

Contamination process and laser-induced damage of HfO₂/SiO₂ coatings in vacuum

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The performances of HfO₂/SiO₂ single- and multi-layer coatings in vacuum influenced by contamination are studied. The surface morphology, the transmittance spectrum, and the laser-induced damage threshold are investigated. The results show that the contamination in vacuum mainly comes from the vacuum system and the contamination process is different for the HfO₂ and SiO₂ films. The laser-induced damage experiments at 1064 nm in vacuum show that the damage resistance of the coatings will decrease largely due to the organic contamination.

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As the laser systems will be operating in the vacuum of space, the quality of the laser coatings in vacuum becomes more and more important. For example, various space laser systems such as the Mars Orbiter Laser Altimeter (MOLA1) system and Geoscience Laser Altimeter System (GLAS) have appeared the function decline due to the damage of coatings. The vacuum circumstance is complicated compared with atmosphere. In recent years, the coatings used in vacuum environment have been researched broadly^[1–8]. The hafnia/silica films are widely used in high-peak-power laser systems because of their high damage resistance^[9–12]. In this letter, we study the contamination process of the hafnia and silica films in vacuum. The change of the optical performance and laser-induced damage threshold are investigated.

Single layers of HfO₂ and SiO₂ coatings were deposited by electron beam evaporation on Leybold APS1504 coating machine. BK7 and fused silica were used as substrates. The substrates were well polished and the surface roughness was less than 1 nm. The deposition rates for HfO₂ and SiO₂ were 0.2 and 1.0 nm/s, respectively. The base pressure was 1×10^{-3} Pa and the oxygen gas was fed into the vacuum chamber to 6.0×10^{-3} Pa during deposition. The substrate temperature was about 200 °C. The electronic field will affect only several layers near the surface when the center wavelength of the reflective band of the coatings is equal to the wavelength of the irradiation laser. So the reflective film of HfO₂/SiO₂ is designed to transmit the 1064-nm laser in order to research the laser damage on contaminated coatings. The film structure was (HL)¹⁶L, where H denotes high-index material HfO₂ with one quarter wavelength optical thickness (QWOT) and L denotes low-index material SiO₂, respectively. The designed center wavelength was 950 nm. The optical transmittance of samples was measured by a Lambda950 spectrometer. The surface morphology of coatings was inspected by a Normaski microscope.

The stainless steel vacuum chamber has a volume of 700 mm³. The vacuum system consisted of a mechanical fore-pump and a turbo-molecular pump, capable to achieve a baseline vacuum of better than 10^{-4} Pa in the chamber. Samples were in the vacuum cavity with less than 10^{-3} Pa during the experiment.

The laser damage measurement is taken on the laser damage testing system. The laser source was a 1064-nm Nd:YAG laser with 5-ns pulse duration. The laser damage thresholds of testing samples were measured in the air and vacuum according to the international standard ISO11254. The threshold was derived from the zero damage possibility, which was obtained by the linear extrapolation in the damage possibility picture.

Figures 1 and 2 show the surface of HfO₂ and SiO₂ coatings respectively in air and in vacuum after 7 and 14 days. It is shown that the surface has obviously adherent contaminations. The morphology is different: the contamination is ball profile for SiO₂ and steak profile for HfO₂ due to their adherent characteristics. Figure 3 shows the surface morphology of multi-layer coatings. The adherent contamination is obvious and will be more after days.

The substance of the contaminations attracted by the coatings was analyzed by a gas chromatograph-mass spectrometer (GC-MS). The result showed that the majority species were phthalates and alkanes

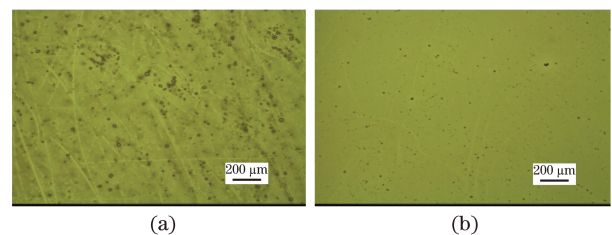


Fig. 1. Surface morphology of HfO₂ coatings in vacuum after (a) 7 and (b) 14 days.

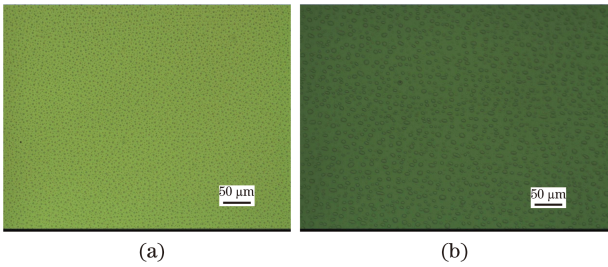


Fig. 2. Surface morphology of SiO₂ coatings in vacuum after (a) 7 and (b) 14 days.

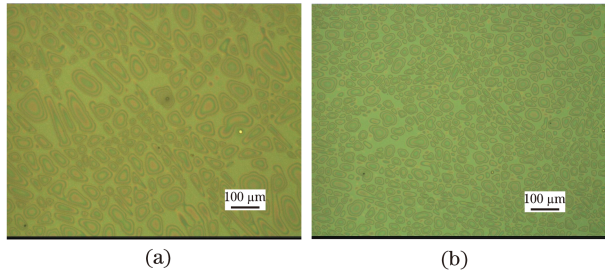


Fig. 3. Surface morphology of HfO₂/SiO₂ multi-layer coatings in vacuum after (a) 7 and (b) 14 days.

(including pentacosane, hexacosane, heptacosane, and otacosane). Phthalates are used in the vacuseal grease commonly, and alkanes maybe come from the oil used in the mechanical pump.

We measured the transmittance spectra of as-grown samples which were stored in vacuum for several days. Figures 4 and 5 show the transmittance variation of single-layer SiO₂ and HfO₂ coatings in vacuum.

As shown in Fig. 4, the transmittance of SiO₂ films have been gradually decreased with the time in vacuum. It means that the contaminations attracted by films absorb the light from visible to infrared, and the thickness of the contamination gradually increases with time in vacuum. As shown in Fig. 5, for HfO₂ coatings, the center wavelength moves to the long-wavelength range and the transmittance decreases when the time increases in vacuum. The reason of wavelength shift and transmittance loss may be complicated. It is known that the refractive index of oil is larger than air and water, and the coatings deposited by electron beam evaporation have many pore structures. We believe that the contaminations attracted by HfO₂ coatings have entered the coatings, increased the effective index of coating material, and made the shift of center wavelength.

The variation of the spectrum indicates that the adherent mechanism is different for the two kinds of coatings. Adherent contaminations gather on the surface of SiO₂ coatings, but enter the HfO₂ layers.

Figure 6 shows the transmittance spectrum of multi-layer coatings after 2, 7, and 14 days in vacuum. We can see that the spectrum moves to the long-wavelength side when the time increases in vacuum. The center wavelength moves about 30 nm after 7 days. The reason of center wavelength shift is that the effective refractive index of hafnia increases. After 14 days in vacuum, the transmittance of multi-layer coatings decreases obviously by about 50% due to the mass contaminations.

The damage thresholds of samples were measured in air and vacuum, respectively. The experimental results

are shown in Fig. 7. As we can see, the damage thresholds of the coatings tested in air decrease after being contaminated in vacuum, and the threshold measured in vacuum will largely decrease compared with that in air.

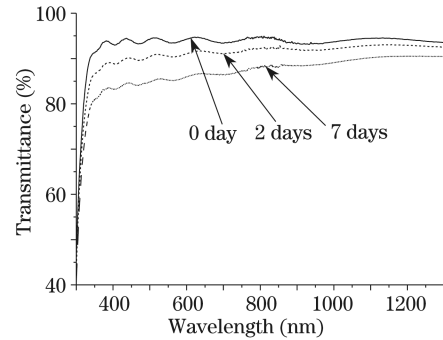


Fig. 4. Transmittance spectra of SiO₂ coatings in vacuum after several days.

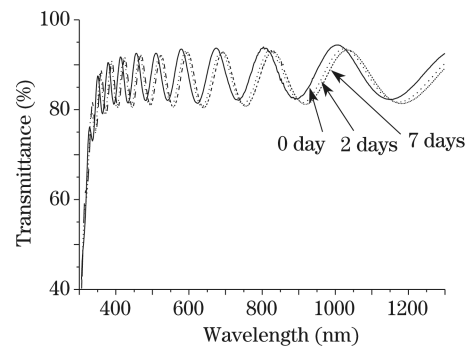


Fig. 5. Transmittance spectra of HfO₂ coatings in vacuum after several days.

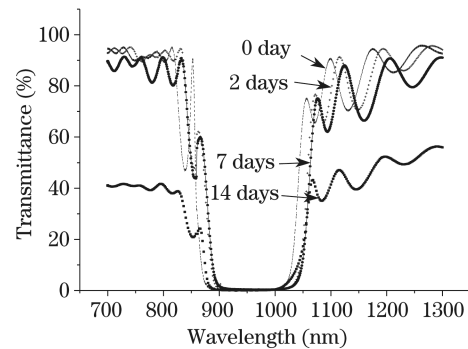


Fig. 6. Transmittance spectra of HfO₂/SiO₂ coatings in vacuum after several days.

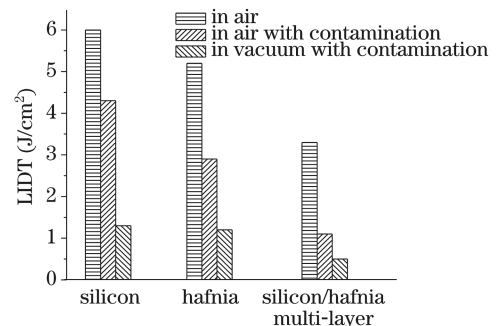


Fig. 7. Laser-induced damage thresholds (LIDTs) of the samples.

As we know, the damage of coatings is caused by the defects which absorb the laser energy. When the defects are irradiated by laser, the temperature will be increased to the melting point of the material and induce the coating damage. In vacuum, the contaminations attracted by coatings tend to absorb more laser energy and make the defect be damaged more easily.

In conclusion, we have studied the contamination process and laser-induced damage of the $\text{HfO}_2/\text{SiO}_2$ coatings in vacuum. It is found that the adherent mechanism is different for the two kinds of coatings and the transmittance changes in vacuum as time goes on. Due to the adherent contamination, the laser damage threshold of coatings in vacuum will largely decrease compared with that in air. However, a lot of work has to be done in order to improve the laser damage resistance in vacuum of coatings used in high-power laser system. This will be further studied in the future.

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