

Influence of Thickness, Substrate and Wavelength on Laser Damage Threshold of SiO₂ Thin Films

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Abstract: Laser damage threshold of optics is key point from the time of invention of laser. In this study the laser induced damage threshold (LIDT) of SiO₂ material which is used in making high reflective (HR) coating and antireflective (AR) coating, is explored on the **basis** of thickness of film, substrate on which film is formed and on two different laser wavelengths which lies in Infrared IR and visible region. Samples are deposited by conventional e-beam deposition method having $\lambda/4$ and $3\lambda/4$ thickness, on BK-7 and on fused silica. LIDT is measured in nano-second regime with Nd: YAG laser at 532nm and 1064nm, damage morphology is studied by scanning electron micrograph (SEM) and suitable damage mechanism is found out. Optical properties of the films are investigated by spectrophotometer and ellipsometry.

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

1. 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 (LIDT)** of optical components¹⁻³. 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 metal or metal oxide used for preparation of dielectric 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 method⁵⁻⁷. 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, multi-photon ionization is main mechanism⁹⁻¹¹.

In this study, the laser induced damage threshold (LIDT) of SiO₂ thin film having odd multiples of quarter wave thickness on two different substrates (BK-7 and FS) are studied with two laser wavelengths 1064nm and its second harmonics 532nm and effect of change in parameters on LIDT is explored.

2. Experimental Procedure

Samples of SiO₂ single quarter wave optical thickness (QWOT) and odd multiple of quarter wave optical thickness (3 QWOT) are prepared in Box type coating plant (Model BC-600 Hind High Vacuum). Thicknesses of samples are monitored by quartz crystal monitor. Basic thickness of samples are calculated by using formula

$$(2.1) \quad d = \frac{m\lambda}{4n}$$

where 'd' is thickness of thin film, ' λ ' reference wavelength, 'n' refractive index of film material and 'm' is integer. A thick layer of sample material is deposited on BK-7 or fused silica substrate then measure transmission spectra and note wavelength corresponding to minimum three peaks or valises in the transmission spectra in high reflective or antireflective coating. Then by using formulae given below, calculate the given peaks or valises correspond to how much $\lambda/4$ thickness of the given sample material.

$$(2.2) \quad K_2 = \frac{\lambda_2}{\lambda_2 - \lambda_1}$$

$$(2.3) \quad K_3 = \frac{2\lambda_1}{\lambda_2 - \lambda_1}$$

where K_2 and K_3 are multiple of $\lambda/4$ i.e., $3\lambda/4$, $5\lambda/4$ so on and λ_1 , λ_2 , λ_3 are wavelength of first, second, third peaks or valises respectively in reflection or transmission spectra. Consider a thin film sample having thickness 'X'. Let 'Y' be the wavelength corresponds to $5\lambda/4$ thickness of sample.

So 'Y' wavelength is equal to $5\lambda/4$ at 'X' thickness of the sample 1 wavelength is equal to $5\lambda/4$ at $\frac{X}{Y}$ thickness and 532 wavelength is equal to

$5\lambda/4$ at $\frac{X}{Y} \times 532$ unit thickness, Let this is equal to some factor 'Z' thickness.

So 'Z' thickness is equal to $5\lambda/4$ at 532 unit.

Hence $\lambda/4$ thickness at 532 is equal to $\frac{Z}{5}$ unit thickness. Using these above formulae one can calibrate quartz crystal monitor thickness.

Now chamber is loaded, a pressure of 5×10^{-2} mbar is achieved using rotary pump and roughing valve; simultaneously the diffusion pump started. After running plant 20 minutes chamber pressure is achieved as said above order then close roughing, open backing and high vacuum valve; obtain pressure order of 5×10^{-5} mbar. Substrate in the chamber is kept at 220°C with the help of gradient heater. Supply argon gas and 10 minutes ion beam cleaning is applied after this pressure. Finally close the argon gas and pump the chamber by diffusion pump till the pressure reach 2×10^{-5} mbar. Now chamber is ready for coating, apply oxygen gas and maintain pressure 3×10^{-4} mbar. Switch on e-beam and monitor beam, kept it in the middle of the crucible and increase DC current till 40-70 Ampere. SiO_2 sublimate, control the rate of evaporation 3-5Å and continue evaporation till the required

thickness is achieved. Completing this process, cooling of chamber is must, if we bring hot sample out then there is a probability of embedding the impurity from environment.

3. Results and discussion

3.1 Optical Properties

Transmission of SiO_2 thin films are measured by carry50 spectrophotometer and shown in figure 1. Transmission of bare BK-7 is about 91% in IR and visible region and decrease rapidly in near ultraviolet region. When SiO_2 single quarter wave layer (1QWOT) and SiO_2 odd multiple quarter wave layer (3QWOT) is deposited on BK-7 substrate then transmission is increased. In case of fused silica (FS) bare substrate have transmission 92% and it also increased as single and multiple quarter wave thickness deposited. There is a large transmission band of fused silica from IR to UV region. Hence transmission band of fused silica is larger than BK-7 glass¹².

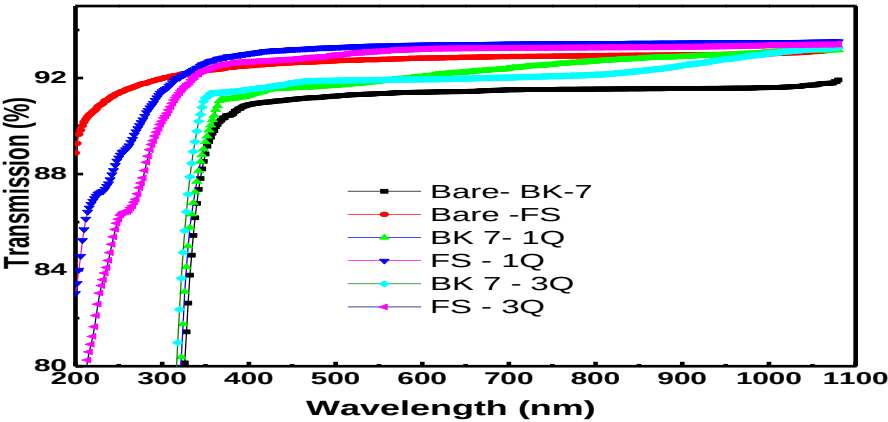


Figure 1: Transmission spectra of bare BK-7, Fused silica, single and three quarter wave SiO_2 thin film

3.2. Electric field and Laser Damage Threshold

Electric field values of SiO_2 samples are measured by open filter software for both s and p polarizations as shown is figures 2(a) to 2 (d). Electric field on the incident surface at 532nm wavelength is 0.988 units for 1QWOT and 3QWOT thicknesses and at1064nm is 0.99 units i.e. some increment in its value for both 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

because of the bulk SiO_2 like behavior of the thin films^{13,14,18}. Electric field has negligible effect on LIDT of SiO_2 film as have higher value for 1064nm than 532nm as shown in figures 2(a), 2(c) and table 1 but LIDT have reverse order. 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 explained on the basis of thermal mechanism. Defects and inclusion present in the film 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 shown on the thin film¹⁶. Thicknesses of film have little effect on the LIDT of SiO_2 film on both substrates as shown in table 1, on the other hand wavelength of laser and substrate have more effect on the LIDT of silica film¹⁵. Refractive index difference in fused silica substrate and SiO_2 film is very small compare to refractive index difference in BK-7 and SiO_2 film. So, the adhesion and nucleation of film better and form more better stoichiometry of film in fused silica than BK-7 substrate. Due to this bulk like behavior of fused silica/ SiO_2 film have higher damage threshold at both thicknesses as compare to BK-7/ SiO_2 film^{1,17}.

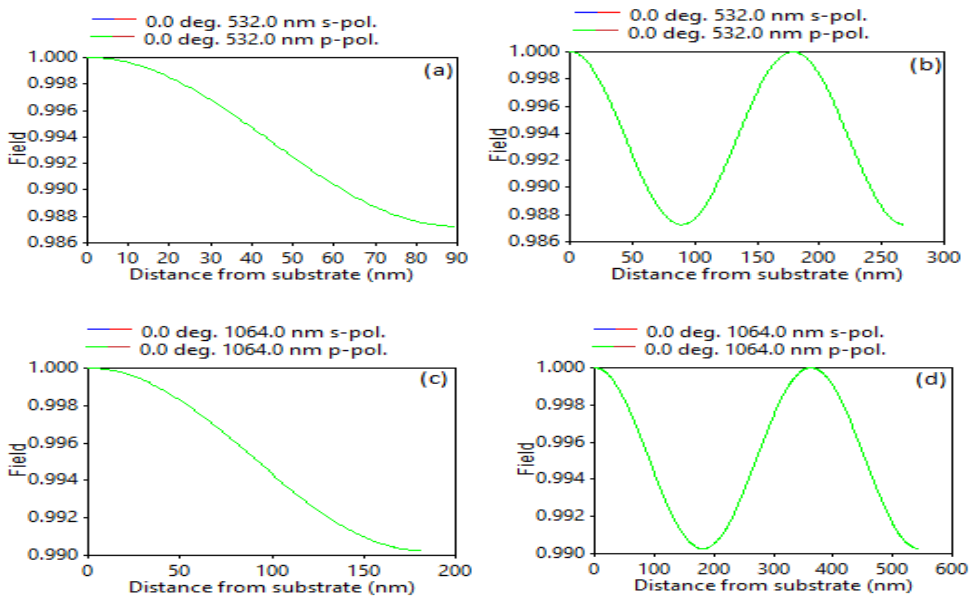


Figure 2: 2(a) Electric field pattern of single quarter wave thick layer of SiO_2 at 532 nm wavelength, 2(b) Electric field patterns of three quarter wave thick layer of SiO_2 at 532 nm wavelength, 2(c) Electric field pattern of single quarter wave thick layer of SiO_2 at 1064 nm wavelength and 2(d) Electric field pattern of three quarter wave thick layer of SiO_2 at 1064 nm wavelength.

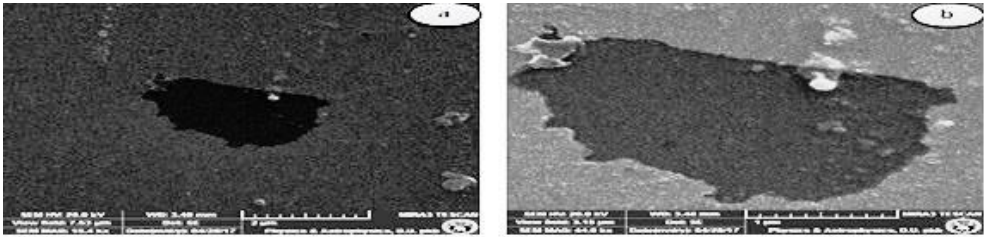


Figure 3: Damage morphology of SiO₂ thin film

3.3 Damage Morphology

Damage morphology of SiO₂ thin film is shown in the figures 3(a) and 3(b), laser incident on the film remove the film completely. The morphology indicates that damage is initiated by very small defects, but the precursor size does not have direct link to the size of the damage pits. Indeed, the pit diameter is linked in a complex way to the size of the laser damage precursor and also depends strongly on the thermal and mechanical properties, residual stresses and adhesion of the film. It is observed that the pit diameter in silica thin films can reach ten times the initiator diameter¹⁸⁻²⁰.

Table 1: LIDT and other parameters of SiO₂ samples.

Samples	Transmission	Electric	LIDT(J/cm ²)	LIDT(J/cm ²)
	(%)	field(a.u.)	BK-7	Fused Silica
1QWOT at 532	92	0.988	7.96	10.84
3QWOT at 532	92	0.988	9.09	12.16
1QWOT at 1064	93	0.991	10.90	14.46
3QWOT at 1064	93	0.991	12.72	15.50

4. Conclusion

Laser induced damage threshold (LIDT) of SiO₂thin film having single quarter wave optical thickness and three quarter wave optical thickness, on two different substrates BK-7 and fused silica are investigated. Nd: YAG laser which is operated on twowavelengths 532nm and 1064nm are used for measurement. There is a little effect of film thickness and negligible effect of electric field value on the LIDT, but a significant influence of wavelength and substrate is observed. LIDT of samples with BK-7 substrate have lower value than fused silica samples because there is very small refractive index

difference between SiO₂ and fused silica, so adhesion and stoichiometry of film is better in fused silica than BK-7 which have comparative greater index difference. LIDT of samples at 1064nm is greater than 532nm which is due to energy deposited per photon at 1064 nm is lower than 532nm. Hence LIDT is higher in IR region than visible region.

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References

1. L. Gallais, H. Krol, J. Capoulade, M. Cathelinaud, L. Roussel, G. Albrand, J. Natoli, M. Commandre, M. Lequime and C. Amra, Comparative study of IR and UV laser damage resistance of silica thin films deposited by Electron Beam deposition, Ion Plating, Ion Assisted Deposition and Dual Ion Beam Sputtering, *Proc. of SPIE*, **5963** (2005) 59630Z.
2. C. Carr, H. Radousky, A. Rubenchik, M. Feit and S. Demos, Localized Dynamics during Laser-Induced Damage in Optical Materials, *Phys Rev Lett.*, **92(8)** (2004).
3. K. Mikami, S. Motokoshi, T. Somekawa, T. Jitsuno, M. Fujita, and K. Tanaka, Temperature Dependence of Laser-Induced Damage Thresholds by Short Pulse Laser, *Proc. of SPIE*, **8530** (2012) 853005.
4. H. Krol, L. Gallais, M. Commandre, C. Besset, D. Torricini, G. Lagier, Influence of polishing and cleaning on the laser-induced damage threshold of substrates and coatings at 1064 nm, *Opt. Engg.*, **46(2)** (2007) 023402.
5. L. Gallais, J. Capoulade, J. Natoli, M. Commandre, M. Cathelinaud, C. Koc, M. Lequime, Laser damage of Silica and Hafnia thin films made with different deposition technologies, *Proc. of SPIE*, **6720** (2007) 67200S.
6. L. Gallais, H. Krol, J. Natoli, M. Commandre, M. Cathelinaud, L. Roussel, M. Lequime, C. Amra, Laser damage resistance of silica thin films deposited by Electron Beam Deposition, Ion Assisted Deposition, Reactive Low Voltage Ion Plating and Dual Ion Beam Sputtering, *Thin Solid Films*, **515** (2005) 3830–3836.
7. D. Milam, W. Lowdermilk, F. Rainer, J. Swain, C. Carniglia, and T. Hart, Influence of deposition parameters on laser-damage threshold of silica-tantala AR coatings, *Appl. Opt.*, **21** (1982) 3689.
8. M. Alvisi, G. Nunzio, M. Ferrara, M. Perrone, A. Rizzo, S. Scaglione and L. Vasanelli, Effects of substrate temperature on the laser damage threshold of sputtered SiO₂ films, *J. Vac. Sci. Technol. A*, **16(6)** (1998).

9. M. Meroa, J. Liua, A. Sabbaha, J. Jasaparab, K. Starkec, D. Ristauc, J. Mcivera and W. Rudolph, Femtosecond pulse damage and pre-damage behavior of dielectric thin films, *Proc. of SPIE*, (2003) 4932.
10. M. Turowski, M. Jupe, L. Jensen, and D. Ristau, Laser-induced damage and nonlinear absorption of ultra-short laser pulses in the bulk of fused silica, *Proc. of SPIE*, **7504** (2009) 75040H.
11. L. Gallais, J. Capoulade, J. Natoli, and M. Commandre, Investigation of nano defect properties in optical coatings by coupling measured and simulated laser damage statistics, *Jour. of Appl. Phys.*, **104** (2008) 053120.
12. M. Alvisi, G. Nunzio, M. Giulio, M. Ferrara, M. Perrone, L. Protopapa and L. Vasaneli, Deposition of SiO₂ films with high laser damage thresholds by ion-assisted electron-beam evaporation, *Appl. Opt.*, **38 (7)** (1999).
13. M. Protopapa, M. Alvisi, F. Tomasi, M. Giulio, M. Perrone, and S. Scaglione, Influence of standing-wave electric field pattern on the laser damage resistance of HfO₂ thin films, *J. Vac. Sci. Tech. A*, **20(3)** (2002).
14. B. Newnam, D. Gill and G. Faulkner, *Influence of standing-wave fields on the laser damage resistance of dielectric films*, Los Alamos Scientific Lab., N. Mex, USA.
15. W. Tao and Y. Lihong, Research on SiO₂ film laser damage threshold, *Proc. Of SPIE*, **8416** 84162D.
16. S. Papernova and A. Schmid, Correlations between embedded single gold nanoparticles in SiO₂ thin film and nanoscale crater formation induced by pulsed-laser radiation, *J. of Appl. Phys.*, **92(10)** (2002).
17. K. Yoshida, T. Yabe, H. Yoshida, and C. Yamanaka, Mechanism of damage formation in antireflection coatings, *J. Appl. Phys.*, **60(4)** (1986).
18. L. Gallais, J. Capoulade, F. Wagner, J. Natoli, M. Commandre, Analysis of material modifications induced during laser damage in SiO₂ thin films, *Optics Commu.*, **272** (2007).
19. L. Gallais, J. Capoulade, J. Natoli and M. Commandre, Investigation of nano defect properties in optical coatings by coupling measured and simulated laser damage statistics, *J. Appl. Phys.*, **104** (2008) 053120.
20. S. Papernova and A. Schmid, Two mechanisms of crater formation in ultraviolet-pulsed-laser irradiated SiO₂ thin films with artificial defects, *J. of Appl. Phys.*, **97** (2005) 114906.