Comparative study of IR and UV laser damage resistance of silica thin films deposited by Electron Beam deposition, Ion Plating, Ion Assisted Deposition and Dual Ion Beam Sputtering

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ABSTRACT

The laser damage resistance of optical coatings is a critical point for a large number of applications. However improving this resistance is often hard to obtain because of the large number of parameters in the deposition processes than can modify the laser damage threshold and the lack of detailed and exploitable studies published on this subject. Then, the aim of this work is to test and analyze the laser damage resistance of a usual material for high power applications (silica) deposited in various conditions. The thin films of different thicknesses were specially deposited using different techniques available at the Institut Fresnel: Dual Ion Beam Sputtering, Electron Beam Deposition, Ion Assisted Deposition and Ion Plating. The laser-induced damage thresholds of these coatings were determined at 1064nm and 355nm using nanosecond pulsed YAG lasers, with a 1-on-1 test procedure. Other diagnostic tools were used to complete the study and make potential correlations with laser damage: photothermal techniques, luminescence spectrocopy, optical profilometry, dark field and Nomarski microscopy. The comparative study of these results highlight different laser damage behaviors of the silica coatings that we correlate to the density and the nature of the defects.

Keywords: laser damage, silica, thin film, EBD, DIBS, IAD, IP

1. INTRODUCTION

Producing laser damage resistant optical coatings has a considerable interest for high power laser applications. However, improving this resistance is often a difficult task because of the large number of parameters in the manufacturing processes than can can affect the properties of the resulting layers and modify the laser damage threshold.[1–7] Then the aim of this work is to test and analyze the laser damage resistance of a material commonly used for high power applications (SiO_2) deposited in various conditions. We used the different techniques available at the Fresnel Institute, to produce thin films in well known conditions.

In the first part of this paper we describe how we prepared and produced silica coatings using the different deposition techniques. Then the laser damage testing measurements are described and the results are compared and discussed. In the last parts, complementary non-destructive measurements are made to analyze the laser damage mechanism on these samples.

2. SAMPLE DESCRIPTION AND PREPARATION

For this study, we have chosen Herasil glasses specially polished for high power applications. To clean these substrates we used an automatic aqueous cleaning procedure, involving ultrasonic immersion and detergents followed by DI water rinsing and drying. To check the cleaning for each deposition batch, a bare silica reference sample was associated to each batch, and inspected after cleaning with a dark field microscope.

To produce the samples, we used different evaporation techniques: Dual Ion Beam Sputtering, Electron Beam

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Deposition, Ion Plating and Ion Assisted Deposition. For each deposition technique, samples of different mechanical thicknesses were produced (200nm and 1000nm) in order to investigate the influence of this parameter on the laser damage threshold. Two samples were produced for each thickness, to control the reproducibility of the process. Then the total number of thin film samples produced and tested in this study is 16. Some bare substrates were also tested as reference samples.

3. LASER DAMAGE THRESHOLDS MEASUREMENT

3.1. Testing facility and procedure

The test apparatus used for laser-damage testing has been described in detail in an other paper,[8] and only a brief description is given here. We used a first YAG laser beam with 1.064-μm wavelength and 5-ns effective pulse duration. The beam was focused down to a spot diameter of 12-μm on the sample. A second YAG laser was used for 355nm testing (spot diameter of 8-μm and 6ns effective pulse duration). The sample was observed with an *in situ* optical microscope (magnification from x50 to x500), which ensures real-time observation and recording of the irradiated zone. Any image modification after shot is our damage criterium (micronic damage can been detected).

Using this apparatus, we have measured laser damage probability curves with the 1-on-1 test procedure.[9] We recall that these curves are obtained by counting the number of damaged regions at each fluence F, which allows to estimate the damage probability P(F). We adopted a very refined test procedure in comparison with the ISO standard[9] : a first plot is made by testing 50 points for each energy (with 20 different energies), then a "zoom" is done on the low part of the curve, in order to find with accuracy the laser damage threshold of the sample. This second test is made by testing 100 points for each energy (with 10 different energies). Finally the laser damage measurement is the result of a statistic made on 2000 different measurement points for each sample.

3.2. Laser damage measurements

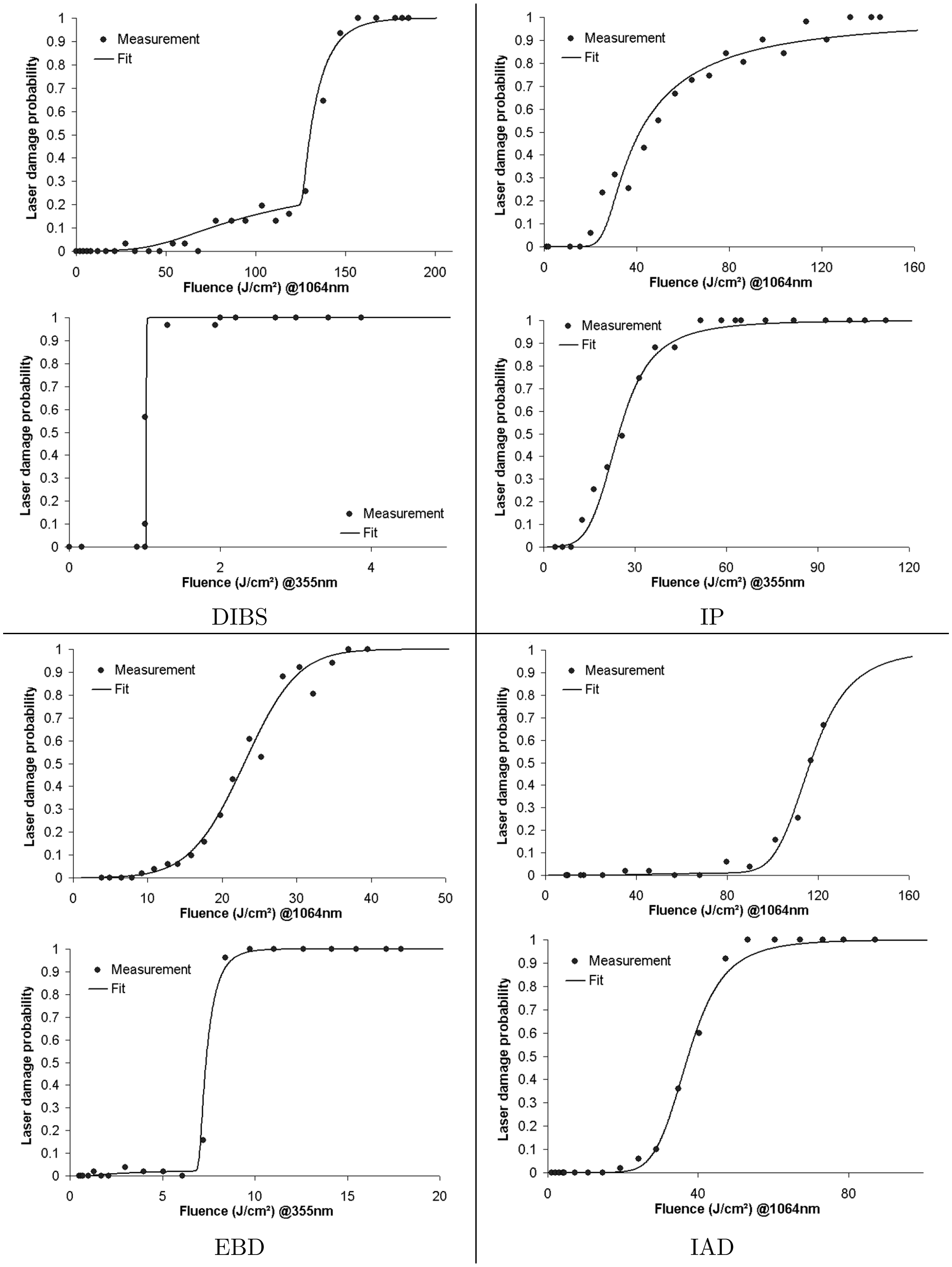
We have plotted on figure 1, different curves representative of each kind of samples and wavelength. Obviously, each kind of sample exhibit a different behavior as regards the curve shape and the threshold. A difference can also appear between samples of different thicknesses : this is not shown on the graphs but it will be discussed below. The shape and the threshold are linked to the initiator characteristics (density, nature,...), that can be obtained by fitting the experimental curves with an adapted model (see ref[10]). This has been done on each curve in this study and the result is shown on the curves in plain line in the figures. A very good agreement has been obtained between our data and the model, evidencing different randomly distributed initiators on each samples. The results obtained from this analysis are the following :

- On the DIBS samples, at 1064nm we observe two types of laser damage precursors : one with a threshold of 30 $\pm 5J/cm^2$ and density of $600 \pm 200/mm^2$ and the other of 110 $\pm 20J/cm^2$, with a higher density of $5 \pm 2 \times 10^4/mm^2$. We observe no difference in the threshold or number of the precursors between the samples of different thicknesses, which means that for these samples the precursors could be interface defects (as opposed to defects embedded in the bulk of the layer)
  At 355nm however there is a threshold value for laser damage and no distribution of the laser damage probability. Then in this case the thin film must be absorbing (threshold is also very low) and ablated at the threshold. This is correlated to the morphology observed: a small pit in the center of the beam.

- As concerns the EBD samples, we observe at 1064nm one kind of precursor with a threshold of 12 $\pm 2J/cm^2$ and with a density depending of the thin film thickness : $1 \pm .5 \times 10^4/mm^2$ for the 200nm samples and $5 \pm 1 \times 10^4/mm^2$ for the 1000nm samples. From these results it comes out that the number of defects for this kind of technique is proportional to the film thickness. It is good indication that they must be embedded inside the bulk of the film all along the deposition process.
  At 355nm two kinds of initiators appears : the first appears at 4 $\pm 1J/cm^2$ with a distribution lower than 40 defects by mm^2, and the other at 6 $\pm 1J/cm^2$, but it is the first kind that is limiting the laser damage resistance.

- On the IP samples we detect one kind on precursors on the 200nm thickness samples and two kind of precursors on the 1000nm samples at 1064nm. The corresponding threshold and densities for the 200nm samples are 20 $\pm 2J/cm^2$, $7 \times 10^3/mm^2$. The 1000nm samples where found to be less resistant to laser damage, with defects having a threshold of 10 $\pm 1J/cm^2$ (density = $300/mm^2$) and 12 $\pm 1J/cm^2$ (density = $2 \times 10^4/mm^2$). We can note that if we do not see two kind of precursors for the 200nm samples, it could be due to the limitation of the technique : defect densities less than $100/mm^2$ are hard to detect with the sot size used. Nevertheless, for this deposition technique, the dependance with thickness suggests a production of these defects along the deposition process.
  In the UV the same defects (threshold of 10 $\pm 1J/cm^2$ and density of $9 \times 10^3/mm^2$) were found on all the samples.

- The IAD samples have presented the same behavior at 1064nm: two kind of defects, one with a threshold of 100 $\pm 10J/cm^2$ and with a density of $3 \times 10^4/mm^2$ the other with a lower density ($400/mm^2$) and a threshold of 30 $\pm 3J/cm^2$. However in the UV, a difference appears between samples of 200nm and 1000nm due to very low distributed defects that appears in the thickest layers. These coatings have a very high laser damage threshold at 355nm (the substrate value is almost reached for the 200nm samples).



DIBS

IP

EBD

IAD

Figure 1: Measurement and fit of laser damage probability curves of thin film tested at 1064 and 355nm.

3.3. Comparison of laser damage resistance

To summarize, the low damage threshold (lower fluence where a damage is observed) is given in figure 2 and 3.

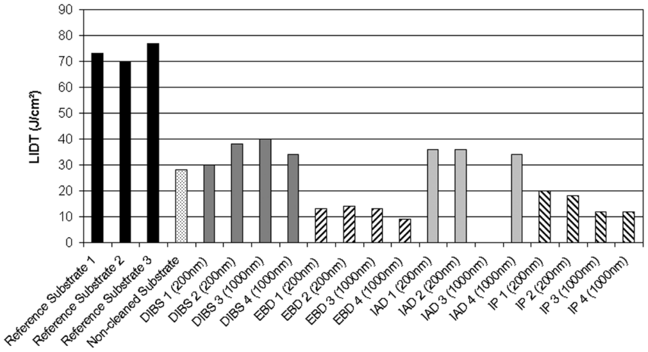


Figure 2: Laser induced damage threshold of thin films and substrates tested at 1064nn, 5ns.

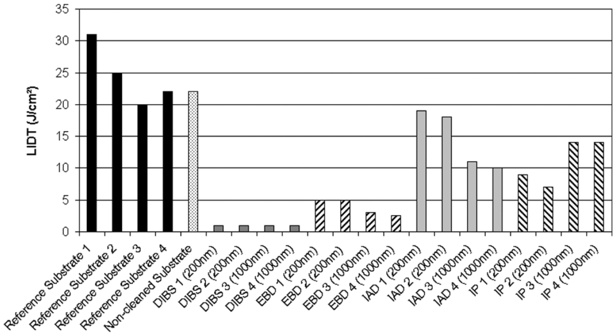


Figure 3: Laser induced damage threshold of thin films and substrates tested at 355nn, 5ns.

4. ANALYSIS OF THE LASER DAMAGE MECHANISM

To go further in our investigation, we have made *post mortem* measurements that can give information on the origin and mechanism of laser damage in our samples.

4.1. Initiation

Microscope measurements (Dark field and Nomarski) were done on each samples. These measurements clearly confirm the mechanism of initiation by randomly distributed defects, as observed with the laser damage probability curves. Indeed, as we can see on figure 4 where we plot the laser damage morphology observed by Nomarski microscope, the final aspect of the damage shows different deep pits corresponding to what we assume to be the localization of the initiators, and a shallow print of the laser spot in the layer. The damage in these case is certainly the result of an initiation by precursors under the laser spot, then due to a plasma formation in the air, the surface has been heated and ablated under the spot. We can note that the initiation do not always occur where the fluence is maximum (center of the spot), and that the size of the pits is different depending of the technique (the initiators and the layer properties are certainly different).

4.2. Material modification

To finish, we made measurements on the modified material with two diagnostics available at the Institut Fresnel and devoted to laser damage studies. The first one is a photothermal microscope, based on the photothermal deflection technique, that allows measurement of small absorption at 1064nm with a micronic resolution (details

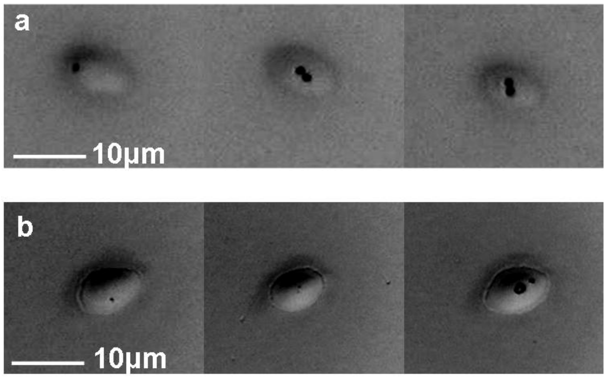


Figure 4. 1064 nm Laser damage morphologies observed by Nomarski microscopy on EBD (a) and DIBS (b) thin films.

in ref[11]). The second one is an apparatus that allows simultaneous absorption, scattering and luminescence mappings of surfaces with the used of a 244nm pump beam (details in ref[12]).We present on figure 5 an analysis of a damage on a SiO_2 thin film that has been created near the low damage threshold.

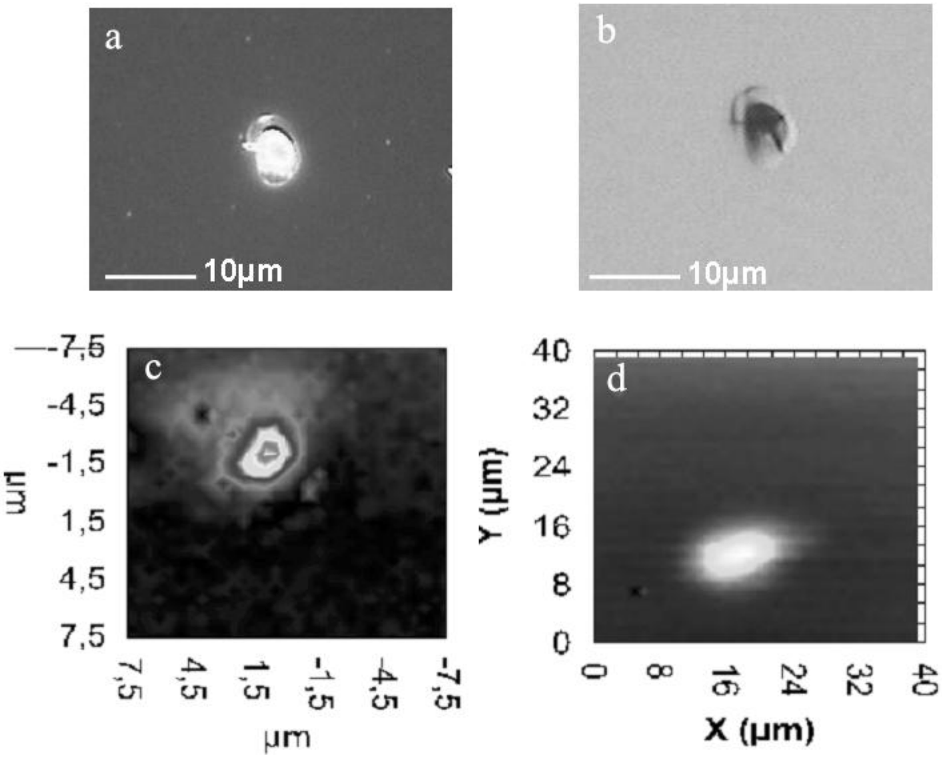


Figure 5. Observation of the material modification on a laser damage. a: dark field microscopy, b: Nomarski microscopy, c: 1064nm absorption mapping, d: luminescence mapping (excitation at 244nm, and integration of the signal in the visible).

We observe on this damage that the surrounding material has been strongly modified by the thermomechanical processes involved : the silica is now absorbing and present some luminescence in the visible. These measurements are in correlation with previous works[13, 14] where it has been showed that the damage mechanism in silica thin films could be initiated by nanoscale absorbing defects. The damage process in this case starts with absorption by the particle, then the energy is transferred during the pulse from the heated defect to the surrounding matrix, causing the conversion of SiO2 into an absorbing medium, which leads to a macroscopic damage.

We can note that the same behaviors have been observed on all the samples made with the different depositions techniques.

To finish there is not doubt that subsequent shots on this location where the material is absorbing will induce some growth of the damage.

5. CONCLUSION

We have measured and analyzed the laser damage resistance of silica coatings made with different conventional deposition techniques. At 1064nm, the DIBS and IAD samples have shown high resistance to laser damage, and the IAD and IP samples were found to have very high laser damage threshold et 355nm.

We have shown that damage was initiated by different kind of defects depending of the deposition technique. The initiation mechanism has been analyzed with specific tools and we have been able to highlight different steps of the damage process.

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