Influence of polishing and cleaning on the laser-induced damage threshold of substrates and coatings at 1064 nm

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Abstract. Laser-induced damage threshold (LIDT) results on silica substrates and coatings are presented for near-infrared applications. Different polishing and cleaning processes are evaluated. In particular, we investigate the influence of polishing and cleaning on the LIDT of silica substrates and optical coatings deposited by dual-ion-beam sputtering. Laser damage tests were performed at 1064 nm with a 5-ns-pulse Nd:YAG laser, and experiments were made on surfaces of optical components using a 12-μm-diameter focused beam. Accurate damage probability curves are plotted thanks to a reliable statistical measurement of laser damage. Use of a statistical model permits us to deduce the densities of laser damage precursors. We find a significant improvement of the LIDT of our coatings on choosing the appropriate cleaning and polishing process.
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Subject terms: laser-induced damage; polishing; cleaning; SiO_2; Ta_2O_5; antireflection coating.

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1 Introduction

Laser-induced damage in optical components remains a key problem for high-power optics. Due to interaction of photons with dielectric materials, electromagnetic energy can be converted to electronic, thermal, chemical, and mechanical energy, and lead to irreversible degradation of optical properties (transmission, reflection, absorption), or *damage*. Thus much attention has been paid, during the last 30 years, to laser-induced damage in optical components, especially with regard to the study of fundamental mechanisms of damage initiation and growth.[1] It is today widely accepted that material breakdown of large-bandgap transparent insulators irradiated by nanosecond-pulse lasers is often linked to the presence of nanosized defects, located at surfaces and interfaces or in the bulk of coatings and substrates. Different theoretical and experimental studies have confirmed that in the nanosecond regime, absorbing nanometer-sized particles could be responsible for the initiation of the damage process.[2–6] In most cases these defects are not identified, since they themselves may be on the nanoscale and be distributed at low concentration. In the case of surface damage, defects could be contaminants coming from the steps of polishing[7–10] and cleaning, and also from the deposition techniques and materials involved in the coatings.[11–13]

One way to obtain information about these defects, also referred as nanoprecursors, is to plot laser damage probability curves. Indeed, the damage probability is linked to the probability of the presence of nanoprecursors under the irradiation laser beam, and depends on their physical characteristics: threshold and density. This probability can be expressed as a function of fluence, thanks to a statistical model[14] that permits one to fit the curves and to extract useful information about nanoprecursors: the number of nanoprecursor classes and the density and threshold for each class. This analysis tool allows us to compare precursors involved in the different processes (polishing and cleaning) and to estimate their real influence on laser-damage resistance.

First we describe the experimental setup that permits acquisition of accurate probability curves at 1064 nm with small spot size, and then we describe the data analysis method. In Sec. 3, the influence of polishing and cleaning on the laser damage resistance of bare silica substrates is studied. In Sec. 4, the influence of polishing on the laser damage resistance of single and multilayer coatings at 1064 nm is presented.

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2 Experimental Setup and Analysis Method

The apparatus used for laser-damage testing has been described in detail in another paper,[15] and only a brief description is given here. The setup involves a YAG laser beam with 1064-nm wavelength and 5-ns pulse duration. The beam is focused down to a spot diameter of 12 μm (at $1/e^2$) on the sample. The energy of the incident beam is measured with a pyroelectric detector. The spatial profile of the focused beam is analyzed with an optical system linked to a CCD camera, and the temporal profile is measured with a fast photodiode.

Samples are observed through an *in situ* optical microscope (magnification, 50× to 1000×), used in Nomarski mode.[15] Laser damage is defined as an irreversible modification of the sample observed through the microscope. An image of the irradiated zone is acquired before and after each shot, using a CCD camera connected to a computer. Then the difference between the two images is turned into a binary image. The damage criterion is defined as a given number of white pixels. A resolution better than 1 μm is reached.

The damage test procedure[16] 1-on-1 is used to measure laser damage probability curves on our samples. We obtain these curves by counting the number of damaged regions at each fluence F in order to estimate the probability curve $P(F)$. Each curve $P(F)$ is plotted with 1000 data points that involve 20 different fluences and 50 tested regions at each fluence.

We link the probability of damage, $P(F)$, to the probability of presence of a defect that receives more energy density than its intrinsic threshold. This probability is given by a Poisson law and can be expressed as a function of fluence F (the energy per unit of surface):

$$P(F) = 1 - \exp\left[- \int_0^F g(T) \cdot \frac{\pi w^2}{2} \ln\left(\frac{F}{T}\right) \mathrm{d}T \right] \quad (1)$$

with w the laser spot radius at the laser waist (half width at $1/e^2$). A Gaussian distribution $g(T)$ of nanoprecursor thresholds is considered,[14] taking into account that all defects of a given class do not fail at the same fluence due for example to a possible size and absorption distributions:

$$g(T) = \frac{2\sqrt{2} \cdot d}{\Delta T \cdot \sqrt{\pi}} \exp\left[- \frac{1}{2}\left(\frac{T - T_0}{\Delta T/2}\right)^2 \right] \quad (2)$$

The ensemble function $g(T)$ depends on three parameters: the threshold mean value T_0, the threshold standard deviation ΔT (full width at $1/e^2$), and the defect density d. The relationship between $g(T)$ and defect density d is obtained from the normalization condition

$$\int_0^\infty g(T)\, \mathrm{d}T = d. \quad (3)$$

This model allows us to fit laser damage probability curves and to extract information such as the number of nanoprecursor classes and the densities d and thresholds T_0 of nanoprecursors for each class. The accuracy is ±20% for the nanoprecursor density d, and ±5% for the threshold T_0.

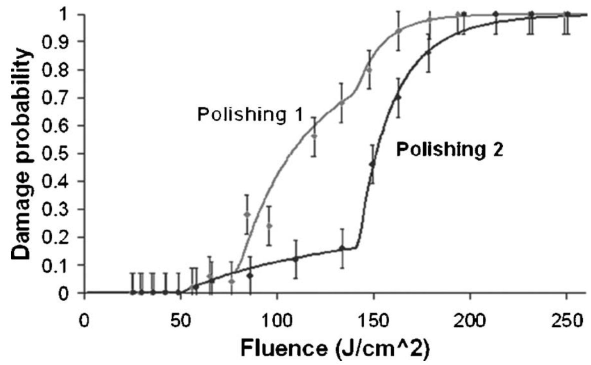

Fig. 1 Influence of polishing on laser damage resistance of a silica substrate.

The laser-induced damage threshold (LIDT) of the component is defined as the greatest fluence for which the measured probability $P(F)=0$.

3 Influence of Polishing and Cleaning on the Laser Damage Resistance of Bare Silica Substrates

The fabrication steps of optical coatings are clearly involved in the laser damage resistance of a component.[10–13] Our aim is to optimize the surface quality of silica substrates before coating deposition. Thus the influence of two polishing processes and three types of cleaning on the laser damage resistance of an Infrasil® silica substrate is particularly studied. We prepared one sample for each combination of polishing and cleaning processes. Samples are transported in special boxes to avoid any contamination. Then all laser damage measurements are made under laminar flow. This systematic analysis permits us then to determine the best polishing and cleaning processes for better laser damage resistance of the final component.

First, we compare two samples cleaned with the same cleaning procedure (cleaning 3 in our study) but polished according to two different processes. Laser damage probability curves are plotted in Fig. 1, and information about nanoprecursors (densities and thresholds for each class) is given in Table 1. For polishing process 1, we observe three classes of nanoprecursors, whereas for polishing 2, only two classes are present. The comparison of densities and

Table 1 Comparison of nanoprecursors classes versus polishing processes.

	Polishing 1	Polishing 2
T_1=LIDT	49 J/cm^2	49 J/cm^2
d_1	3×10^3 mm^{-2}	3×10^3 mm^{-2}
T_2	80 J/cm^2	—
d_2	3.4×10^4 mm^{-2}	—
T_3	141 J/cm^2	142 J/cm^2
d_3	1.5×10^5 mm^{-2}	1.4×10^5 mm^{-2}

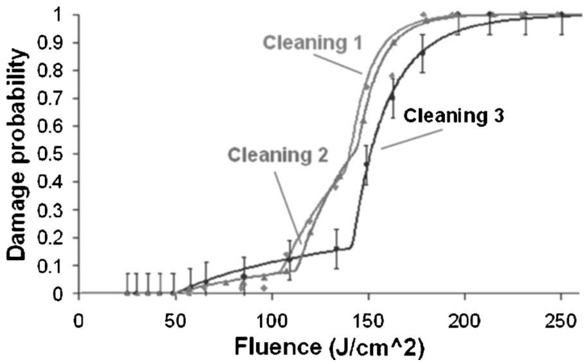

Fig. 2 Influence of cleaning on laser damage resistance of a silica substrate.

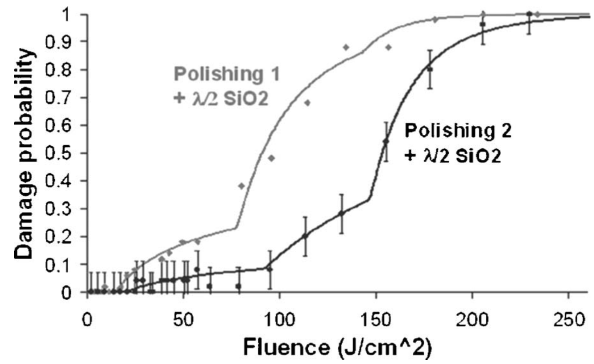

Fig. 3 Influence of polishing on laser damage resistance of SiO_2 single thin films deposited on silica substrate.

thresholds for each class (Table 1) shows that the first and third classes of nanoprecursors are the same for the two polishing processes and that the second class of nanoprecursors does not exist for polishing 2. Even if the LIDT remains the same (49 J/cm^2), we improve the general laser damage resistance of silica substrate with polishing 2. For example, if we use this silica substrate with a fluence of 140 J/cm^2, in case of polishing 1 we have a 70% probability of damage, whereas in the case of polishing 2 we have only 20%. The same measurements for cleanings 1 and 2 show that whatever the cleaning process, polishing 2 is always better than polishing 1.

Then, we compare two samples polished with the same best polishing procedure (polishing 2) but cleaned according to three different processes: manual wiping with acetone (cleaning 1), and two automatic aqueous procedures involving ultrasonic immersion and detergents followed by deionized water rinsing and drying (cleanings 2 and 3). Laser damage probability curves are plotted in Fig. 2, and information about nanoprecursors (densities and thresholds for each class) are given in Table 2. For cleaning processes 1 and 2, there are three classes of nanoprecursors, whereas for cleaning 3, only two classes of nanoprecursors are present. The comparison of densities and thresholds for each class (Table 2) shows that the first and third classes of nanoprecursors are present whatever the cleaning process and that the second class of nanoprecursors has been removed with cleaning process 3. Even if the LIDT still remains the same (49 J/cm^2), we improve the general laser damage resistance of silica substrates with cleaning 3.

This study of the first fabrication steps (polishing and cleaning) permits us to improve the laser damage resistance of Infrasil silica substrates by choosing the appropriate procedure. It highlights also that polishing and cleaning have to be studied together. For all samples, however, the first class of nanoprecursors ($T=49$ J/cm^2, $d\approx 3\times 10^3$ mm^{-2}) is always present and limits improvement of the LIDT. Better polishing and cleaning processes have to be set up in order to decrease the influence of this first class of nanoprecursors and to considerably increase the LIDT.

4 Influence of Polishing on the Laser Damage Resistance of Coatings at 1064 nm

We have shown that polishing and cleaning have an influence on the laser damage resistance of silica substrates. Our aim is now to evaluate the influence of polishing on the laser damage resistance of optical coatings at 1064 nm. The same type of Infrasil silica substrates is used, and all samples are cleaned with best process cleaning. Three types of coatings were realized with the dual ion-beam sputtering (DIBS) technique: silicon dioxide (SiO_2) single thin films, tantalum pentoxide (Ta_2O_5) single thin films, and Ta_2O_5/SiO_2 antireflection coatings. The optical thickness of the single thin films was λ/2 at 1064 nm in order to avoid any influence of electric field. The antireflection coating was a two-layer design with $R<10^{-4}$ at 1064 nm. For

Table 2 Comparison of nanoprecursor classes versus cleaning processes.

	Cleaning 1	Cleaning 2	Cleaning 3
T_1=LIDT	50 J/cm^2	50 J/cm^2	49 J/cm^2
d_1	1.8×10^3 mm^{-2}	1.8×10^3 mm^{-2}	3×10^3 mm^{-2}
T_2	105 J/cm^2	113 J/cm^2	—
d_2	3×10^4 mm^{-2}	4.5×10^4 mm^{-2}	—
T_3	140 J/cm^2	145 J/cm^2	142 J/cm^2
d_3	2×10^5 mm^{-2}	1.8×10^5 mm^{-2}	1.4×10^5 mm^{-2}

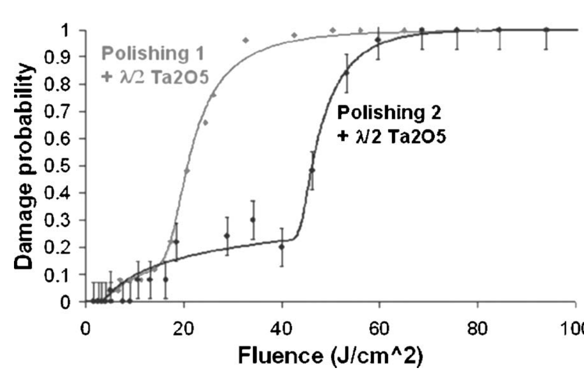

Fig. 4 Influence of polishing on laser damage resistance of Ta_2O_5 single thin films deposited on silica substrate.

Table 3 Comparison of nanoprecursor classes of Ta_2O_5 single thin films deposited on silica substrate versus polishing processes.

	Polishing 1	Polishing 2
T_1=LIDT	5 J/cm^2	5 J/cm^2
d_1	1.7×10^3 mm^{-2}	1.9×10^3 mm^{-2}
T_2	18 J/cm^2	44 J/cm^2
d_2	6×10^4 mm^{-2}	1.5×10^5 mm^{-2}

each coating we realized two samples, obtained with different polishing processes (polishings 1 and 2). Then laser-induced damage measurements were made for the six different components.

First we compare the LIDTs of two SiO_2 single thin films deposited on a silica substrate. Laser damage probability curves are plotted in Fig. 3. With polishing 1, the LIDT of the SiO_2 single thin film is 9 J/cm^2, whereas with polishing 2 it increases to 24 J/cm^2. Use of a judicious polishing process permits an increase of the coating LIDT by a factor 2.6. This result shows that the quality of the interface between substrate and coating is critical for laser damage resistance of SiO_2 films.

Then we compare the LIDTs of two Ta_2O_5 single thin films deposited on the same type of silica substrate. Laser damage probability curves are plotted in Fig. 4, and information about nanoprecursors (densities and thresholds for each class) is given in Table 3. We notice that the first class of nanoprecursors is the same for the two samples, whereas the threshold of the second nanoprecursors class increases by a factor 2.4. In Ta_2O_5 films, nanoprecursors that limit the LIDT seem to be intrinsic to the material and not related to the substrate-film interface quality. However, a better polishing process improves the general laser damage resistance of Ta_2O_5 single thin films. Indeed, if we use this coating with a fluence of 40 J/cm^2, in the case of polishing 1 the damage probability is 100%, whereas in the case of polishing 2 it is only 20%.

The laser-induced damage measurement of the two-layer Ta_2O_5/SiO_2 antireflection coatings permits us to estimate the influence of some fabrication steps on multilayer coatings (Fig. 5 and Table 4). First, we notice that LIDT of the multilayer component is limited by the high-index material. Indeed, the LIDT of the antireflection coating is 6.5 J/cm^2, and that of the high-index material is 5 J/cm^2. According to the overall behavior of the curves, we can also note that the polishing process has a determining influence on the laser damage resistance of multilayer coatings.

Table 4 Comparison of nanoprecursors classes of an antireflection coating deposited on silica substrate versus polishing processes.

	Polishing 1	Polishing 2
T_1=LIDT	6.5 J/cm^2	6.5 J/cm^2
d_1	200 mm^{-2}	200 mm^{-2}
T_2	30 J/cm^2	—
d_2	5×10^3 mm^{-2}	—
T_3	51 J/cm^2	50 J/cm^2
d_3	1.8×10^5 mm^{-2}	1.4×10^5 mm^{-2}

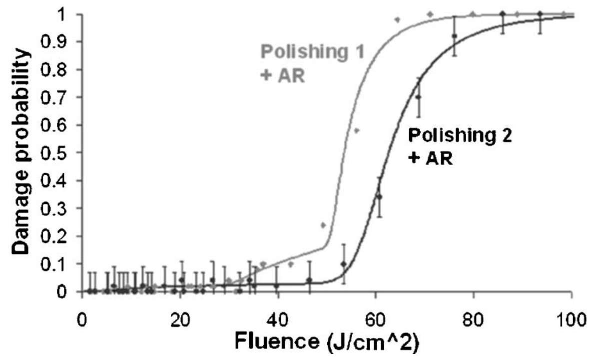

Fig. 5 Influence of polishing on laser damage resistance of Ta_2O_5/SiO_2 antireflection coatings.

5 Conclusion

We have presented a detailed analysis of the most important steps involved in manufacturing multilayer coatings. The choice of polishing process linked to an appropriate cleaning procedure is critical. Indeed, the LIDT of a SiO_2 single thin film can vary from 9 to 24 J/cm^2 according to the procedure chosen. Laser damage measurement on a Ta_2O_5 single thin film deposited on a silica substrate shows that it is responsible for a limitation of the LIDT, as it brings nanoprecursors with a lower threshold than those from the substrate surface.

The behavior of an antireflection coating under a high-power laser is linked to steps of fabrication. To build multilayer components with high laser-induced damage threshold, improvement in fabrication steps has a great role to play.

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