

# Influence of substrate surface properties on laser-induced damage properties of TiO<sub>2</sub> thin films

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## **ABSTRACT**

Various investigations have focused on the laser-induced damage of thin films. However, substrate surface properties have not been taken into account. TiO<sub>2</sub> thin films with a one-quarter wavelength optical thickness were deposited by electron beam evaporation on two types of substrates with different surface properties. The surface morphologies of TiO<sub>2</sub> thin films were captured by atomic force microscopy, and the laser-induced damage threshold of TiO<sub>2</sub> thin films was measured. The relationship between the substrate surface properties and the laser-induced damage properties of TiO<sub>2</sub> thin films was observed and analyzed.

## **1. INTRODUCTION**

Optical coatings are important components in a laser system that is easily damaged, especially under conditions of high laser power [1]. Increasing attention has been given to the study of the fundamental mechanisms of damage initiation and growth [2]. At present, research on laser-induced damage of optical thin films proceeds along two major directions. One direction concerns the characteristics of the laser. The laser-induced damage threshold (LIDT) always increases with increasing wavelength both in the 1-on-1 testing regime and in the n-on-1 testing regime [3-5]. In addition, numerous studies have shown that the beam size of the laser [6] and the pulse width of the laser beam are both important factors that affect the LIDT. The other research direction involves the characteristics of the optical film itself, such as the film material, film design, and optical absorption [7-10]. Film materials with a high refractive index lead to a high film LIDT, and an SiO<sub>2</sub> interfacial layer with a 1/4 optical thickness (1 QWOT) has been shown to be effective and flexible for increasing the LIDT of dielectric anti-reflective (AR) coatings [11]. Meanwhile, a number of researchers have reported that the optical properties, micro-defects, and the electric field distributions of optical films are also critical influences on their LIDT [12-14].

However, focusing a preponderance of attention on the laser and the optical film has apparently caused these previous studies to neglect the important role played by the surface properties of the substrate, even if the surface properties of the optical film has been considered when the defects, such as contaminants, impurities, and nodules, of the optical film have been regarded as one of the main causes for the films failure under the laser

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irradiation [14, 15]. Therefore, the nature of these present studies motivates an investigation of the influence of the surface properties of the substrate on the laser-induced damage properties of thin films. The aim of the present research was to focus on the influences of the substrate surface properties, and investigate differences in substrate surface properties and the influence of these differences on the laser-induced damage of optical thin films.

## 2. EXPERIMENTAL DETAILS

### 2.1. Test Samples

To analyze the influence of the substrate surface properties on the laser-induced damage properties of thin films, we selected two different types of the BaK 7 substrates with different surface properties, and four samples were prepared for each type. First, we compared these two types of substrates using a Zygo interferometer ( $\lambda = 632.8$  nm) which is shown in Table 1. The average of peak-to-valley (PV) value of substrate 1 is about two times higher than that of substrate 2, and the average root mean square (RMS) surface roughness of substrate 1 is about 7 times higher than that of substrate 2.

Table 1: Comparison of Two Types of Substrates ( $\lambda = 632.8$  nm)

Sample number	Substrate 1		Substrate 2	
	PV( $\lambda$ )	RMS( $\lambda$ )	PV( $\lambda$ )	RMS( $\lambda$ )
1	0.414	0.060	0.228	0.012
2	0.278	0.045	0.166	0.010
3	0.568	0.142	0.138	0.010
4	0.309	0.052	0.188	0.013
Average	0.392	0.074	0.180	0.011

TiO<sub>2</sub> films were deposited on all substrate samples using electron beam evaporation. Each substrate was first cleaned with the mixture of alcohol and ether. The working pressure in the vacuum chamber was  $9 \times 10^{-6}$  mbar. The deposition temperature was 25 °C at room temperature. The optical film thickness, which was approximately  $1/4 \lambda$  ( $\lambda = 1064$  nm), was controlled using an optical monitor.

### 2.2. Experimental Apparatus

The apparatus used for laser-induced damage testing is illustrated in Fig. 1. The setup involves an Nd:YAG laser with a 1064 nm wavelength and 10 ns pulse duration, which was focused to provide a far-field circular Gaussian beam with a diameter of 0.8 mm. The incident beam energy was measured using an energy meter. The laser-induced damage properties were examined in the 1-on-1 regime according to the international standard ISO 11254 [16]. Using a CCD camera connected to a computer, an image of the irradiated zone was acquired before and after each pulse. Then, after image processing, the area of the damaged zone was evaluated in terms of the number of pixels, and the damage criterion was defined as 5 pixels.

## 3. RESULTS AND ANALYSIS

### 3.1. Optical Properties of Thin Films

The transmittance spectra of the thin films deposited on the two types of substrates are shown in Fig. 2, where the transmittance of the thin film deposited on substrate 1 is higher than that deposited on substrate 2 at a wavelength of 1064 nm. The transmittance depends only on the

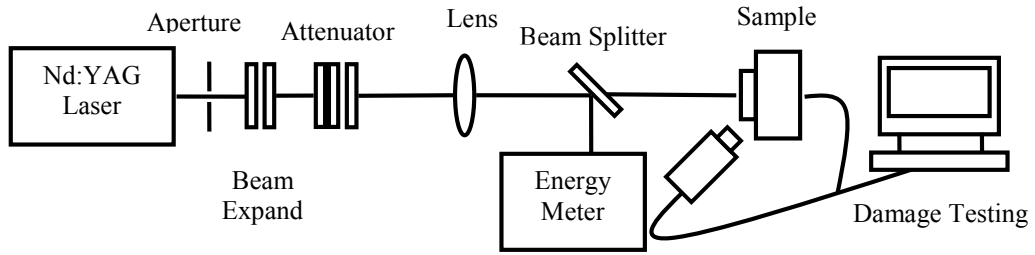


Fig.1 Experimental apparatus of the laser-induced damage threshold testing system

refractive index, thin-film thickness, and the wavelength. When a laser propagates through the sample, a portion of the incident light penetrates into the film, a component of which penetrates into the substrate, and the remainder of the light beam penetrates into the air. According to the Fresnel principle, the transmittance of the film can be calculated by

$$T = \frac{(n_{\text{air}} - n_{\text{sub}})^2 \cos^2 \delta + (n_{\text{air}} n_{\text{sub}} / n_{\text{film}} - n_{\text{film}})^2 \sin^2 \delta}{(n_{\text{air}} + n_{\text{sub}})^2 \cos^2 \delta + (n_{\text{air}} n_{\text{sub}} / n_{\text{film}} + n_{\text{film}})^2 \sin^2 \delta} \quad (1)$$

where,

$$\delta = \frac{2\pi}{\lambda} n_{\text{film}} d_{\text{film}}$$

$n_{\text{air}}$ ,  $n_{\text{film}}$ , and  $n_{\text{sub}}$  represent the refractive index of the air, the film, and the substrate, respectively. The RMS surface roughness of the thin films deposited on the two different types of substrates are nearly equivalent, as shown in Fig. 3, which illustrates that the type of substrate has little effect on the light scattering of the  $\text{TiO}_2$  films. In our work, we assume that the overall thin-film thickness on a sample is uniform, so that the refractive index of the film is the only factor that affects the transmittance of the overall sample. Fig. 2 indicates that the transmittance of the substrate 2 is lower than that of the substrate 1 for the two transmittance minima, indicating that the refractive index of the thin film on substrate 2 has increased. The results indicate that the film deposited on the substrate with a lower PV value has a higher packing density, which is consistent with previous findings [17]. On the other hand, the surface properties of the substrate have a certain influence on the packing density of the film, and lower substrate PV and RMS surface roughness values result in a thin film of a higher packing density.

### 3.2. Laser-induced Damage Properties of Thin Films

To distinguish the extent of laser-induced damage to the thin films deposited on the two different substrate types, the two samples were irradiated using an equivalent laser energy of 140 mJ at 1064 nm, and the damage sustained by the  $\text{TiO}_2$  films on the two different substrate types were captured by a ECLIPSE L150 Nikon optical microscope at the same magnification, as shown in Fig. 4. The damaged region shown in Fig. 4(a) is obviously larger than that shown in Fig. 4(b). It is clear from these results that the thin film deposited on substrate 2 is less susceptible to laser-induced damage.

The LIDT is an important index for evaluating the laser-induced damage properties of a thin film. In our work, 10 gradient laser energy levels and 10 tested regions at each laser

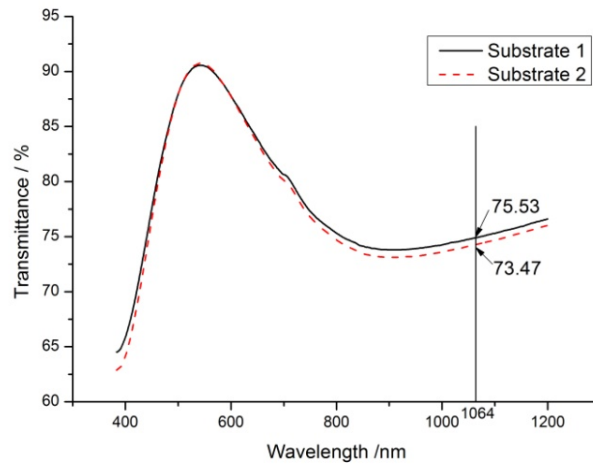
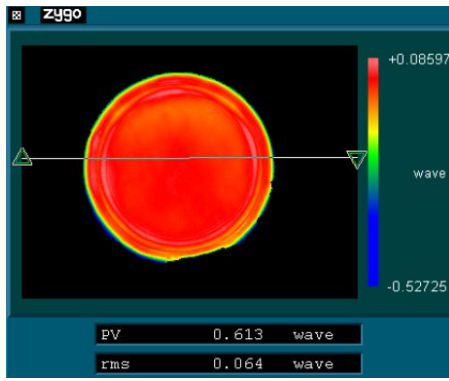
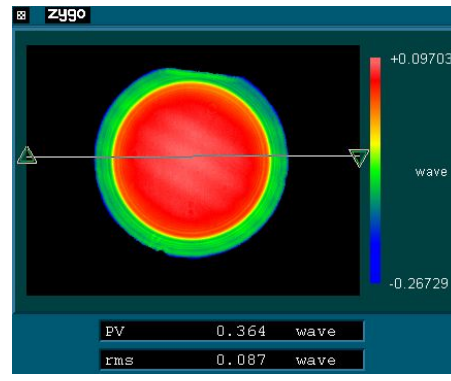


Fig. 2 Comparison of the optical transmittance of the different substrate samples

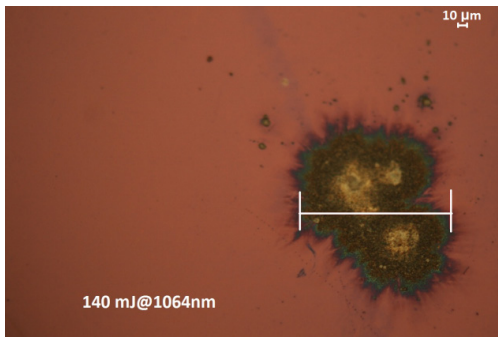


(a)

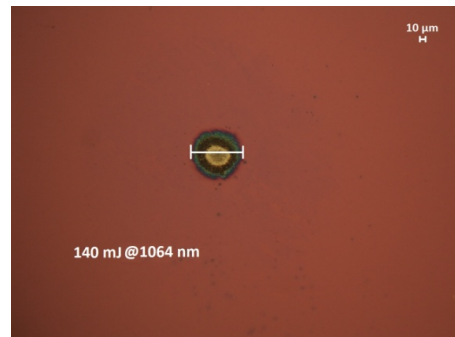


(b)

Fig.3 Surface properties of the TiO<sub>2</sub> films deposited on substrate 1 (a) and substrate 2 (b)



(a)



(b)

Fig.4 Damage of the TiO<sub>2</sub> films deposited on substrate 1 (a) and substrate 2 (b)

energy level were applied to each sample. The laser-induced damage probabilities of various laser energies are plotted in Fig. 5. The straight line is fitted according to the rule of zero-probability damage. In Fig. 5, the blue square points and blue line represent the LIDT of the TiO<sub>2</sub> film deposited on substrate 1, and the red triangular points and red line represent that of the TiO<sub>2</sub> film deposited on substrate 2. The slope of the blue line is clearly larger than that of the red line. The intersection points of the blue and red lines with the abscissa, marked as point B and point A in Fig. 5, indicate the points of zero laser damage probabilities for the thin films deposited on substrate 1 and substrate 2 respectively. The laser damage energy at a zero laser damage probability for the TiO<sub>2</sub> film on substrate 2 is 106.60 mJ, and that for the TiO<sub>2</sub> film on substrate 1 is 86.39 mJ. According to the rule of zero laser damage, the laser damage threshold is given as follows:

$$\text{LIDT} = \frac{\text{Laser damage energy at zero probability}}{\text{Laser spot area}} (\text{J/cm}^2) \quad (2)$$

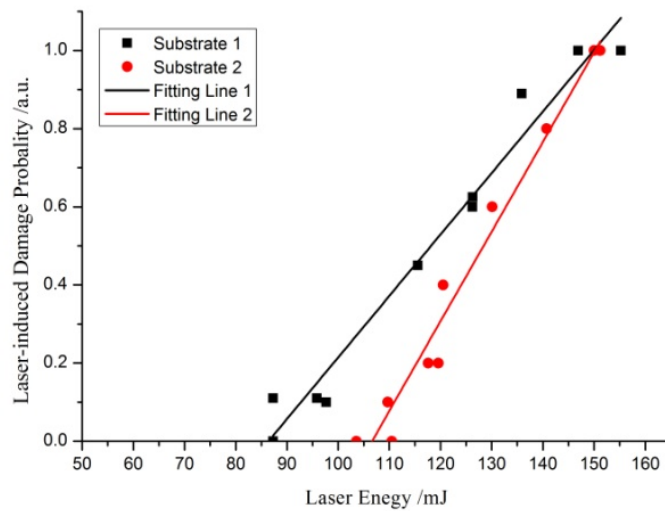


Fig.5 Calculation of the laser-induced damage thresholds of the samples

From this equation, we obtained the laser damage threshold of these samples, that is 17.20 J/cm<sup>2</sup> for the TiO<sub>2</sub> film on substrate 1, and 21.22 J/cm<sup>2</sup> for the TiO<sub>2</sub> film on substrate 2. The laser damage threshold for an equivalent thin film on substrate 1 is lower than that on substrate 2. It indicates that films deposited on substrates with different substrate surface properties have different laser damage thresholds, and that substrate surfaces with low PV and RMS surface roughness values result in a higher LIDT.

### 3.3. Electric Field Intensity Distribution of Thin Films

When the incident laser transmits through a thin film, a standing wave must occur along the direction of the incident light in the film and substrate. It has been well established that the

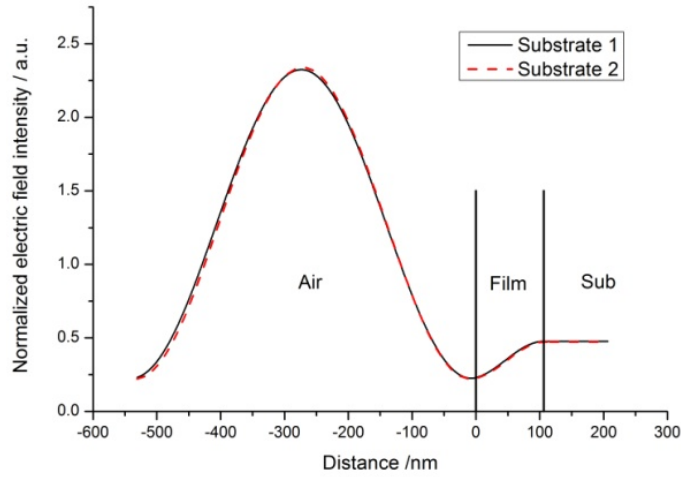
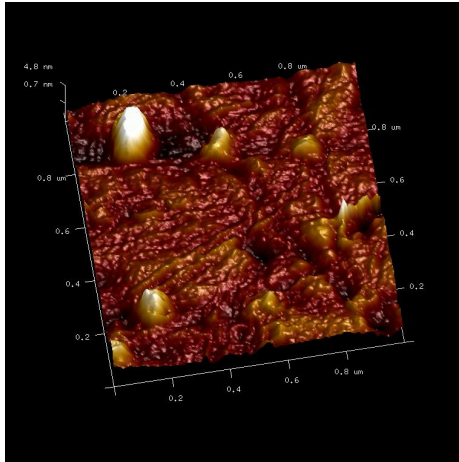
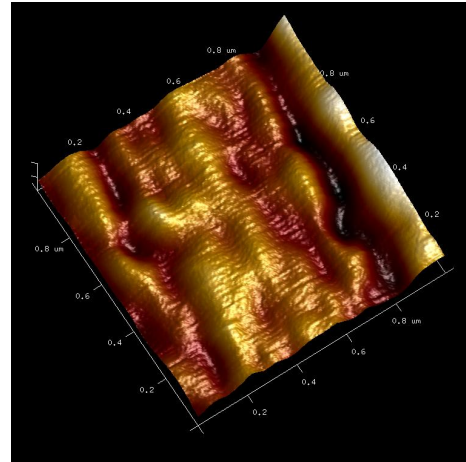


Fig.6 Electric field intensity distribution of the different samples



(a)



(b)

Fig.7 Surface morphologies of the films deposited on substrate 1 (a) and substrate 2 (b) by AFM

film-air interface is the location where atomic bond breakages first occur [18], and a film with a higher electric field at the film-air interface is most likely to be damaged [19]. Therefore, the thickness of the thin film significantly affects the laser-induced damage of a thin film. To accurately measure the thickness of a thin film, two special TiO<sub>2</sub> thin-film samples were deposited both on substrate 1 and substrate 2. The diameter of each TiO<sub>2</sub> film is only 10 mm, and the edges of the films are both very sharp. Considering the inaccuracy of the measurement of thin-film thickness by the ZYGO interferometer, we tested each sample twice (one is the substrate surface, and the other is the film surface), and data processing was performed to obtain an accurate thin-film thickness using MATLAB software. The main idea of data

processing is to subtract the surface properties of the substrate from those of the tested surface of the thin film to obtain the actual thickness of a thin film at any location on a sample, which is calculated by averaging the sharp edge of difference. Because the data tested before and after coating suffer a rotation between testing, we flattened a section of the different substrates using a polisher, and a 10 mm length straight edge can be seen in Fig. 3. Using this edge, the rotation can be easily eliminated. As a result, accurate thicknesses of the  $\text{TiO}_2$  films on these two substrates were obtained, and the thickness of the thin film deposited on substrate 1 is 116.1 nm, whereas that on substrate 2 is 106.6 nm, and the thickness of the  $\text{TiO}_2$  film deposited on substrate 1 is 9% greater than that deposited on substrate 2. This indicates that the substrate surface properties have an influence on the thin-film thickness, although the thin film replicates the surface properties of the substrate. The substrate surface with a larger PV value affects the carrier mobility in the process of thin-film growth, resulting in a lower packing density of the thin film and then higher physical thickness of the film, which would lead to increased light scattering and to an increased number of nodules [20], which is significant because defects in thin films are one of the main factors that affect their LIDT [21]. On the contrary, the microstructure of the thin film growing on substrate 2 is compact and stable.

In addition, we have calculated the electric field intensity (normalized electric field intensity squared) at the film-air interface using TFCalc thin film design software based on Maxwell's equations, as shown in Fig. 6. The horizontal axis of the graph represents the distance from the film-air interface, which is defined as the initial point, where the distance is negative in the direction of the air and is positive toward the film. The Fig. indicates that the electric field intensities at the film-air interfaces of the thin films deposited on substrate 1 and substrate 2 are negligibly different. Therefore, the substrate with high PV and RMS surface roughness values, while producing a thicker film, has a negligible influence on the electric field intensity distribution at the air-film interface. In other words, the surface properties of the substrate have an influence on the thin-film microstructure and surface properties, but not on the electric field intensity distribution. This conclusion is reinforced by measurements of the film surface morphologies of these two samples imaged by atomic force microscopy (AFM) before laser irradiation. Fig. 7 indicates that the surface of the thin film deposited on substrate 1 is comprised of deep grooves and a convex hull and the microstructure of film deposited on substrate II is more compact. The increase in the packing density of the film results in an increase of the LIDT of a film irradiated by a high-power laser, because such a surface would induce greater heating [22, 23].

#### 4. CONCLUSION

In this work, we investigated the influence of the substrate surface properties on the characteristic of laser-induced damage of  $\text{TiO}_2$  thin films.  $\text{TiO}_2$  films of a 1/4 optical thickness were deposited by electron beam thermal evaporation on two types of substrates with different surface properties, and the optical and laser-induced damage properties were analyzed. For the  $\text{TiO}_2$  film deposited on the substrate with high PV and RMS surface roughness values, the optical properties, such as the refractive index, the transmittance, and the thin-film thickness, increased, but the LIDT clearly reduced.

The substrate with high PV and RMS values causes the deposition rate to decrease and the mobility of the atoms of the thin film material to diminish, resulting in a lower packing density of the thin film and an increased probability of film surface defects. Thus, the thin-film thickness increases. Meanwhile, the optical transmittance and surface roughness of



the resulting thin film also increases. An increase in all the above properties substantially reduced the laser-induced damage threshold of the thin film based on the thin-film thickness and microstructures, although the electric field intensity at the film-air interface of the thin film is not affected.

The behavior of the TiO<sub>2</sub> thin film under a high-power laser is therefore linked to the fabrication step. Control of the deposition time and choice of a substrate with low PV and RMS surface roughness values plays a substantial role in improving the laser-induced damage properties of the thin film.

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