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

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Conventional HfO_2/SiO_2 and $Al_2O_3/HfO_2/SiO_2$ 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.

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Dielectric coatings at wavelength of 532 nm are used in Ti: sapphire chirped-pulse amplification laser system[1], which has drawn more and more attention to the fabrication of 532 nm dielectric coatings with high quality. For high power laser system, the laser-induced damage theshold (LIDT) is an important factor that defines the quality of one optical coating component. Compared with 1064 and 355 nm optical coatings[2−9], researches conducted on 532 nm optical coatings are relatively few[10−12]. In order to further enhance the potential LIDT of 532 nm high reflective (HR) coatings, we are inspired by the proposal of double stack design for 355 nm HR coatings. The proposal has brought increase in the LITD of 355 nm HR coatings[7,8]. The so called double stack coating utilizes the laser resistive stacks as upper protection layers and highly reflective stacks as bottom layers. In this article, conventional HfO_2/SiO_2 stack and $Al_2O_3/HfO_2/SiO_2$ double stack are designed and investigated.

The film stack of conventional design was S/4L(HL)^{10}H6L/Air. The double stack design was the combination of two oxide coating stacks, which was expressed as S/4L(HL)^{5}4L(ML)^{15}M6L/Air. The thicknesses of conventional and double stack coatings are about 2600 and 4780 nm, respectively. Stacks (HL)^5 is used to offer high reflectivity, and stacks (ML)^{15} is utilized to resist laser damage. Here, the center wavelength is 532 nm, S refers to substrate, and H, L, M denotes HfO_2, SiO_2, Al_2O_3 layers with quarter-wavelength optical thickness (QWOT), respectively. Al_2O_3 is chosen as the candidate for original material in the double stack due to its high LIDT[13]. The original materials are produced by Merck in Germany, reaching the industrial purity level.

Samples of two designs were prepared on super polished BK7 glass substrates with diameters of 50 mm by electron beam evaporation in a coating machine (SYRUS 1110, Leybold Optics, Germany). The substrates were pre-cleaned ultrasonically in de-ionized water before they were loaded into the coating chamber. During the coating deposition, the only difference between two designs is the deposition process of HfO_2 layer. For the conventional HR coatings, hafnium was oxidized as the HfO_2 layer. For the double stack HR coatings, hafnia was used as the original material, because indirectly water-cooled crucible was used during the deposition of Al_2O_3 layers, heat radiation of which was not so well to deposit hafnium.

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 uncertainly of the measurement process.

The chemical composition of coatings was obtained by a thermal scientific Kα X-ray photoelectron spectroscopy

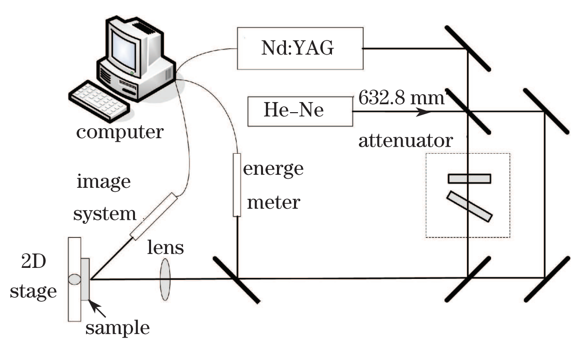

Fig. 1. Experiment setup for laser damage test.

(XPS). 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 Al_2O_3.

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/cm^2), which could reach 16.0 J/cm^2. 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.

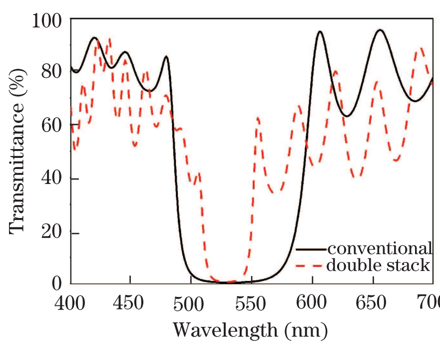

Fig. 2. (Color online) Transmittance spectra for samples of conventional and double stack coatings.

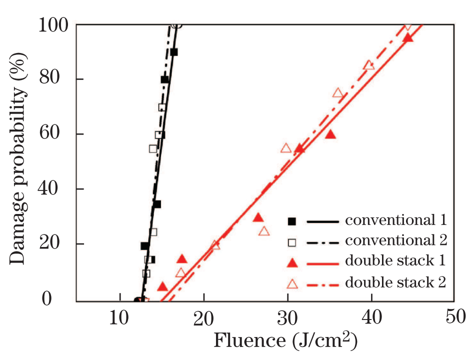

Fig. 3. (Color online) Damage probability curves and linear fitted curves of two samples for conventional and double stack coatings.

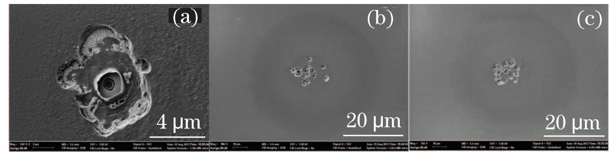

Fig. 4. Damage morphologies of conventional HR coatings for laser fluence at (a) 13.2, (b) 15.6, and (c) 16.7 J/cm^2.

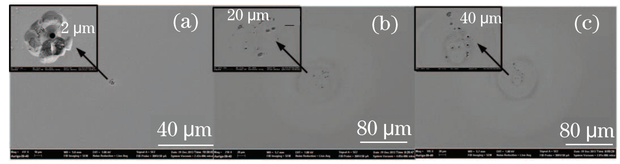

Fig. 5. Damage morphologies of double stack HR coatings for laser fluence at (a) 17.6, (b) 35.3, and (c) 44.5 J/cm^2.

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/cm^2). To investigate the mechanisms of such improvement after introducing double stack design, some characterization was conducted to identify the nature of damage initiators.

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 HfO_2 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 HfO_2 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 Al_2O_3/SiO_2 layers of the double stack. So the LIDT was determined by the nodule defects. The ejection happened easily when premelting Al_2O_3 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 Al_2O_3/SiO_2 layers worked under high laser fluence irradiation.

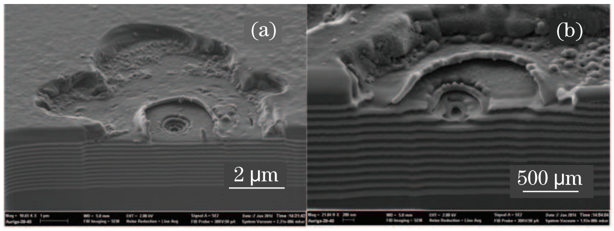

Fig. 6. Cross sections of damage sites shown in Fig. 4(a).

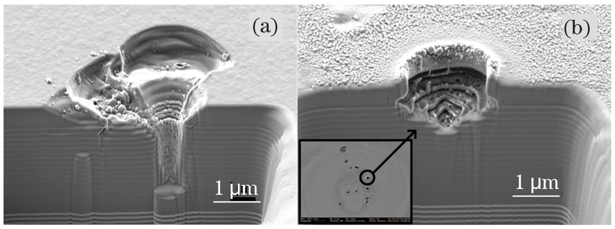

Fig. 7. Cross sections of damage sites shown in Figs. 5(a) and (c).

In conclusion, the Al_2O_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_2O_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_2O_3 layer to reduce nodular defects in top Al_2O_3/SiO_2 stacks could be much more effective.

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