

Catastrophic Failure of Contaminated Fused Silica Optics at 355 nm

**F. Y. Génin
M. R. Kozlowski
R. Brusasco**

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**Lawrence
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Catastrophic failure of contaminated fused silica optics at 355 nm

F. Y. Génin^{a)}, M. R. Kozlowski, and R. Brusasco,

Lawrence Livermore National Laboratory,
Laser Materials Department, Livermore, California 94550

ABSTRACT

For years, contamination has been known to degrade the performance of optics and to sometimes initiate laser-induced damage to initiate. This study has started to quantify these effects for fused silica windows used at 355 nm. Contamination particles (Al, Cu, TiO₂, and ZrO₂) were artificially deposited onto the surface and damage tests were conducted with a 3 ns Nd:YAG laser. The damage morphology was characterized by Nomarski optical microscopy. The results showed that the damage morphology for input and output surface contamination is different. For input surface contamination, both input and output surfaces can damage. In particular, the particle can induce pitting or drilling of the surface where the beam exits. Such damage usually grows catastrophically. Output surface contamination is usually ablated away on the first shot but can also induce catastrophic damage. Plasmas are observed during illumination and seem to play an important role in the damage mechanism.

The relationship between fluence and contamination size for which catastrophic damage occurred was plotted for different contamination materials. The results show that particles even as small as 10 μm can substantially decrease the damage threshold of the window and that metallic particles on the input surface have a more negative effect than oxide particles.

Keywords: Surface contamination, laser-induced damage, damage morphology, fused silica, 355 nm.

1. INTRODUCTION

The development and construction of high fluence lasers for inertial confinement fusion such as the National Ignition Facility (NIF) at Lawrence Livermore National Laboratory (LLNL) or the Laser Mégajoule (LMJ) in France continues to generate strong interest in the behavior of optical components under intense laser illumination. The design of such lasers has created significant technological challenges in the area of laser glass, KDP crystal growth, surface finishing and 355 nm fused silica damage. The damage of fused silica lenses at 355 nm is of particular concern since designers have pushed the peak fluence to about 14 J/cm², a level which is close to the damage threshold of the best fused silica samples available today.

It is well known that contamination particles falling onto optical surfaces will aggravate laser damage problems.¹⁻⁴ The effect of contaminants on the onset of damage must therefore be characterized and quantified to predict the survivability of optics on the beam line. The results will allow us to set cleanliness requirements so as to prevent or delay damage and avoid catastrophic events caused by the failure of critical optical components. It will also determine the risk level of damage if contamination is not sufficiently controlled.

While experience on existing lasers such as the Nova (LLNL) and the Omega (University of Rochester) lasers have shown that contamination can lower the damage threshold of optical components, very little has been done to quantify these effects and better identify the important parameters. Damage of transmissive optics on Nova, for example, has mostly been found on output surfaces. It was commonly believed that contamination on the output surface of lenses (i. e. the surface where the beam exits the

^{a)} Electronic Mail: fgenin@llnl.gov

window) caused such damage. This study showed that input surface contamination can also initiate output surface damage and that it can in fact have even a more negative effect than output surface contamination.

These important findings have been obtained from the initial experiments from a matrix of variables to be investigated that includes contamination material (metals, oxides, organics), particle size, shape and mass, substrate material (fused silica, phosphate glass, KDP, multilayer mirrors and polarizers), wavelength, environment (air, vacuum), fluence level and number of shots. An article published recently presented the results of the study of aluminum particles on fused silica irradiated in air at 1064 nm and 355 nm.⁵ The study was performed for contamination on both input and output surfaces of the window (see Fig. 1). In order to control the size and shape of the contamination particles, 1 μm thick dots of sizes ranging from 10 μm to 250 μm were sputter-deposited onto the silica. Shavings of pure material deposited onto the silica were also studied; the results proved to be very reproducible and showed that the behavior of dots and shavings are qualitatively the same.

In the article presented here, the method to study contamination effects is the same. The tests concentrate on damage during repetitive illumination at 355 nm in air of aluminum, copper, titania and zirconia contamination particles on bare fused silica surfaces. The discussion in this article is mainly focused on damage morphologies. To clarify these discussions, damage was classified into two categories: *massive* and *catastrophic*. Massive damage refers to damage which will affect the performance of the optic beyond NIF tolerance limits but is stable on subsequent shots at the same fluence. Catastrophic damage (also named massive unstable) refers to the case where the glass begins to crack or be ablated during repetitive illumination. Since damage during these experiments was quite severe, the article will be centered on the relationship between contamination size and fluence levels inducing catastrophic damage.

2. EXPERIMENTAL PROCEDURE

The experiments were conducted in the following sequence:

- preparation of silicon nitride membranes with circular openings to be used as masks; six different circle sizes were etched: 10, 20, 30, 50, 150 μm and 250 μm ,
- deposition of the contaminant by sputtering aluminum, copper, titania or zirconia through the masks onto the fused silica substrates,
- laser damage testing at 355 nm in air of the dot particles on the substrates,
- characterization of the laser-induced damage after each shot by Nomarski optical microscopy; the tests were terminated when damage grew catastrophically.

The experimental procedure to prepare the silicon nitride masks and the sputtered dots is reported elsewhere.⁵ The thickness of the sputtered deposit was 1 μm . Six particle sizes were investigated: 10, 20, 30, 50, 150, and 250 μm .

The glass substrates chosen for this study were Corning 7980 Zygo superpolished fused silica. The damage threshold of the surface of the fused silica samples was tested to establish the baseline; the threshold of the uncontaminated surface were above 15 J/cm² at 355 nm. The thickness of the substrates was 11 mm and had a diameter of 50.5 mm.

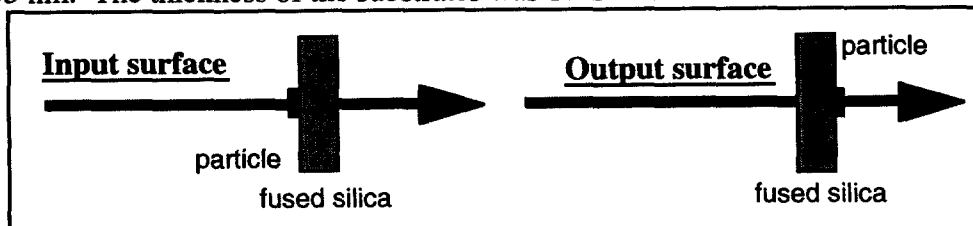


Fig. 1: Schematic diagram of the testing set-up for contamination particles on the input and the output surfaces.

The laser damage tests were carried out using a 355 nm, 3-ns pulse from a Nd:YAG laser. The laser was focused to provide a far field circular Gaussian beam with a diameter of about 1 mm at $1/e^2$ of the maximum intensity. The tests were conducted in s-polarization at near-normal incidence and the fluence varied between 4 and 17 J/cm². The test sites were examined before and after irradiation by Nomarski and back light microscopy.

3. RESULTS

The method used during the tests provided very reproducible damage morphologies.⁵ This section will present these damage morphologies for aluminum, copper, titania and zirconia contaminants on input and output surfaces. The typical damage morphologies on the input surface for these materials are shown in Figs. 2 - 6. Figure 7 summarizes the relationship between fluence and input surface contamination size for the various materials. Figure 8 shows the damage morphologies for output surface contamination and Fig. 9 is the figure analogous to Fig. 7 for output contamination. The materials chosen for the study are typical metals and oxides that contaminate the optics. For comparable particle sizes, metals triggered catastrophic damage at lower fluences. The behavior of shavings deposited on silica (which will not be reported here) was found to be very similar to that of the sputtered particles,⁵ even though the adhesion properties are significantly different.

3.1 Input surface contamination:

Damage can initiate at two locations for input surface contamination: at and around the particle on the input surface but also on the output surface of the window. During the first pulse, a plasma ignites at the particle (above a fluence of about 1.5 J/cm²). Metallic particles often melt and can re-deposit in the surroundings. Oxide particles often break up into small pieces. Each subsequent pulse can then interact with the damaged zone until plasmas no longer ignite at which point damage is usually "stable". For the input surface, the morphology is that of a scalded surface with a large number of small craters (see Figs. 2a) - 5a)). The damage morphology shown in these figures is referred as to massive (and not catastrophic) because such damage, although quite significant in size, eventually stops propagating during repetitive illumination.

The output surface of the window can also be damaged. A plasma can often be observed at that location. In particular, at fluences above 5 J/cm², a print of the shape of the input surface contamination particle is sometimes recognizable on the output surface (see Figs. 2 and 6). For fluences higher than a given threshold (which varies with particle size), the exit surface is strongly attacked. The damage morphology is that of pits (see Fig. 2b) - 5b)). These pits on the output surface grow very quickly during repetitive illumination by drilling upstream of the light into the bulk; such damage is referred as to catastrophic. The damaged region seems to couple to the light easily and the fused silica is further damaged after each shot.

It was determined that output surface damage occurs as a result of diffraction modulation caused by the obscuration on the input surface. In short, the particle on the input surface produces an obscuration. The light is diffracted and a pattern with the shape of the particle can propagate downstream. This effect is often referred as to the Arago effect.⁶ Some preliminary modeling on this effect and on the interaction of plasmas with the substrate during damage is reported in these proceedings.⁷ The full analysis of the experimental results related to these effects will be published in the future.

The relationship between fluence and input surface contamination size that induce catastrophic damage is plotted on a damage map in Fig. 7. The results show that for a given size, the contamination material has a strong influence on the fluence level that can induce catastrophic failure. This map represent a useful tool to determine the size at which contamination must be controlled on the laser and which types of materials to avoid. In this case, input surface contamination was found to have the potential for inducing catastrophic failure of the glass at fluences as low as 5 J/cm² for the 250 μ m copper particles. The maps

can explain the reason why lasers such as Nova designed to operate under 4 J/cm^2 did not suffer major damage incidents even in the absence of tight cleanliness requirements. However, they show that contamination problems will have to be addressed very seriously on the NIF and LMJ lasers for which the 355 nm peak design fluence is 14 J/cm^2 .

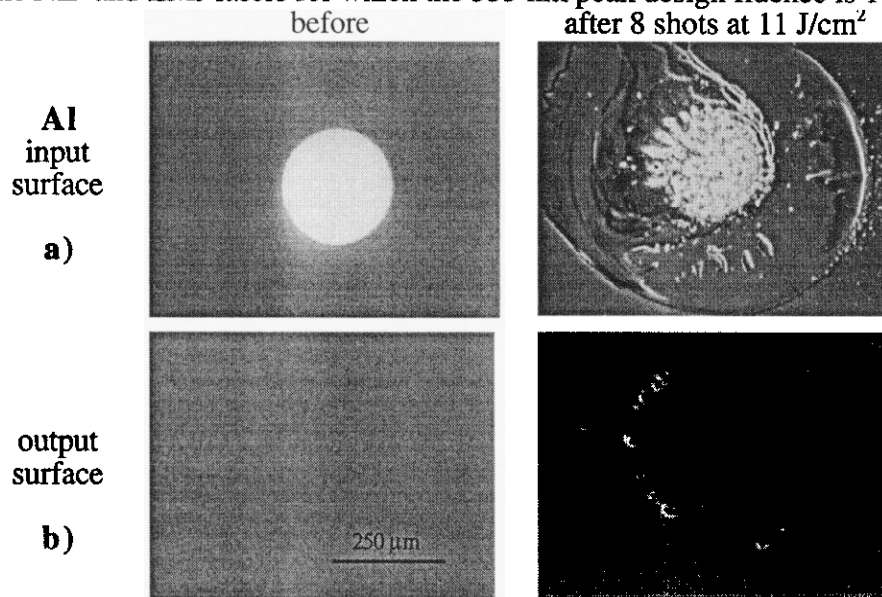


Fig. 2: Nomarski optical micrograph of the damage morphology of a) the input and b) output surfaces of the fused silica window after 8 shots at 355 nm and 11 J/cm^2 . The damage was initiated by a sputtered Al particle located on the *input surface*. The particle was $1 \mu\text{m}$ thick and $250 \mu\text{m}$ in diameter. The input surface damage is massive. The output surface damage would have grown catastrophically during subsequent shots. The output surface damage follows the outline of the input surface particle.

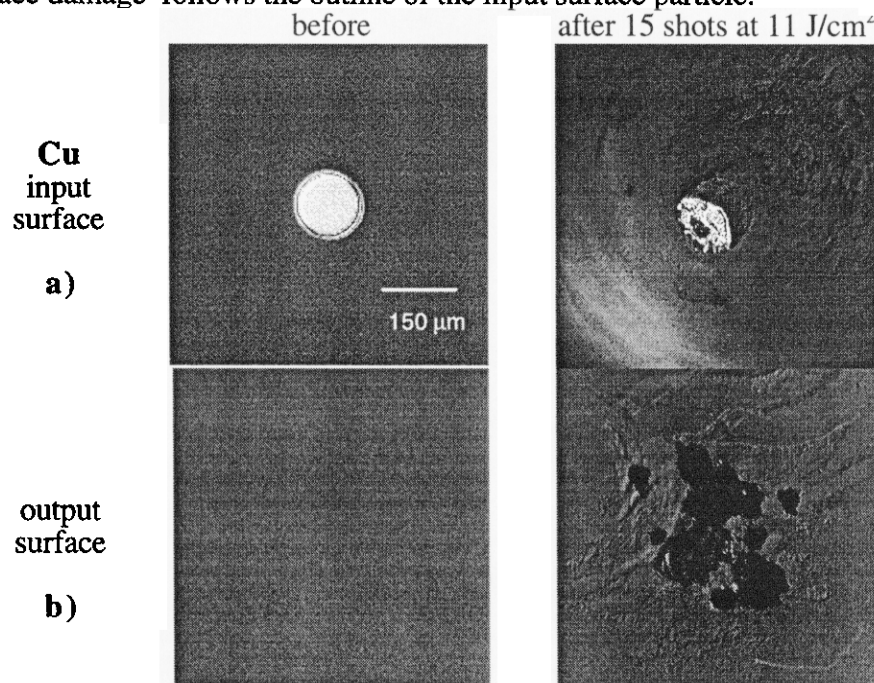


Fig. 3: Nomarski optical micrograph of the damage morphology of a) the input and b) output surfaces of the fused silica window after 15 shots at 355 nm and 11 J/cm^2 . The damage was initiated by a sputtered Cu particle located on the *input surface*. The particle was $1 \mu\text{m}$ thick and $150 \mu\text{m}$ in diameter. The output surface damage is catastrophic.

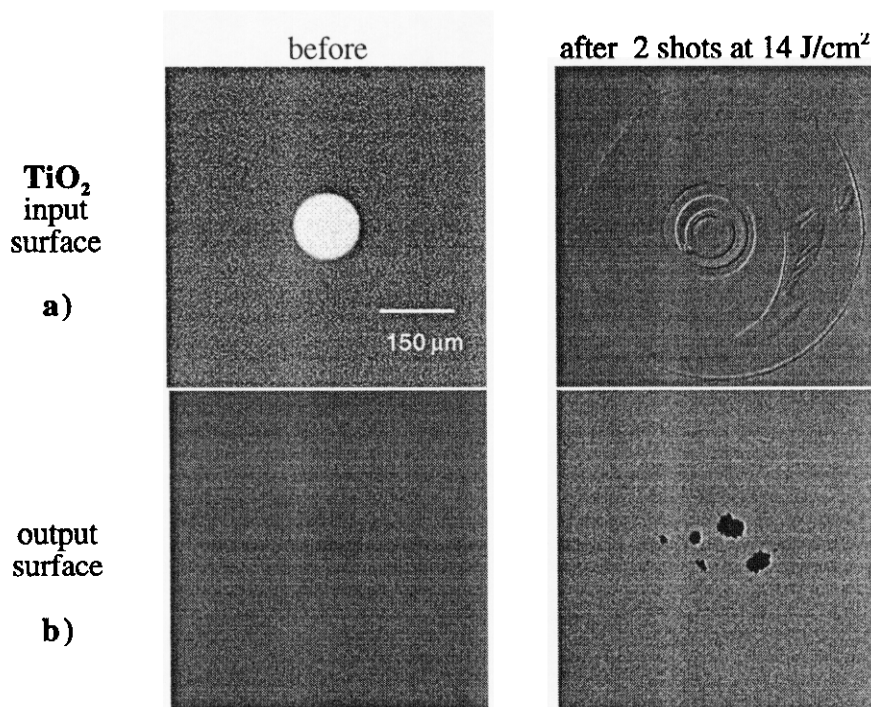


Fig. 4: Nomarski optical micrograph of the damage morphology of a) the input and b) output surfaces of the fused silica window after 2 shots at 355 nm and 14 J/cm². The damage was initiated by a sputtered TiO₂ particle located on the *input surface*. The particle was 1 μm thick and 150 μm in diameter. The output surface damage is catastrophic.

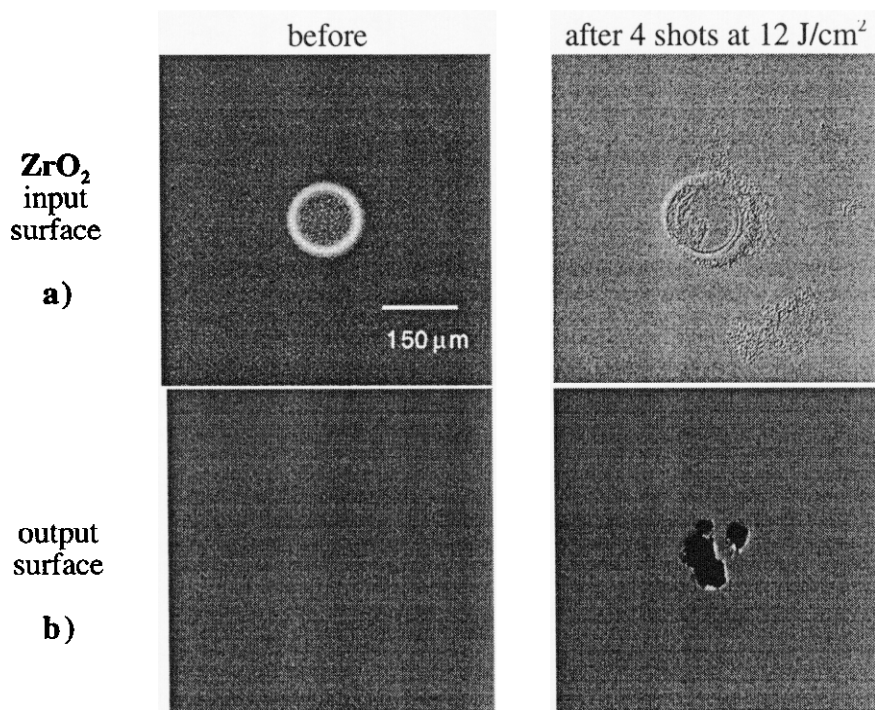


Fig. 5: Nomarski optical micrograph of the typical damage morphology of the input and output surfaces of the fused silica window after 4 shots at 355 nm and 12 J/cm². The damage was initiated by a sputtered ZrO₂ particle located on the *input surface*. The particle was 1 μm thick and 150 μm in diameter. The output surface damage is catastrophic.

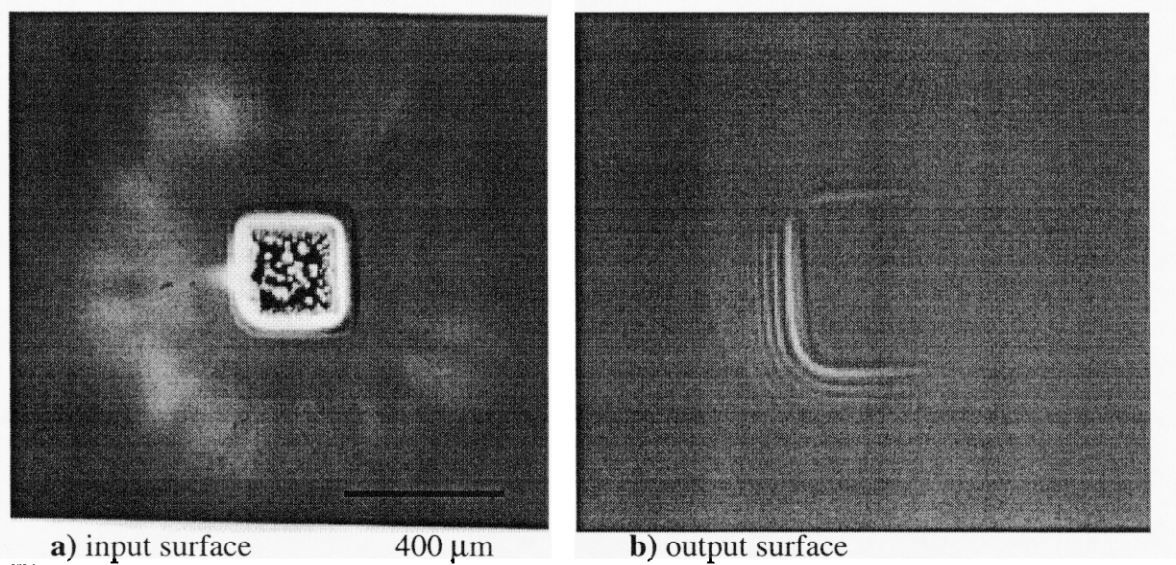


Fig. 6: Nomarski optical micrograph of the typical damage morphology of the fused silica a) input and b) output surface after 1 shot at 355 nm and 12.9 J/cm^2 . The damage was initiated by a square particle ($1 \mu\text{m}$ thick and $360 \mu\text{m}$ wide) located on the *input surface*. The damage on the output surface clearly follows the shape of the input surface contamination particle.

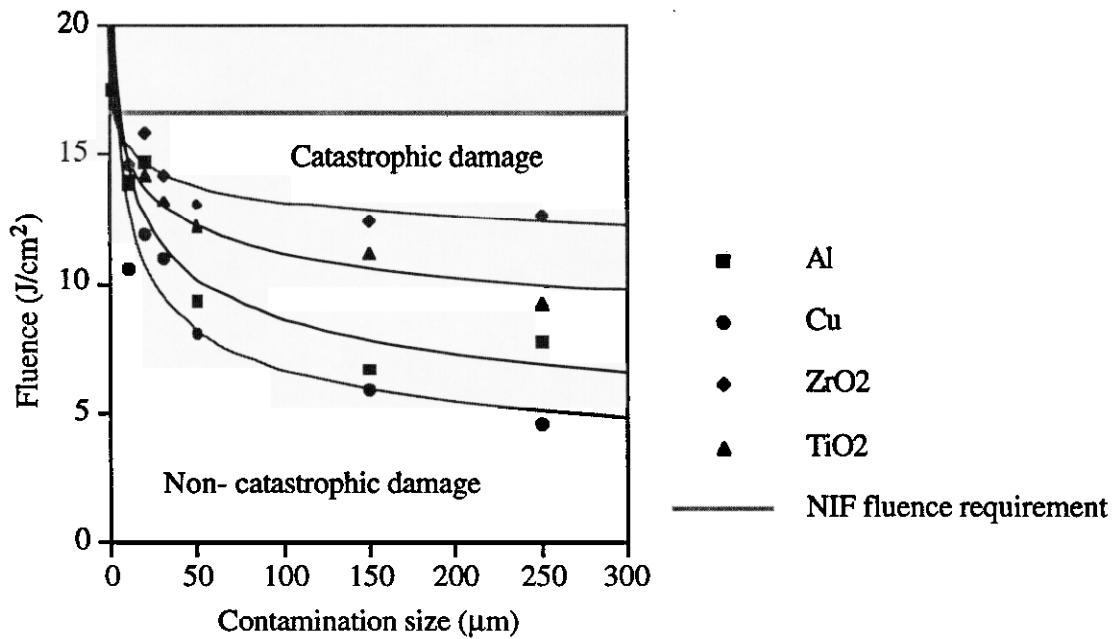


Fig. 7: Damage stability map at 355 nm showing the dependence of the onset of catastrophic damage on the fluence level and particle size for $1 \mu\text{m}$ thick sputtered particles of aluminum, copper, titania and zirconia located on the *input surface* of the silica window.

3.2 Output surface contamination:

Output surface contamination behaves differently from input surface contamination. The particle is usually ablated on the initial shot leaving a damaged area that traces the shape of the particle (see Figs. 8a) - d)).

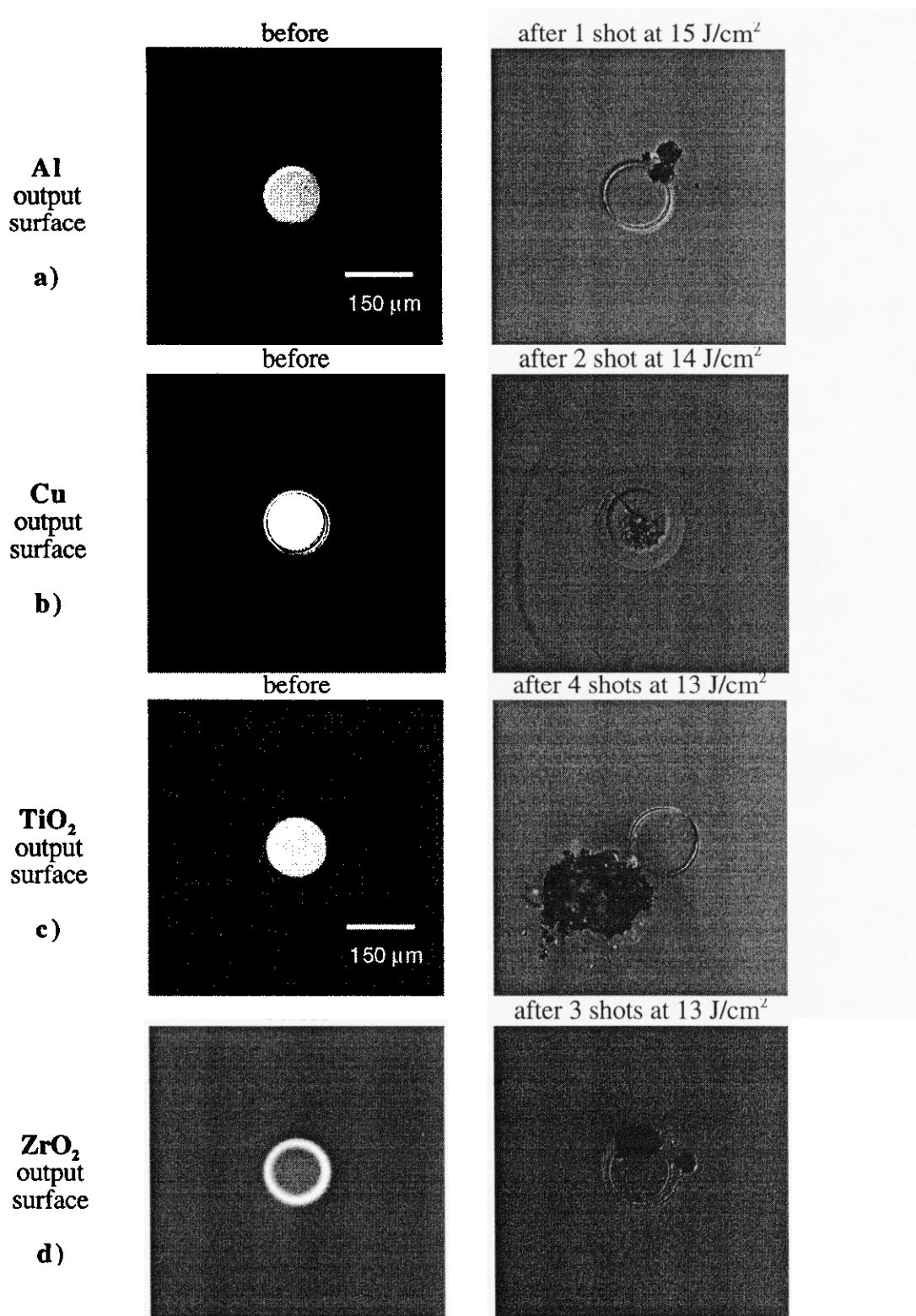


Fig. 8: Nomarski optical micrographs of the damage morphology at 355 nm of the output surface of the fused silica windows for particles of a) Al after 1 shot at 15 J/cm², b) Cu after 2 shot at 14 J/cm², c) TiO₂ after 4 shots at 13 J/cm², and d) ZrO₂ after 3 shots at 13 J/cm². All the particles (located on the *output surface*) were 1 μm thick and 150 μm in diameter. For these test conditions, the output surface damage is catastrophic.

The first shot usually ignites a plasma at the particle. Subsequent shots do not usually produce plasmas and no further damage on the surface occurs. However, if the silica surface is cracked during a given shot, the damage is very likely to grow catastrophically at some points as shown in Fig. 8.

A damage map (analogous to Fig. 7) was also plotted for output surface contaminants. The results are summarized in Fig. 9. The map shows that the effects of output surface contamination is not as severe as those of the input surface. Output surface contamination was found to have the potential for inducing catastrophic failure of the glass at fluences as low as 6 J/cm^2 for the $250 \text{ }\mu\text{m}$ Cu particles. The tests also showed that failure occurs at different fluence levels for different materials. In particular, copper contaminants had a more negative effect than other types of contaminants. The reasons for these differences between contamination materials are currently being investigated.⁷

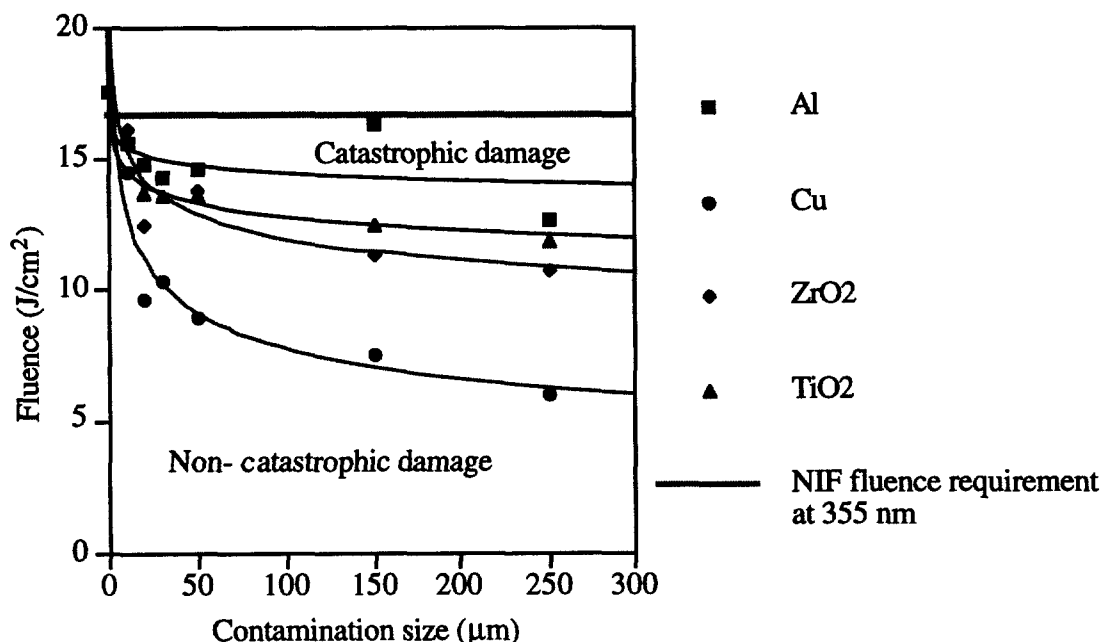


Fig. 9: Damage stability map at 355 nm showing the dependence of the onset of catastrophic damage on the fluence level and particle size for $1 \text{ }\mu\text{m}$ thick sputtered particles of aluminum, copper, titania and zirconia located on the *output surface* of the silica window.

4. CONCLUSION

Contamination of optics has been known for many years to degrade the performance of high power lasers. These effects were quantified for the case of aluminum, copper, titania and zirconia particles on bare fused silica windows. An artificial contamination technique was used to study systematically the damage (threshold and morphologies) initiated by input and output surface contamination particles during illumination at 355 nm in air. The results show that for fluences above 1.5 J/cm^2 , a plasma can ignite at the contamination particle. The damage morphologies were very reproducible from particle to particle for the same fluence and size. These morphologies were different for particles on the input vs. output surface.

Input surface contamination tends to splatter during repetitive illumination, leaving a burnt surface with a large number of small craters. A plasma is sometimes observed on the output surface too and a print of the shape of the particle is often found on this surface.

The output surface can begin to damage catastrophically during repetitive illumination when pits are initiated. These pits grow easily by drilling into the glass.

Output surface contamination is often ablated away during the first shot and leaves massive damage behind. However, when the fluence is high, a crack can form during the initial shot and catastrophic damage can occur.

Both input and output surface contaminants have therefore the potential for initiating catastrophic failure of the output surface at fluences lower than the damage threshold of clean optics. The results are summarized quantitatively on damage stability maps and show that fused silica can damage catastrophically at fluences below 14 J/cm^2 if a $10 \text{ }\mu\text{m}$ copper particle on the surface is on the path of the beam. Finally, input surface contamination has a more negative influence on the optics' survivability than output surface contamination.

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6. REFERENCES

1. I. A. Fersman and L. D. Khazov, "The effect of surface cleanliness of optical elements on their radiation resistance", *Optiko-Mekhanicheskaya Promyshlennost* **37**, 69 (1971), translation: *Soviet Journal of Optical Technology*, **37**, 627 (1971).
2. G. R. Wirtenson, "High fluence effects on optics in the Argus and Shiva laser chains", *Optical Engineering* **18**, 574 (1979).
3. B. E. Newnam, "Optical materials for high-power lasers: recent achievements", *Laser Focus* **18**, 53 (1982).
4. J. B. Heaney, H. Herzig, and J. F. Osantowski, "Auger spectroscopic examination of MgF_2 -coated Al mirrors before and after UV irradiation", *Applied Optics* **16**, 1886 (1977).
5. F. Y. Génin, J. Furr, K. Michlitsch, M. R. Kozlowski, and P. Krulevitch, "Laser-induced damage of fused silica at 355 and 1064 nm initiated at aluminum contamination particles on the surface", to be published in the 28th annual Boulder Damage Symposium proceedings (1996).
6. K. D. Moller in "*Optics*", University Science Books, Mill Valley, CA, 160 (1988).
7. M. D. Feit, A. M. Rubenchik, D. R. Faux, R. A. Riddle, A. Shapiro, D. C. Eder, B. M. Penetrante, D. Milam, F. Y. Génin, and M. R. Kozlowski, "Modeling of laser damage initiated by surface contamination", in these proceedings.
