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Study on high-reflective coatings of different designs at 532 nm

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Conventional HfO\(_2\)/SiO\(_2\) and Al\(_2\)O\(_3\)/HfO\(_2\)/SiO\(_2\) double stack high reflective (HR) coatings at 532 nm are deposited by electron beam evaporation onto BK7 substrates. The laser-induced damage threshold (LIDT) of two kinds of HR coatings is tested, showing that the laser damage resistance of the double stack HR coatings (16 J/cm\(^2\)) is better than that of the conventional HR coatings (12.8 J/cm\(^2\)). Besides, the optical properties, surface conditions, and damage morphologies of each group samples are characterized. The results show that laser damage resistance of conventional HR coatings is determined by absorptive defect, while nodular defect is responsible for the LIDT of double stack HR coatings.

The transmittance of all samples was measured by a spectrometer (Lambda1050UV/VIS/NIR, Perkin-Elmer, USA). The 1-on-1 damage test was according to ISO 11254-1 \[14\]. The laser pulse produced by the Nd:YAG laser with a frequency doubling crystal was irradiated on the sample surface through the optical path, as shown in Fig. 1. The pulse duration is 10 ns. Twenty sites were irradiated by the same pulse energy and the damage probability was recorded. The fluence decreases until no damage occurs. The relative error of damage probability was about ±10% due to the uncertainty of the measurement process.

The chemical composition of coatings was obtained by a thermal scientific Kα X-ray photoelectron spectroscopy...
The surface condition of coatings and damage morphologies after laser irradiation were captured by a Carl-Zeiss Auriga field emission scanning electron microscope (SEM). The cross-sectional micrograph was obtained by focused ion-beam (FIB) technique.

The transmittance spectra of both HR coatings are shown in Fig. 2. All the transmittance at 532 nm is lower than 1%. The difference of the spectra has little influence on the performance of coatings at the central wavelength. The width of the reflectance band of the double stack coatings was greatly narrowed due to the relatively low refractive index of Al₂O₃.

XPS was utilized to measure the chemical composition of coatings. The atomic ratio of O/Si is 1.536. The atomic ratio of O/Hf for conventional coatings is 1.983. And the atomic ratio of O/Al for double stack coatings is 1.430.

SEM was exploited to obtain the information of surface condition, nodules were counted and the average density was calculated for the two kinds of HR coatings. The double stack coatings have much higher nodule density (2.909 mm⁻²) than the conventional coatings (0.234 mm⁻²).

1-on-1 damage test was done for many samples of each design. The damage probability versus the incident laser fluence is shown in Fig. 3. The LIDT of double stack HR coatings presented a 25% increase than the conventional HR coatings (12.8 J/cm²), which could reach 16.0 J/cm². Moreover, the slope of the linear fitted curve reflects the density of sensitive defects in the coating stacks [15]. The defect density of the conventional HR coatings was much higher than that of the double stack, which could be concluded from the much steeper curve.

The double stack did not experience any damage at fluence below 13 J/cm², and the LIDT was determined by the first occurrence of a single crater with a diameter of 4 μm. This threshold was 16.0 J/cm², which is higher than the 12.8 J/cm² for the conventional coatings. The probability of damage initiators in the double stack coatings was greatly narrowed due to the relatively low refractive index.

The 100% probability damage threshold was greatly improved in double stack HR coatings, namely the LIDT of some defects in double stack HR coatings have the potential to reach a relatively high value (nearly 45 J/cm²).

Precise information about damage initiators could be characterized by SEM. The typical damage morphologies of the conventional coatings were shown in Fig. 4. There was single crater with little plasma scald at low laser fluence near the LIDT (Fig. 4(a)). With laser fluence increased, many small craters linked together with plasma scald surrounded (Figs. 4(b) and (c)). Damage morphologies of the double stack coatings are shown in Fig. 5. Unlike the conventional coatings, damage initiators were changed from low (Fig. 5(a)) to high (Figs. 5(b) and (c)) laser fluence irradiation corresponding to the change of damage crater species. From the above, the damage initiators were defects for both HR coatings.

Damage craters induced by laser fluence slightly above LIDT were considered to be the damage initiators in coatings. The FIB technology was applied to position the bottom of the damage craters accurately.

For the conventional HR coatings, Fig. 6 is the FIB results of Fig. 4(a). The first HfO₂ layer is melted that can be seen from Fig. 6(a). Figure 6(b) shows that the damage initiator is nano-absorptive defect in the third HfO₂ layer. The origin of the absorptive defect in the conventional coatings may be the incomplete oxidation of hafnium during the deposition process.

For the double stack HR coatings, the FIB result of damage crater in Figs. 5(a) and (c) is illustrated in Fig. 7. FIB cross-sectional micrograph in Fig. 7(a) corresponds to Fig. 5(a) near the LIDT, shows that the damage initiator is nodular defect in Al₂O₃/SiO₂ layers of the double stack. So the LIDT was determined by the nodule defects. The ejection happened easily when pre-melting Al₂O₃ coating material, which would introduce much more nodular defects. One typical damage site in Fig. 5(c) was cut by FIB showed in Fig 7(b), which was induced by high laser fluence. The figure revealed that the absorptive defects in Al₂O₃/SiO₂ layers worked under high laser fluence irradiation.
In conclusion, the Al$_2$O$_3$/HfO$_2$/SiO$_2$ double stack HR coatings present higher LIDT and 100% probability damage threshold than the conventional HfO$_2$/SiO$_2$ HR coatings. The damage initiators of two designs are different. The absorptive defects in the HfO$_2$ layers are dominant in the conventional HR coatings. While defects in the upper Al$_2$O$_3$/SiO$_2$ stacks are responsible for the damage of the double stack HR coatings. The nodular defects are responsible for the near LIDT damage, and the absorptive defects work at high laser fluence irradiation. This work points the way to enhance the LIDT of 532 nm dielectric coatings. For the conventional coatings, avoiding absorptive defects in HfO$_2$ layers is imperative. While for the double stack coatings, improving the deposition process of Al$_2$O$_3$ layer to reduce nodular defects in top Al$_2$O$_3$/SiO$_2$ stacks could be much more effective.

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References