavelength, 'n' refractive index of film material and 'm' is integer. A thick layer of sample is deposited on BK7 substrate then transmission spectra are measured and noted the corresponding wavelength of minimum three peaks or valleys in the transmission spectra of high reflective TiO_2 or SiO_2 antireflective coating. Then by using formulae given below we calculate how much λ/4 thickness corresponds tothe given peaks or valleys

$$K_2 = \frac{\lambda_1}{\lambda_2 - \lambda_1} \quad (2)$$

$$K_3 = \frac{2\lambda_1}{\lambda_3 - \lambda_1} \quad (3)$$

where K2 and K3 are multiples of λ/4 i.e 3λ/4, 5λ/4…… so on and λ_1 , λ_2 , λ_3 are wavelengths of first, second, third peaks or valleys respectively. Consider thick layer having thickness X, and Y be the wavelength corresponds to 5 λ/4 thickness i.e. Y is equal to 5 λ/4 at this layer thickness of X.

Thus 532 is equal to 5 λ/4 at $\frac{X}{Y}\times 532$ unit thickness, which is equal to some factor Z of thickness.

So Z thickness is equal to 5 λ/4 at 532 units.

Hence λ/4 is equal to $\frac{Z}{5}$ unit thickness. Using these formulae we calibrate quartz crystal monitor thickness.

Now chamber is loaded with substrate and starting materials in crucibles and a pressure of 5×10^{-2} mbar is achieved using rotary pump and roughing valve and simultaneously the diffusion pump started. After running plant for 20 minutes, the desired chamber pressure is achieved then closes the roughing valve and, opens the backing and high vacuum valves till we obtain pressure of order of 5×10^{-5} mbar. Substrate in the chamber is kept at 220°C with the help of gradient heater. Supply argon for 10 minutes, ion beam cleaning is performed after this pressure. Finally close the argon gas and pump the chamber by diffusion pump till the pressure reaches 2×10^{-5} mbar. Now chamber is ready for coating, oxygen gas is supplied and pressure of 3×10^{-4} mbar is maintained for SiO_2 and 3.5×10^{-4} mbar for TiO_2. The e-beam is switched on and beam is monitored, and is kept in the middle of the crucible and DC current is increased till 40-70 Ampere in case of SiO2 and up to 180-240 A for TiO_2 material. SiO_2 gets sublimated, the rate of evaporation is controlled to 3-5Å/s and 1-2 Å/s for TiO_2, evaporation is continued till the required thickness is achieved. Completing this process, chamber is cooled, if as hot sample is brought out then there is a probability of embedding the impurity from environment. Post annealing ofTiO_2/SiO_2 multilayers is done in air at a temperature of 600 °C for 20 hours and its effects on LIDT are studied.

RESULTS AND DISCUSSION

STRUCTURAL AND OPTICAL PROPERTIES

Grazing ray incidence X-ray diffraction patterns of SiO_2 single quarter wave and TiO_2 single quarter wave (thickness) of as deposited[12] and annealed samples are taken and patterns show that all the samples are amorphous in nature, figure1. After annealing, there is no change in phase of TiO_2 sample. TiO_2/SiO_2 multilayers are also deposited by the same method and with same parameters as single layers are deposited. Hence multilayered samples are also amorphous and amorphous phase has highest damage threshold among all existing phases of TiO_2.[13]

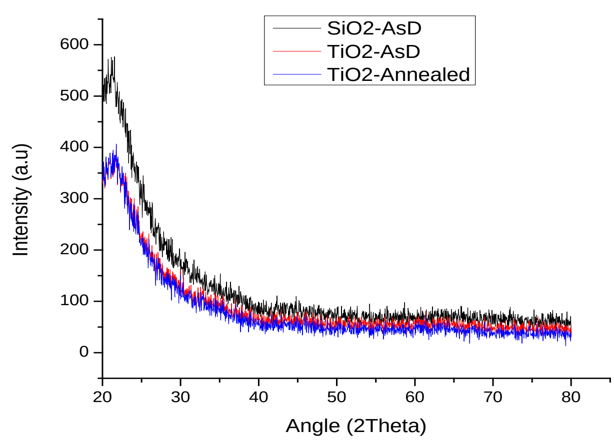


Figure 1. X-ray diffraction pattern of all samples.

Transmission of SiO_2 thin films is measured by carry 50 spectrophotometer and the transmission spectra are shown ii figure 2. Transmission of bare BK7 is about 91% in IR and visible region and it decreases rapidly in near ultraviolet region. When SiO_2 single quarter wave layer (1QWOT) and SiO_2 odd multiple quarter wave layer (3QWOT) are deposited on BK7 substrate then transmission is increased. There is a large transmission band from IR to visible region and transmission decreases rapidly in ultra-violet region for BK7 glass.[14] Maximum reflection of TiO_2 single layer is approximately 27.61% and it increases as the number of layers increases and it is 72.91% for $HL^{\wedge 2}H$ multilayer and after annealing reflection slightly decreases as shown in the figure 2(b) and refractive index increases and extinction coefficient decreases, hence absorption decreases.[15,16]

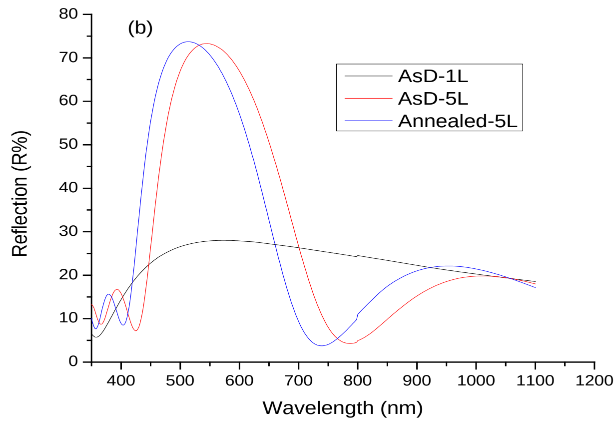
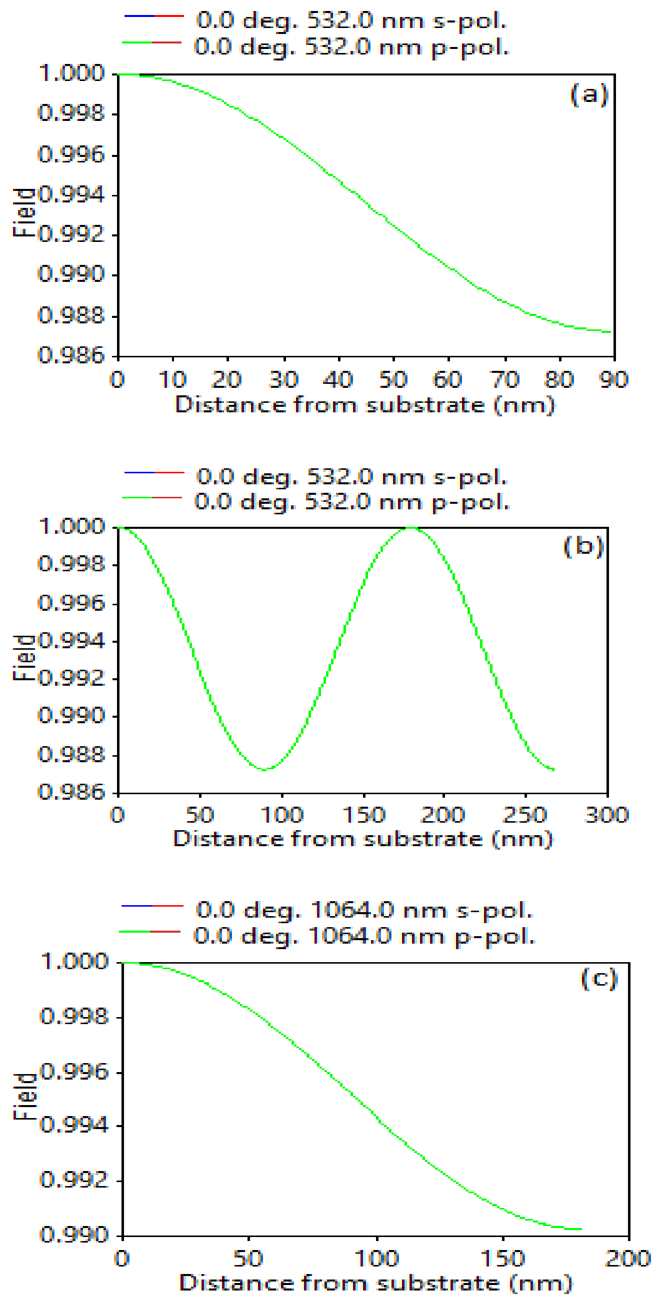


Figure 2 (a) Transmission spectra of bare BK7, single and multiple quarter wave SiO_2 thin film and 2(b) reflection spectra of multilayers (as deposited and annealed) samples.

ELECTRIC FIELD AND LASER DAMAGE THRESHOLD

Electric field values of SiO_2 and TiO_2/SiO_2 multilayer samples are determined by open filter software for both s and p polarizations as shown is figure 3(a) to 3(f). Electric field on the incident surface at 532nm wavelength is found to be 0.988 units for 1QWOT and 3QWOT thicknesses and at1064nm it is 0.990 units i.e. some increment in its value for both the thicknesses. Electric field achieves maximum value inside 3QWOT thickness films at 532nm and 1064nm but these maximum fields have negligible effect on laser induced damage threshold value because of the bulk SiO_2 like behavior of the thin films.[17,18,26] Electric field has negligible effect on LIDT of SiO_2 film as have higher value for 1064nm than that for 532nm as shown in figure 3 and Table1 but LIDT exhibits reverse order. In multilayer samples the electric field intensity has low value at incidence surface in comparison to single layer SiO_2 samples but due to interface effect in multilayers, electric field value at first interface is more than that at incidence surface and in multilayer TiO_2/SiO_2 electric field at first interface is very high values of 2.523 units at 532nm and 2.298 units for 1064nm wavelength and this high value affects the LIDT of multilayers as shown in Table 1. LIDT of SiO_2 thin film in nano second regime is measured with Nd: YAG laser having maximum energy of 1J at 1064nm wavelength and 500mJ at 532nm wavelength and is explained on the basis of thermal mechanism.[19] Defects and inclusion present in the films are responsible for damage of film. The absorption index of defects is high, so defects in the film absorb energy from laser radiation and transfer this energy to lattice till the macroscopic damage is observed on the thin film [20]. Thicknesses of film has little effect on the LIDT of SiO_2 film as shown in Table1, on the other hand wavelength of laser has more effect on the LIDT of silica film.[21] For multilayered thin film, damage threshold is explained by absorption of radiation by defects and electric field on first interface, LIDT of TiO_2/SiO_2 multilayer is comparatively lower than that for SiO_2 single layer as the electric field intensity at interface is very high which reduces the damage threshold.[22,23] In multilayer LIDT at 1064nm is higher than that at 532nm wavelength which is due to comparatively lower value of electric field at interface and lower value of energy per photon incident in 1064nm light[19]. LIDT of multilayer is further increased by annealing of the samples, annealing does not affect the electric field value but reduces the defect density in the coating[19] and reduces the absorption of the samples as shown in Table 1 and it increases the damage threshold. The high value of LIDT of SiO_2 may be understood as refractive index difference in BK7 substrate and SiO_2 film is very small. So the adhesion and nucleation of film is better and forms better stoichiometry of film. Due to this SiO_2 thin film behaves like bulk SiO_2[24] and has higher damage threshold at both the thicknesses than multilayer samples in both as deposited and annealed samples, at both wavelengths.[1,25]



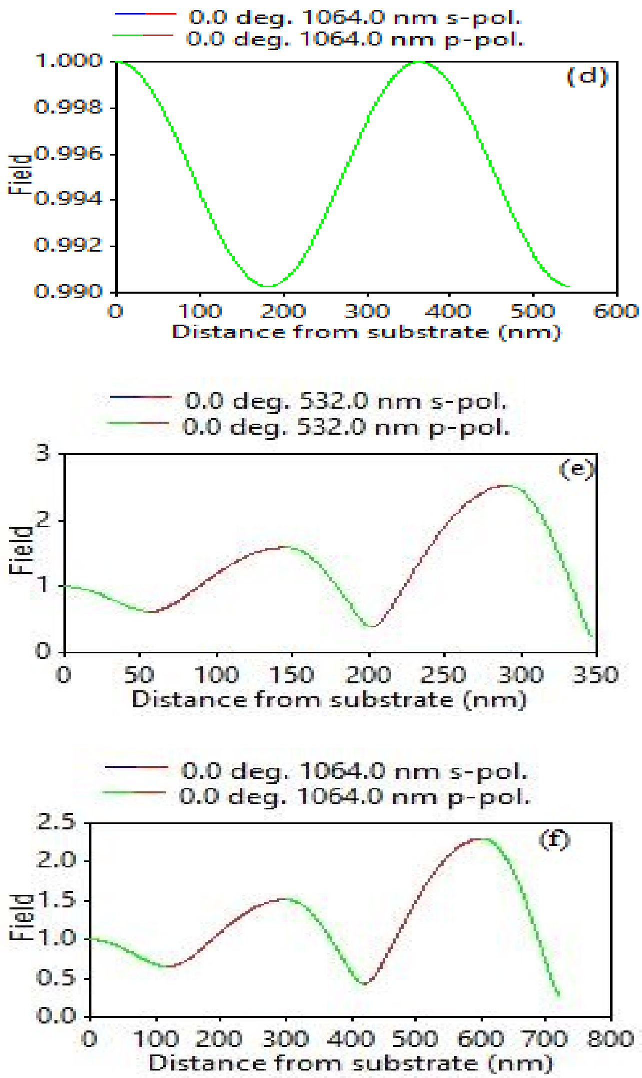
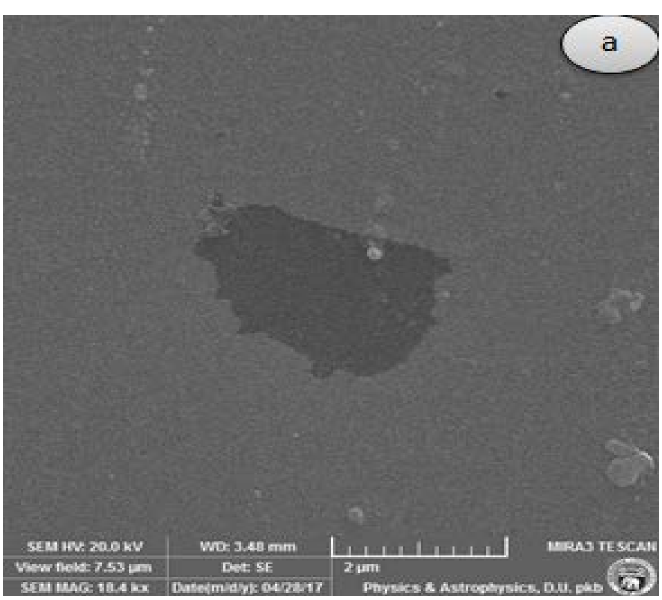


Figure 3. Electric field of single quarter wave thickness of SiO_2 at 532nm and 1064nm 3(a)-(b) and 3(c) -(d) three quarter wave thickness of SiO_2 at 532nm and 1064nm and TiO_2/SiO_2 multilayers at 532nm and 1064nm in 3(e)- 3(f) respectively.



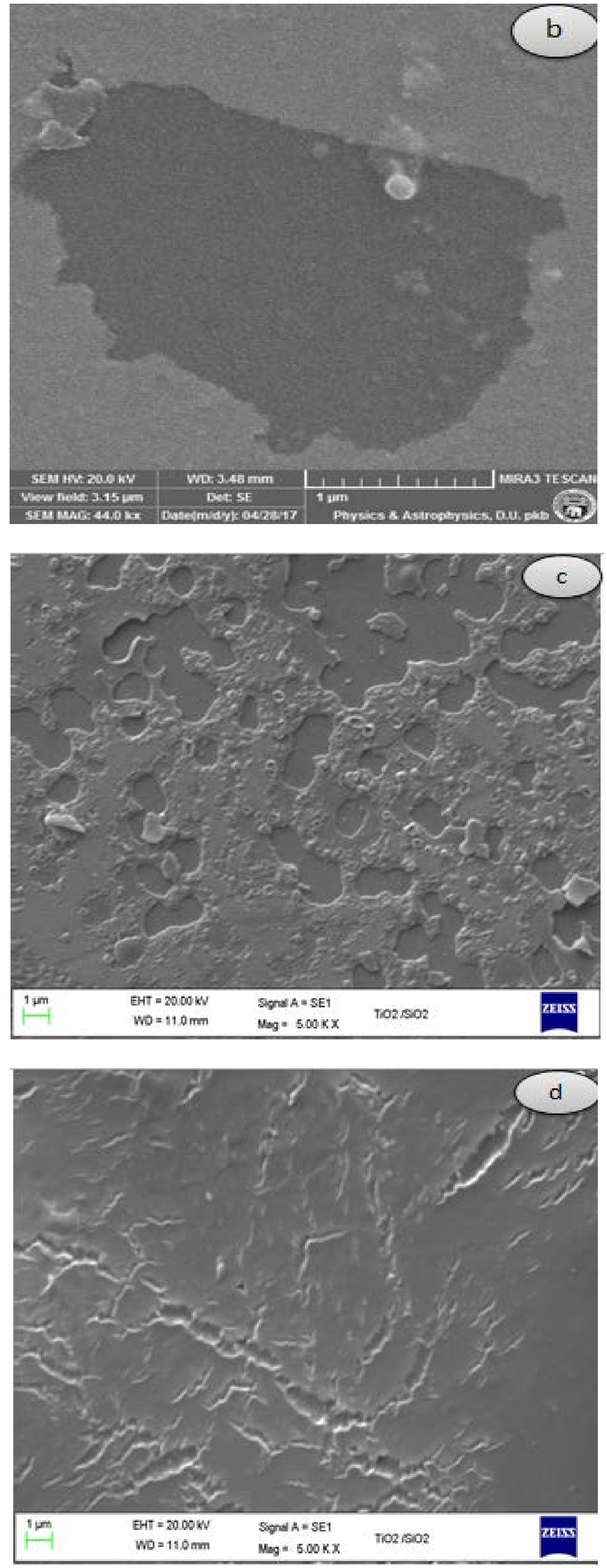


Figure 4. Damage morphology of SiO_2 thin film at two different magnification 4(a), (b) and TiO_2/SiO_2 multilayer for as deposited and annealed samples in 4(c), 4(d).

Table.1. LIDT and other parameters of SiO_2 and TiO_2/SiO_2 samples

Sample	Transmission (%)		Electric field (a.u)		LIDT(J/cm^2)	
	@532nm	@ 1064nm	@532nm	@ 1064nm	@532nm	@ 1064nm
1QWOT	92.00	93.00	0.988	0.991	7.96	14.46
3QWOT	92.00	93.00	0.988	0.991	9.09	15.50
HL^2H as deposited	24.92	26.94	2.523	2.298	1.43	1.51
HL^2H Annealed	25.45	27.63	2.523	2.298	2.73	3.34

DAMAGE MORPHOLOGY

Damage morphology of SiO_2 thin film is shown in the figures 4(a)-4(b). Laser incident on the film removes the film completely. Damage morphology of multilayer TiO_2/SiO_2 (as deposited and annealed samples) is shown in figure 4(c)-4(d). The morphology shows that damage is initiated by very small defects, but the precursor size does not have direct link to the size of the damage pits.[26]

Indeed, the pit diameter is linked in a complex way to the size of the laser damage precursor and it also depends strongly on the thermal and mechanical properties, residual stresses and adhesion of the film.[27] It is observed that the pit diameter in silica thin films can reach ten times the damage initiator diameter [26-28]. In multilayer thin film, at the bottom of the pits, no damage precursors are visible, invisible absorbing centres were the probable causes of damage in TiO_2/SiO_2 multilayer coating. In the HR coating, the absorbing centres in these layers, especially at interface, are more probable to induce laser damage. The material surrounding the absorber can be melted or fractured off by the laser irradiation on these absorbing centres.[30,31]

CONCLUSIONS

Laser induced damage threshold (LIDT) of SiO_2 thin film having single quarter wave optical thickness, three quarter wave optical thickness and TiO_2/SiO_2 multilayers on BK7 substrates is investigated. Nd: YAG laser which is operated on two wavelengths 532nm and 1064nm is used for measurement. There is a little effect of film thickness and negligible effect of electric field value on the LIDT in SiO_2 layer samples, but a significant influence of wavelength is observed. In multilayer samples electric field has more effect on LIDT, due to interface effect; LIDT is very small for multilayer as compared to SiO_2 samples. LIDT of multilayer is further increased, approximately doubled on annealing the samples. LIDT of SiO_2 samples has higher value because there is very small refractive index difference between film and substrate, so adhesion and stoichiometry of film is better which enhances the damage threshold. LIDT of samples at 1064nm is greater than that at 532nm which is due to the fact that energy deposited per photon at 1064 is lower than that at 532nm. Hence LIDT is higher in IR region than visible region.

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