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Investigation of high power laser coatings

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Abstract: High laser-induced damage threshold and large aperture were focuses on the studies of high power laser coatings. This paper reports the research activities at our center. Several measures were developed for evaluating characters of laser damage, including determination of laser induced damage threshold and detection of absorption based on surface thermal lensing technique. Defect was deemed to be the initial source of laser damage, and was the main factor restricting the laser damage resistance of optical coatings. The contribution of several kinds of typical defects to laser damage was analyzed, and some deposition measures were adopted to control and eliminate the origin of defect. Furthermore, some post-treatment methods were also employed to alleviate the influence of the defect and to improve the laser damage resistance. Correction mask was introduced to improve the thickness uniformity, and the thickness uniformity can be amended to less than 1% in the range of Φ650 mm. Preliminary investigation related to surface deformation was also conducted.

Key words: high power laser coating; laser induced damage threshold (LIDT); defect; thickness uniformity; oxygen-plasma post-treatment; large aperture; surface deformation

1　Introduction

Optical thin film coating is one of the weakest optics in ICF laser systems. Totally, there are two aspects which influence the development of the laser systems. The first aspect is laser damage resistance of the coatings, which puzzled coating engineers for many years[1]. Although much great progress has been made in theoretical analysis of damage mechanisms and optimization of deposition procedures, there were still many sealed problems restricting the further improvement of the laser induced damage threshold (LIDT)[2-7]. The laser-induced damage threshold of optical coatings is influenced by a large number of factors, such as mechanical properties[8], thermal properties[9], chemical composition[10], crystallographic properties[11], microstructure[12], defects[13-14], coating design[12,15-16], laser parameters[17], application conditions and environmental influences[18]. The most prominent factor is the defects, which is the weakest part in optical coatings and is the initial source of laser damage. To control and eliminate the origin of defect can improve the LIDT effectively, but it was always expensive and difficult[19-20]. As a result, some post-treatment methods were also introduced to improve the laser damage resistance, including laser conditioning[21-23] and annealing[24-25]. The second aspect is the problems encountered when the size of the laser coatings grows to large aperture. This kind of problem includes thickness uniformity and stress-induced surface deformation. Our attention in this paper will be mainly paid to the laser damage behaviors

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of optical coatings and some measures to control or eliminate the influence of defects. Some preliminary study related to the large aperture coatings was also conducted.

2 Laser damage properties detection

2.1 Laser induced damage threshold determination

Laser induced damage threshold was related to damage probability and should be evaluated by damage probability method. In short, a minimum of ten sites was exposed to a given pulsed laser energy and the damage probability was recorded. This procedure was repeated for other specific fluences to develop a plot of damage probability versus energy density. A linear extrapolation to 0 % damage probability yielded the LIDT[26-27].

2.2 Weak absorption detection

Absorption is the source of laser damage. Absorption detection is helpful for understanding laser damage mechanism. Surface thermal lensing (STL) technology is one of the most convenient methods and have higher signal-to-noise ratio[28-30]. Photo-thermal microscopy in the raster-scanning mode based on STL technology has been developed to analyze the absorption distribution of the sample.

3 Defect and its contributions to laser damage

Formation of defects is complicated and it is depended on the deposition materials and procedures. Classification of defects is also difficult because of the limited detection techniques. Generally speaking, there are several kinds of defects, such as nodular, impurity, off-stoichiometry defect and color center. The contribution of these kinds of defects is different form different instances. Most reports about defect-initiated laser damage showed that nodular was the dominate factor of laser damage, but from our researches, it was rarely to find nodular for most kinds of coatings. Fig. 1 shows the typical morphology of the cross section, and there are no nodular exist.

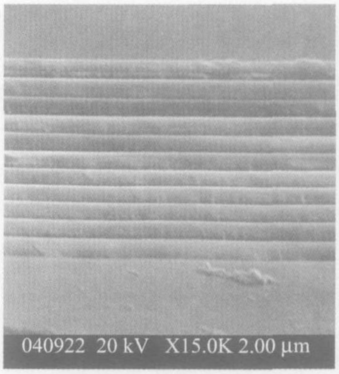


Fig. 1 Typical SEM morphology of optical coating section

However, other kinds of defects will play a great role in laser damage. Firstly, the impurity is the most obvious laser damage initiator, which depends on the purification procedure of the deposition materials. The impurity elements of several kinds of deposition materials were analyzed by Glow Discharge Mass Spectrometer (GDMS), and the results were summarized in Tab. 1. We can find that there are several absorptive elements for $ZrO_2:Y_2O_3$ and the content of metal element platinum is the highest. It is fatal to the laser damage resistance. Secondly, incomplete oxidation of the material atoms during evaporation will cause off-stoichiometry defects, whose absorption is much higher than other part of coatings and which is invisible for most instances.

Tab. 1 GDMS analysis of deposition materials ($ZrO_2:Y_2O_3$)

Elements	Pt	Al	Cu	Ti	Fe
Content ($\times 10^{-6}$)	9790	42	42	12	3.5

4 Some deposition measures to restrain the defects

Restrain defects can improve the laser damage resistance effectively. Two ways were mostly adopted: one is controlling the origin of the defects during deposition procedures, another is wiping off the influence of the defects by post-treatment methods. Some deposition measures will be discussed in this section, and post-treatment methods will be illustrated in the next section.

4.1 Application of ion beam sputtering (IBS) in high power laser coatings

IBS technique has its advantages in restraining the defect, and improves the laser damage resistance consequently. Moreover, the packing density of IBS prepared samples will be higher than that of E-beam deposition, and for most instances the samples is non-crystallization, which usually has higher LIDT than that of crystallization samples. Fig. 2 summarizes the absorption properties at 1 064 nm of the samples, which was detected by photothermal microscopy in the raster-scanning mode based on STL technology. Because raster-scanning was time-consuming, only line scan in 2 mm was performed. It is found immediately that the absorption properties were homogeneous and there was no absorptive defects found. The 1-on-1 LIDT of the mirror was greater than 30 J/cm^2 (1 064 nm, 12 ns, incident angle is 45°, p polarization). From the typical damage morphology (Fig. 3), it is found that the damage is initiated from the surface of the coating and is not related to local defects. That means that the IBS prepared sample can restrain the defects and it is helpful to improve the laser damage resistance. The electric field distribution analysis (Fig. 4) showed that the electric field of outside layer is the highest site and it is just the site damage promoted.

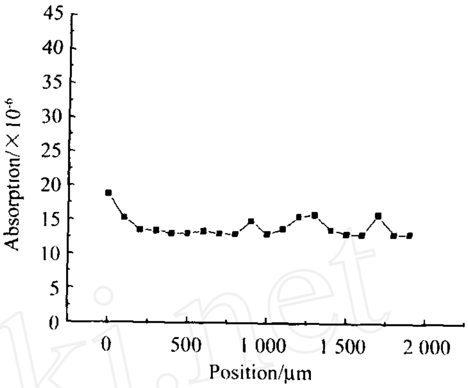


Fig. 2 Absorption detection results of pick-off mirror

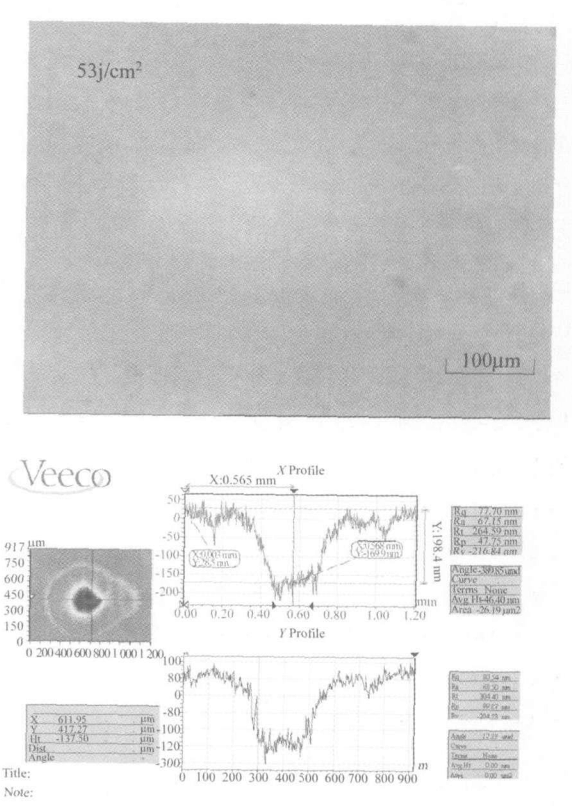


Fig. 3 Damage morphology

4.2 Oxygen partial pressure[31]

Off-stoichiometry defects originate from incomplete oxidation of deposition material atoms or clusters. Suitable oxygen partial pressure is important for eliminating off-stoichiometry defect. The ZrO_2 monolayer was prepared by electron beam evaporation (EBE) with different oxygen partial pressure, varying from 3 ×10^{-3} Pa

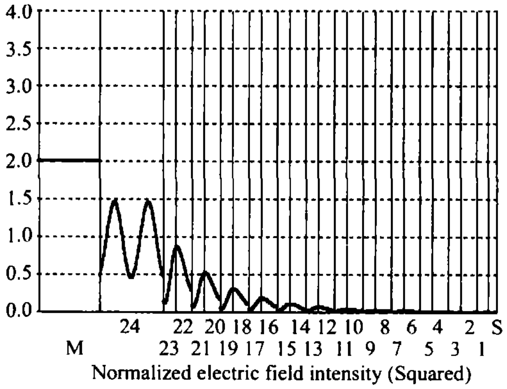


Fig. 4 Electric field distribution analysis of pick-off mirror

to 11 ×10^{-3} Pa.

The refractive index data are indicative of the film density[32]. The refractive indices of the films at ～550 nm were calculated from the transmittance spectra by the use of the envelope method developed by Manifacier *et al*.[33]. As illustrated in Fig. 5, the refractive index decreases with increasing oxygen partial pressure. This means the film packing density also decreases with the oxygen partial pressure.

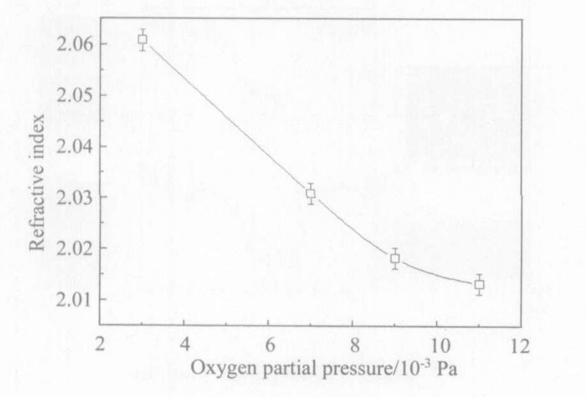


Fig. 5 Refractive index of ZrO_2 at 550 nm for different oxygen partial pressure

Fig. 6 presents the samples' absorptance variation with the oxygen partial pressure. A monotonic decrease of absorptance varying from 125.2 ×10^{-6} to 84.5 ×10^{-6} with increasing oxygen partial pressure was observed in the plot. Off-stoichiometry of oxidation is one of the main causes of the absorption, since high absorbing suboxide components are formed easily during deposition because of oxygen deficiency, and adequate oxygen can repair oxygen vacancies[34]. So the off-stoichiometry of the ZrO_2 films caused by insufficient oxygen content is the main cause of the absorbance differences.

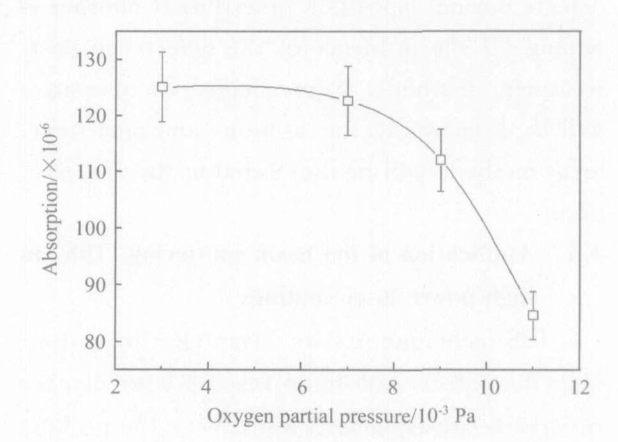


Fig. 6 Absorption of the ZrO_2 monolayers with different oxygen pressure

The dependence of LIDT of the ZrO_2 thin films on oxygen partial pressure is shown in Fig. 7. It shows clearly that the damage threshold increases gradually from 18.5 J/cm^2 to 26.7 J/cm^2 with oxygen partial pressure variations from 3 ×10^{-3} Pa to 9 ×10^{-3} Pa. However, there is a drastic decrease of the LIDT when the oxygen partial pressure exceeds 9 ×10^{-3} Pa.

When oxygen partial pressure varies from 3 ×10^{-3} Pa to 9 ×10^{-3} Pa, the absorption and packing density decrease correspondingly, so it is reasonable that the LIDT of the samples increases. When oxygen partial pressure exceeds 9 ×10^{-3} Pa, the LIDT decreases dramatically. So we can deduce that the low LIDT of sample D may be partially ascribed to excessively loose structure and poor mechanical stability of the films due to the optimum oxygen content being exceeded, and microstructure is the dominant factor to the LIDT.

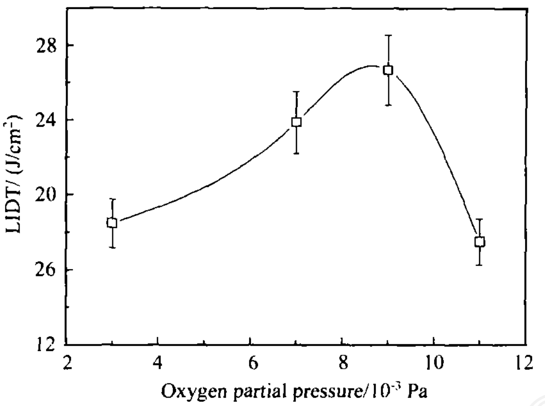


Fig. 7 LIDT of the ZrO_2 monolayers with different oxygen pressures

5 Post-treatment methods

5.1 Oxygen plasma post-treatment[34]

Post-treatment methods were widely introduced to high power laser coating fabrication, such annealing and laser conditioning. These methods can improve the laser damage resistance effectively for some specific instances. As one of the post-treatment measures, oxygen plasma post-treatment also can influence the defect properties, which is related to laser damage behaviors.

EBE prepared ZrO_2 monolayer with thickness of 450 nm was treated for 12 min with low energy oxygen plasma after deposition. The defect density in the monolayer was detected under Nomarski dark field microscope with 100 times magnification. The dimension of these micro-defects is about in μm-scale. Fig. 8 presents the micro-defect density of the samples. A1～A5 and B1～B5 represent random five sites on the sample surfaces before and after treatment, and the average defect density on the five sites are 18.6/mm^2 and 6.2/mm^2 respectively. The results indicate that the defect density reduced evidently after oxygen plasma treatment. Generally speaking, micro-defects on the sample surface are caused by spitting in evaporation process. They often weakly connect with the coatings and could be detached from the coatings easily by outer disturbing, such as ion treatment, laser conditioning, and etc. The absorption properties of the samples before and after treatment are summarized in Fig. 9. The sites with high absorption are the sites where thermal defects exist. When the sample was treated with oxygen plasma, the absorptions become uniformity. This phenomenon is consistent with the defect density alteration after treatment. The LIDT of the sample also increass greatly as shown in Fig. 10.

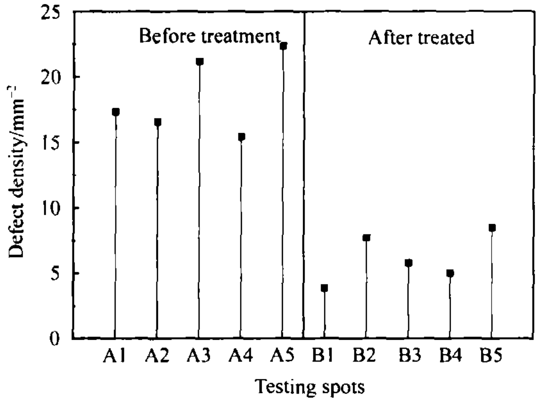


Fig. 8 Micro-defect density variation of the samples after being treated by low energy oxygen plasma

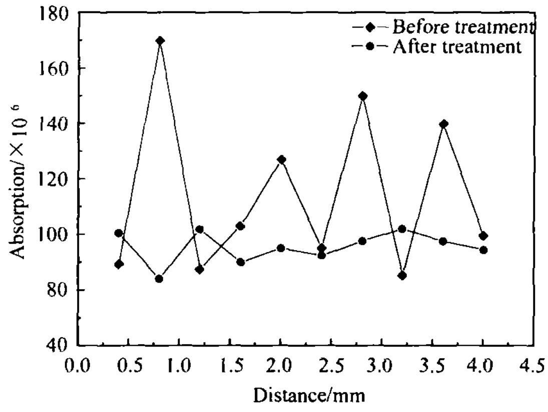


Fig. 9 Absorption of the samples before and after being treated with low energy oxygen plasma

Fig. 11 presents the bright filed microscopy images of damage morphologies. We could see clearly that most of the damage sites on the sample surface are centered with a defect site for the As-grown sample; whereas for the ion treated sample, the damage morphology appears to be intrinsic damage of coating material.

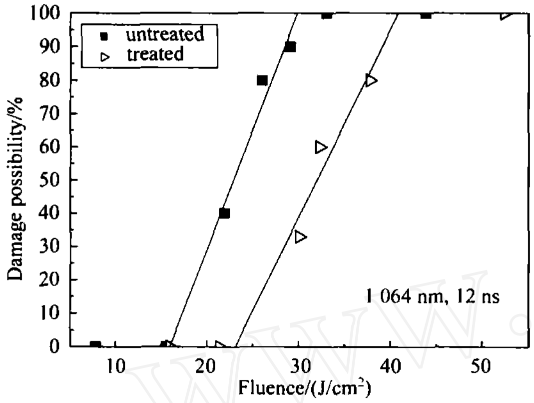
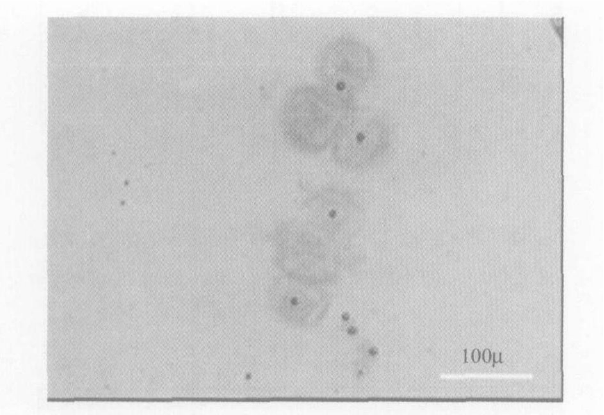
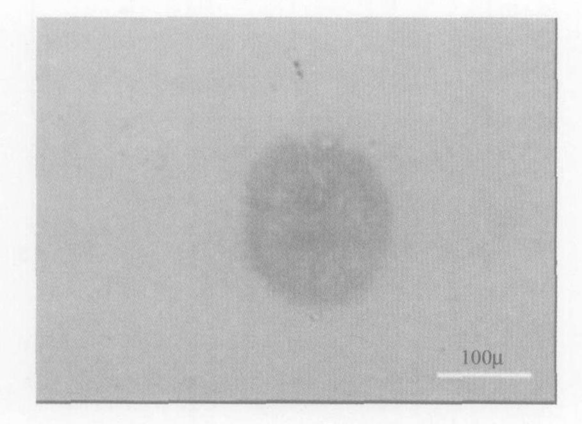


Fig. 10 LIDT of the sample before and after post-treatment



(a) As-deposited sample(22.6 J/cm^2)



(b) Treated sample(26.3 J/cm^2)

Fig. 11 Damage morphologies of the samples

Oxygen plasma treatment may stabilize the micro-defects (for example, eject defects gently) in the coatings so that they are not highly susceptible to laser damage.

6 Several key problems of large aperture coatings

6.1 Uniformity of optical properties

The uniformity of the optical properties of the large aperture coatings was decided by the uniformity of the physical thickness of multilayer, which was related to the configuration of the vacuum chamber and evaporation emission properties of the deposition source for PVD method. According to the specific coating chamber with 1 800 mm diameter, we established the physical

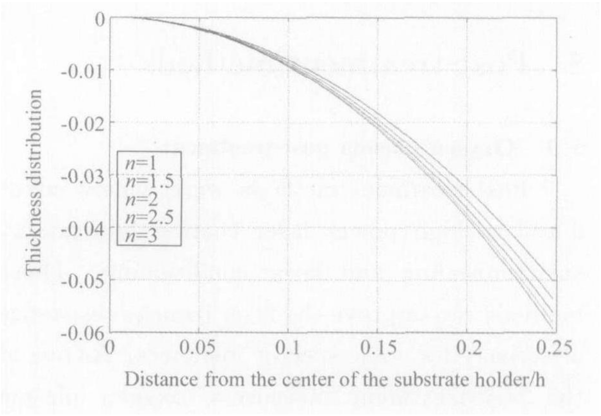


Fig. 12 Theoretical simulation of physical thickness uniformity

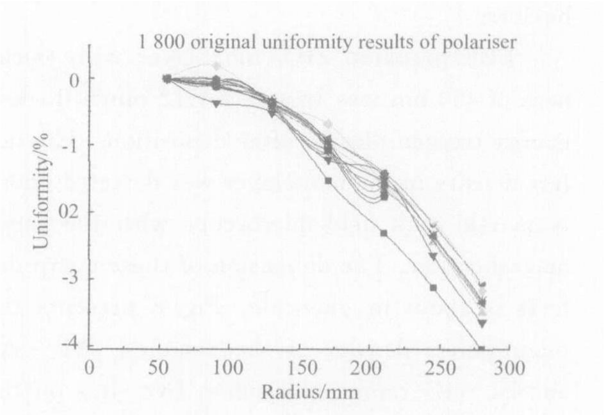


Fig. 13 Experimental results of the uniformity

thickness uniformity model. The theoretical simulation (Fig. 12) of the thickness uniformity is in correspondence with the experimental results (Fig. 13).

Correction mask was employed to amend the thick uniformity of the large aperture coatings, and the optimized experimental result was shown in Fig. 14. The thickness uniformity of polarizer can be amended to less than 1 % in the range of 650 mm.

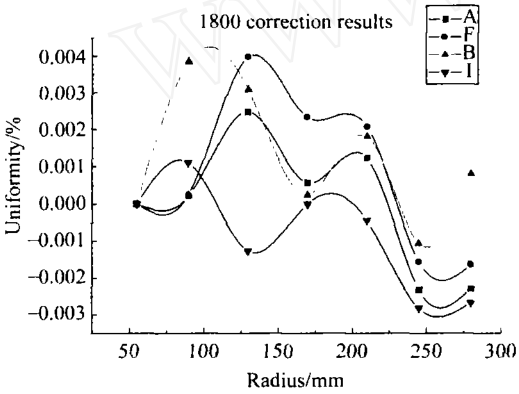


Fig. 14 Uniformity after correction

6.2 Surface deformation

Surface deformation of the laser coatings with large aperture is very complicated. Establishing the correlation between surface deformation and deposition procedure is the basis to discover the problem. HR coatings for 1 053 & 633 nm with different incident angle were chosen to disclosure the regularity. The parameters of the coatings are summarized in Tab. 2. Some statistics of surface deformation are summarized in Fig. 15. The surface deformation was scaled by power value.

It is found that power value increases as the incident angle increases, which means the stress increases as thickness of the multilayer increased. Furthermore, the power value increases as sample dimension increases. The compressive stress appeared in 0° and 45° samples, whereas tensile stress appeared in 22.5° samples. This is a strange phenomenon, and further study need to illuminate this problem.

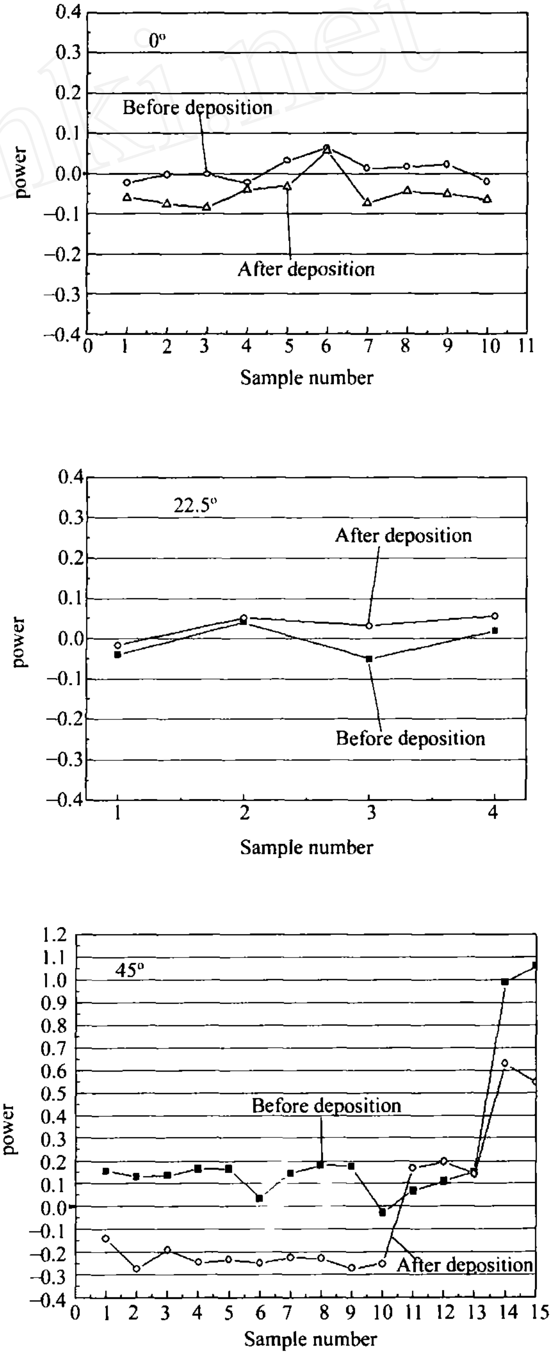


Fig. 15 Statistics of surface deformation

Tab. 2 Parameters of the coatings

HR @1053 &633 ,0 °,P		HR @1053 &633 ,22. 5 °,P		HR @1053 &633 ,45 °,P	
Sample number	Dimension (mm ×mm ×mm)	Sample number	Dimension (mm ×mm ×mm)	Sample number	Dimension (mm ×mm ×mm)
1	Φ50 ×8	1	120 ×88 ×15	1	Φ50 ×8
2	Φ50 ×8	2	120 ×88 ×15	2	Φ50 ×8
3	Φ50 ×8	3	120 ×88 ×15	3	Φ50 ×8
4	Φ50 ×8	4	120 ×88 ×15	4	Φ50 ×8
5	Φ50 ×8			5	Φ50 ×8
6	Φ50 ×8			6	Φ50 ×8
7	Φ50 ×8			7	Φ50 ×8
8	Φ50 ×8			8	Φ50 ×8
9	Φ50 ×8			9	Φ50 ×8
10	Φ50 ×8			10	120 ×88 ×15
				11	120 ×88 ×15
				12	120 ×88 ×15
				13	120 ×88 ×15
				14	340 ×240 ×40
				15	340 ×240 ×40

7 Conclusions

Some exploration work about high power laser coatings was reviewed. Contributions of defects to laser induced damage of optical thin film coatings were analyzed, and some deposition measures were employed to control and eliminate the origin of defects. Furthermore, oxygen plasma post-treatment was introduced to alleviate the influence of the defects. Based on these measures, the LIDT improved effectively. The thickness uniformity of large aperture coatings can be amended to less than 1 % in the range of Φ650 mm by correction mask. Some regulations of stress induced surface deformation were obtained, and further work to explain it is going on.

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