

High Laser Damage Threshold Surface Relief Micro-Structures for Anti-Reflection Applications

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ABSTRACT

Microstructures built into the surfaces of an optic or window, are an effective replacement for thin-film coatings in anti-reflection (AR) and narrow-band filter applications. AR microstructures exhibit particularly noteworthy performance where an average reflection loss of less than 0.2% over a four-octave range (400-1800nm) has been demonstrated, and a loss of less than 0.03% is routinely achieved for narrow-band applications. Because AR micro-textures provide a gradual change in the refractive index at a material boundary, it is expected that light can propagate through the boundary without material damage at energy levels that are much higher than that found with thin-film interference coatings. Recently, it was shown that the laser induced damage threshold (LIDT) of an inexpensive borosilicate glass window containing AR microstructures was nearly 57 J/cm² at 1064nm (20ns pulse). This LIDT is two to three times greater than the damage threshold of single-layer sol-gel AR coatings on fused silica often reported in the literature.

The development of surface relief AR textures for use in high-energy laser applications is presented. Data from scanning electron microscope (SEM) analysis, reflection measurements, and LIDT testing, is shown for high performance AR microstructures fabricated in fused silica, and borosilicate glass. Results of LIDT testing at wavelengths ranging from the near ultraviolet through the near infrared confirm the initial result that AR microstructures can operate at pulsed laser power levels at least two times higher than thin-film coatings. For near infrared applications such as laser weapons and fiber optic communications requiring high performance AR, LIDT levels for AR microstructures in fused silica are found to be at least five times greater than conventional multi-layer thin film coatings. An initial surface absorption test at 1064nm shows that AR microstructures may also exhibit improved lifetimes within continuous wave laser systems.

Keywords: Antireflection, Motheye, Microstructures, Laser Induced Damage Threshold, LIDT, Optical Filters, Waveguide Resonant Gratings, Laser Cavity Mirrors, Thin-Film Coatings

1. INTRODUCTION

The energy output and operational lifetime of high-power lasers depends critically on the elimination of reflections from the many surfaces of windows and optics found within a typical laser system. For example in the laser fusion apparatus at the National Ignition Facility (NIF) of Lawrence Livermore National Laboratories (LLNL), any external reflections or scattered light from the lasing windows or system lenses, gratings, and debris shields, adds spatial noise to the beam, limiting the ability of the system to focus enough power onto the target gas-filled pellets^[1-3]. In other medical and industrial laser-based tools, reflected light can damage sensitive equipment such as imaging detectors or even components of the laser source itself such as pump laser diodes. Further power and lifetime limiting effects such as light scattering and wavefront distortion (due to spatial non-uniformity and thermal lensing^[4]), are introduced by conventional anti-reflection (AR) or high-reflector (HR) treatments based on thin-film coatings.

The deposition of multiple thin layers of dielectric materials onto each external surface of an optic can produce constructive or destructive interference for light within a specific wavelength range propagating through the optic surface. Because of the necessary interference effect, the optical field amplitude present at each material layer interface is large, leading to thermal gradients and rapid damage near coating defects for relatively low optical energy. For broad-band AR coatings, or narrow-band HR coatings on laser mirrors, a great number of film layers are needed.

HR and AR treatments based on surface relief microstructures built directly in a window or optic material, have demonstrated breakthrough performance, and novel functionality, promising increased lifetimes in demanding military

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and commercial applications such as space or aircraft based optical systems^[15,10]. For high power laser applications, early work by Lowdermilk and Milam at LLNL^[5] and by Cook, et.al. of Schott Glass^[6], showed that AR microstructures etched in the surface of various glasses had the potential to attain high laser damage thresholds. An initial investigation into the expected high power handling capability of AR microstructures fabricated with improved processes, is presented below.

2. MICROSTRUCTURE BASED ANTI-REFLECTION TECHNOLOGY

Surface relief textures consisting of sub-wavelength sized features with smoothly varying cross sectional profiles can efficiently suppress the reflection of light, an effect that is widely known as the Motheye principle^[7-8]. The design, fabrication, and performance of AR microstructures has been previously described for a variety of materials and applications^[9-11] where durability, radiation resistance, cost, viewing angle, or broad-band performance are critical.

The performance of three types of recently fabricated AR microstructures is given in Figures 1, 2, and 3. Figure 1 shows an elevation view SEM micrograph of a periodic array of Motheye structures etched in one surface of a zinc selenide window intended for long-wave infrared (LWIR) operation. The cones are about 5 microns tall and spaced on a 2.4 micron grid. Also shown in the figure is a plot of the transmission of the ZnSe window (solid black curve) over the 6 to 15 micron LWIR wavelength range along with the transmission of an untreated ZnSe window (dashed black curve) and an estimate of the maximum transmission through a ZnSe window with no reflection loss from one surface (light gray curve). The average reflection loss over the 8 to 12 micron LWIR band is less than 1.5%.

Figure 2 shows elevation and overhead view SEM micrographs of a periodic array of holes forming an SWS AR texture in one surface of a fused silica window designed for near infrared (NIR) telecommunications applications. An Agilent optical spectrum analyzer with a fiber coupled NIR LED light source was used to measure the normal incidence reflection from the SWS textured surface. A log plot of the measured reflection is also shown in Figure 2 as the solid black curve, along with the measured reflection of an untreated fused silica surface (light gray curve). A curve showing the system noise floor (dashed black curve) is also shown. Very high AR performance is found with an average reflection loss of less than 0.03% over the 1290 to 1360nm range.

The measured performance of a Random texture AR microstructure etched into the surface of a fused silica glass window, is shown below in Figure 3. The dimensions of the features in this carpet-like texture are much less than the visible to NIR wavelengths that will pass through the window, as can be seen by the inset elevation and overhead view SEM micrographs. Broad-band, normal incidence AR performance was measured using a grating based spectrometer and plotted as the dashed gray curve in the figure. Additional reflectance data, plotted as the solid black curve, was measured by FLIR Systems Incorporated of Oregon. Reflectance levels average less than tree-tenths of one percent (0.3%) from 350nm to 1000nm. (The measured reflectance of a untreated fused silica window is shown for reference.)

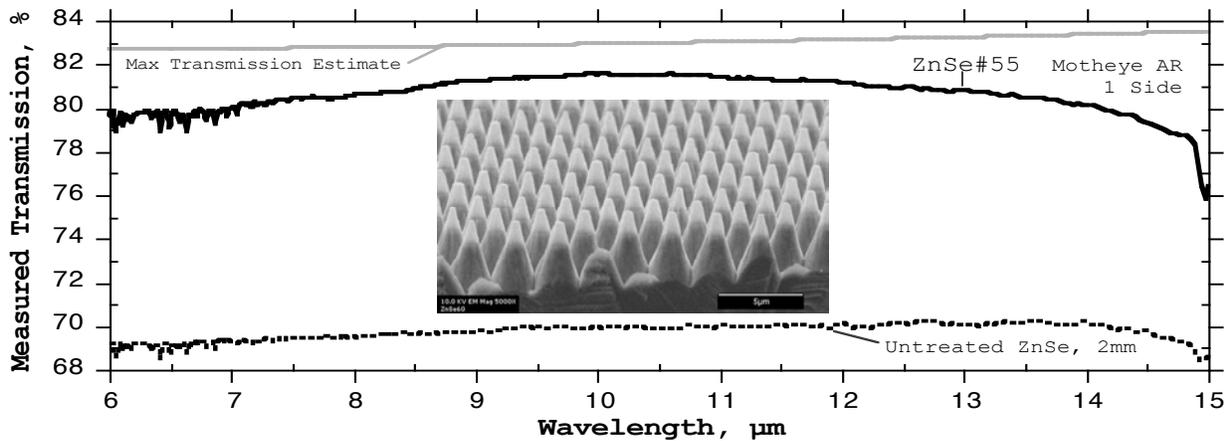


Figure 1: SEM image of a Motheye AR texture etched in a ZnSe window along with the measured LWIR transmission.

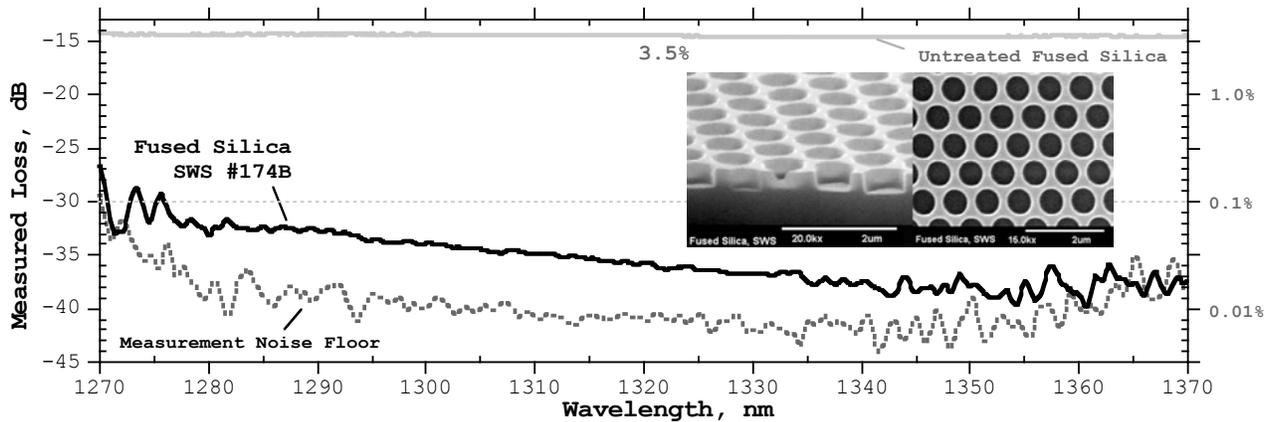


Figure 2: SEM images of an SWS AR texture etched in a fused silica window along with the measured NIR reflection.

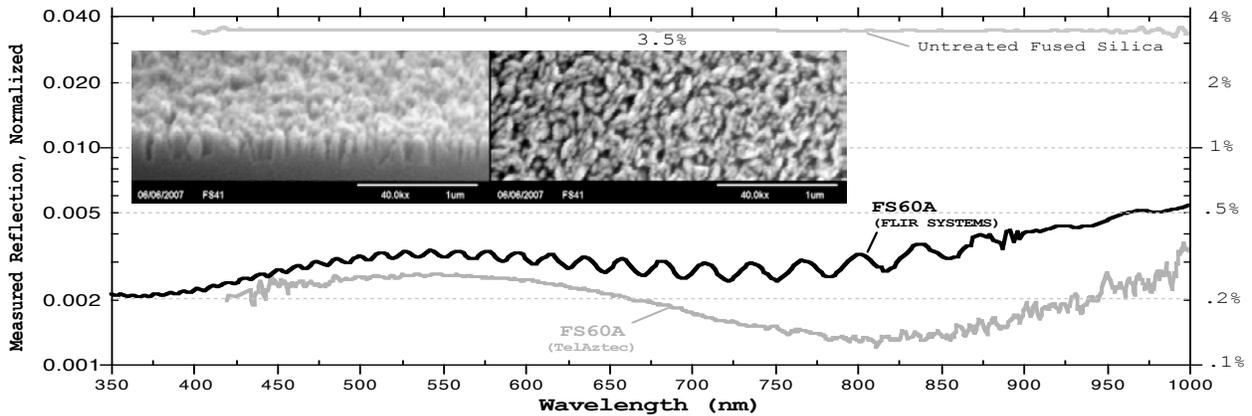


Figure 3: SEM images of a Random AR texture etched in a fused silica window and the measured NUV-VIS-NIR reflection.

3. LASER DAMAGE THRESHOLD MEASUREMENTS

The main focus of this initial investigation into the power handling capability of high performance AR microstructures was for near UV and near infrared applications utilizing fused silica and glass optics. After the high laser damage threshold recorded at 1064nm for AR microstructures etched in a Schott Borofloat 33 glass window ($57\text{J}/\text{cm}^2, 20\text{ns}$)^[11], multiple samples of the more relevant material – fused silica – were processed with Random AR microstructures to allow for additional testing at multiple wavelengths. Calibrated, NIST traceable, standardized laser induced damage threshold tests were conducted by Jeff Runkel at Big Sky Laser, Inc. in Bozeman Montana on eight AR microstructure samples and eight untreated control samples at four laser wavelengths, namely 355nm, 532nm, 1064nm, and 1538nm. In addition, short pulse length (0.5ns) laser damage testing at 351nm, was performed on three AR microstructure fused silica windows by Dr. Semyon Papernov of the University of Rochester’s Laboratory for Laser Energetics (LLE). Lastly, Dr. Josh Rothenburg of Northrup Grumman conducted an initial test of the surface absorption of AR microstructures in fused silica at 1064nm to evaluate the potential power handling of the microstructures in optics intended for use in continuous wave laser systems. Testing results are given below, organized by laser wavelength.

3.1 LIDT Testing at 1064nm

Random AR microstructures were etched into one surface of Corning 7980 fused silica glass windows each 30x30x1mm. (The current fabrication process can create AR textured windows up to 200mm diameter). The depth and density of the microstructures was set for little to no visible light scatter and broad-band visible-NIR normal incidence AR. The goal was to obtain less than 0.3% reflection at 1064nm. Figure 4 shows the measured reflection from two fused silica samples and one borofloat glass window. A grating-based spectrometer was employed with a white light

source coupled to a fiber-optic reflection probe to deliver light and receive light reflected at near normal incidence. Each sample was optically coupled to a broad-band absorber to eliminate back side reflections. The measurement range is 390nm to 1120nm, but the useful data range is limited to 425nm - 975nm by low light levels, fiber transmission, and system noise.

Fused silica windows FS40 (solid light gray curve) and FS44 (solid black curve) show broad-band AR performance with an average reflectance less than 0.4% over the VIS-NIR range. Window FS40 was selected for 1064nm testing due to its downward trend seen for wavelengths longer than 900nm. It is expected that the reflectance at 1064nm was less than 0.25%, a value that is typical for single-layer thin-film AR coatings^[12-13]. Window FS44 was selected for the 532nm testing described below. The measured reflection of an untreated fused silica window is shown as the dark gray curve at the top of the figure. For reference the reflection of AR microstructures in a Schott borofloat 33 glass window, BF108A, is shown as the dashed black curve. The AR performance of BF108A is similar to the performance of the window initially damage tested at 1064nm (57 J/cm², 20ns)^[11].

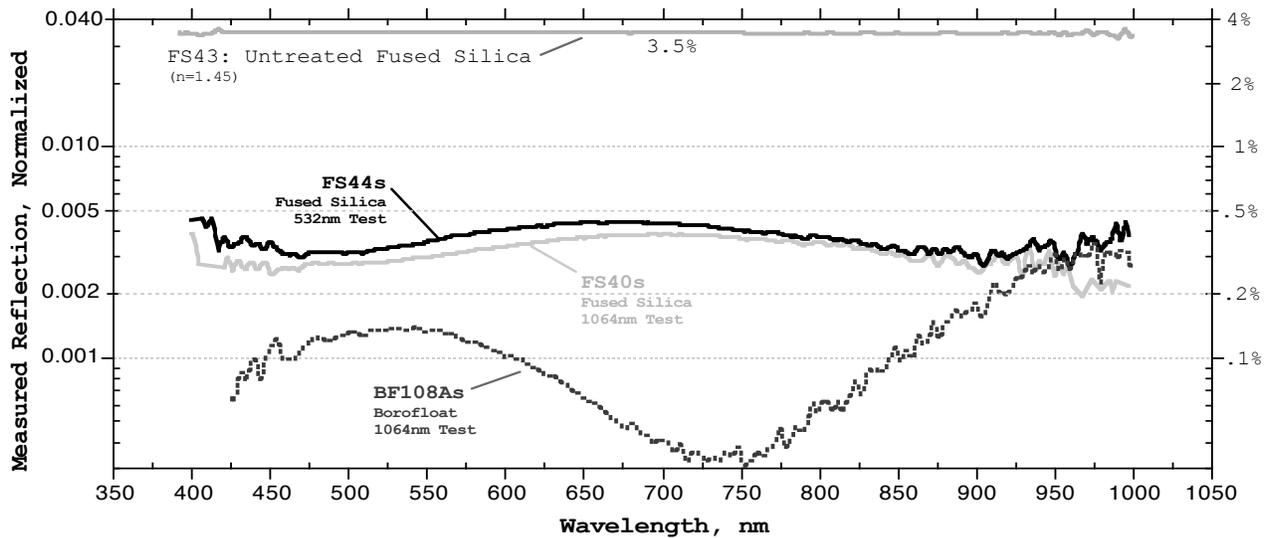


Figure 4: Reflection of VIS-NIR light from Random AR microstructures in glass windows submitted for LIDT testing.

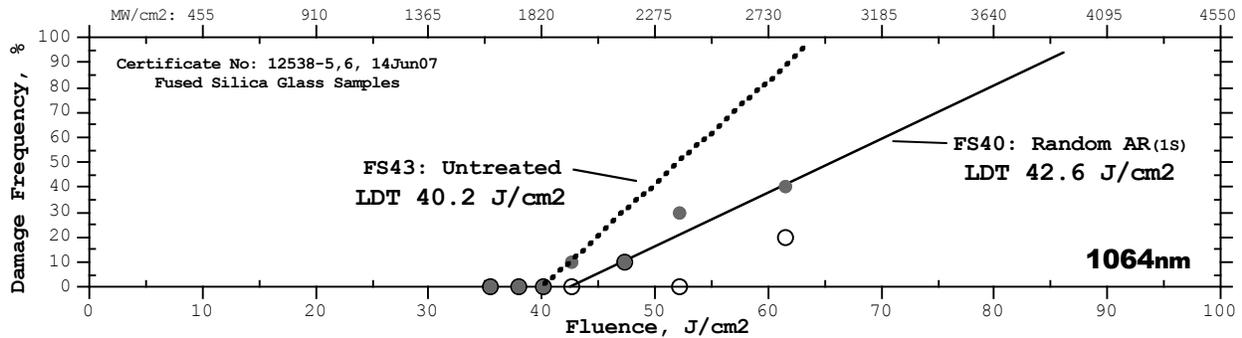


Figure 5: Results of a 1064nm LIDT test of Corning 7980 fused silica glass windows with and without AR microstructures.

It has been reported in the literature that surface preparation is critical for attaining adequate laser damage thresholds for materials in high power laser systems^[13]. Consequently, both the AR micro-structured fused silica window FS40 and the untreated window FS43 were cleaned prior to the damage testing with a standard acid (H₂SO₄:H₂O₂) immersion and solvent rinse followed by a nitrogen blow dry to remove any surface contaminants introduced during the fabrication or characterization processes. Both parts were then shipped to Big Sky Laser for LIDT testing at 1064nm. Big Sky exposed more than 100 locations on each window to 10 different fluence levels using a 1064nm wavelength, linearly

polarized, pulsed laser with a 20ns pulse length and a 0.5mm spot size ($TEM_{00} - 1/e^2$). The pulse repetition rate was 20Hz allowing 200 pulses at each location (10 sec dwell). The criteria for damage was a permanent surface change observed by visual inspection through a microscope configured for Nomarski/Darkfield, 150X magnification. The results, shown above in Figure 5, indicate a damage threshold for the fused silica window with the AR microstructures of 42.6 J/cm^2 , and a damage threshold for the untreated window of 40.2 J/cm^2 . This threshold is a factor of 2 greater than published data for single-layer thin-film coatings on fused silica^[2,13], at least 5 times greater than typical multi-layer AR coating thresholds^[12], and about 33% greater than published data for sol-gel AR coatings^[3].

3.2 Surface Absorption Testing at 1064nm

Dr. Josh Rothenburg of Northrup Grumman Space Technology, conducted an initial test of the thermal absorption at the surface of a fused silica window containing AR microstructures to evaluate the potential power handling of the microstructures in optics intended for use in continuous wave (cw) laser systems. The test was done with a cw laser operating at 1064nm, and is essentially a very sensitive system for measuring the amount of transmission lost due only to absorption at the window surface – the bulk window absorption is factored out. The technique gives a good indication of how much spatial wavefront distortion will be introduced over a large area beam propagating through an AR treated surface. Dr. Rothenburg found that the surface absorption of fused silica windows with AR microstructures was equivalent to or better than an untreated window within the 10 parts per million accuracy of his current measurement system. This initial result appears promising. How the result compares with thin-film AR coatings of interest to Northrup Grumman has yet to be determined.

3.3 LIDT Testing at 532nm

Random AR microstructures were etched into one surface of a Corning 7980 fused silica glass window, FS44, as described above. A second untreated fused silica, designated FS48, was prepared as a control sample. Both parts were cleaned and sent to Big Sky Laser for LIDT testing at 532nm. Similar to the 1064nm testing, Big Sky exposed more than 120 locations on each window to 10 different fluence levels using a 532nm wavelength, linearly polarized, pulsed laser with a 10ns pulse length and a 0.39mm spot size ($TEM_{00} - 1/e^2$). The pulse repetition rate was 20Hz allowing 200 pulses at each location. The criteria for damage was again a permanent surface change as observed by visual inspection through a microscope. The results are shown in Figure 6 where the damage threshold for the fused silica window with the AR microstructures is 23.4 J/cm^2 , and the damage threshold for the untreated window is 11.7 J/cm^2 . (The threshold found for the untreated window is inconsistent with the 1064nm test results and may be low due to inadequate surface cleaning prior to the test.) Note that the damage frequency of the AR microstructure sample remains below 3% for fluence levels between 20 and 40 J/cm^2 , suggesting that the practical level may be closer to 40 J/cm^2 .

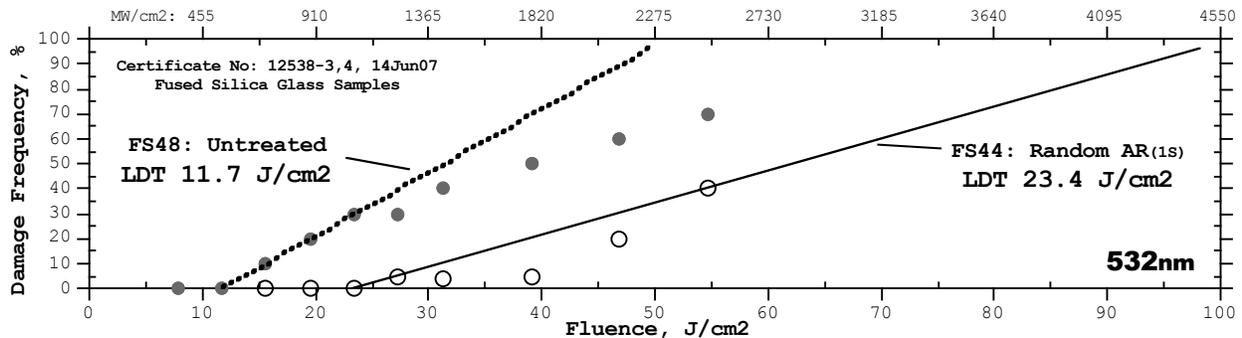


Figure 6: Results of a 532nm LIDT test of Corning 7980 fused silica glass windows with and without AR microstructures.

3.4 LIDT Testing at 1538nm

Because many high power laser radars and free-space communications systems operate at the relatively eye-safe wavelength range near 1550nm, and nearly all long haul optical telecommunications systems operate in the 1520-1590nm wavelength band, pulsed laser damage thresholds of optical materials at these wavelengths is of great practical interest. Random AR microstructures were etched into both surfaces of a highly polished fused silica glass window, 50mm round by 3mm thick, that was originally purchased for applications in the deep UV range. The depth and density

of the microstructures was set for low visible light scatter and less than 1% reflection at 1550nm. In addition, an SWS AR microstructure designed for 1550nm operation (similar to that shown in Figure 2), was cut from a larger window to yield a 10mm round, 2mm thick fused silica sample. Figure 7 shows the measured reflection from the two fused silica samples over the telecommunications C and L bands – 1480nm to 1600nm. An Agilent optical spectrum analyzer was employed with an infrared LED light source coupled to a fiber-optic reflection probe to deliver light and receive light reflected at near normal incidence. The back side reflection of each sample was eliminated by refraction through an index matched fluid.

Fused silica window FS55 (dotted solid black curve) containing Random AR microstructures shows a reflectance of less than 0.75% at 1550nm. Much higher AR performance is observed for fused silica window SWS1 (solid black curve) showing an average reflectance less than 0.03% (-30dB) at 1550nm, a level that is demanded by most fiber-optic applications. The measured reflection of an untreated fused silica window is also shown as the dark gray curve at the top of the figure, and the measurement system background noise level is shown as the dashed gray curve indicating that reflectance levels as low as 0.01%, or -40dB, can be measured with this system.

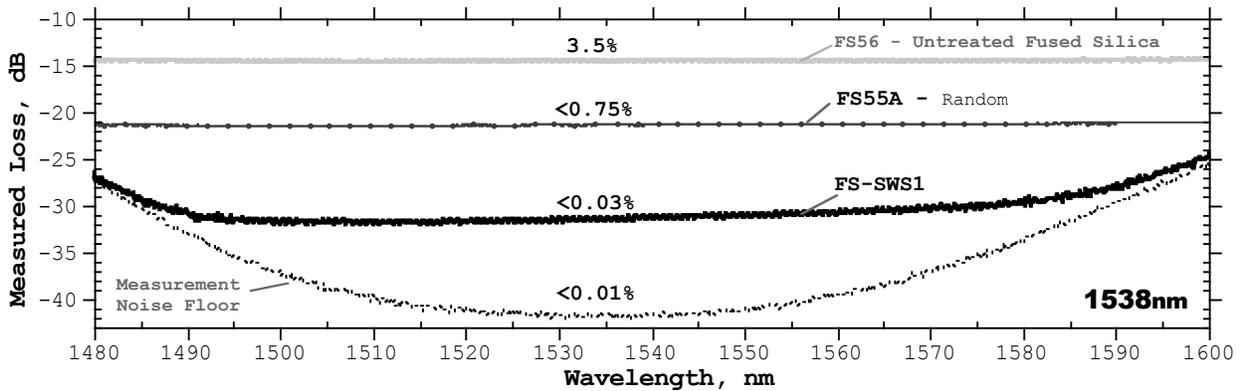


Figure 7: Reflection of NIR light from Random and SWS AR microstructures in glass windows submitted for LIDT testing.

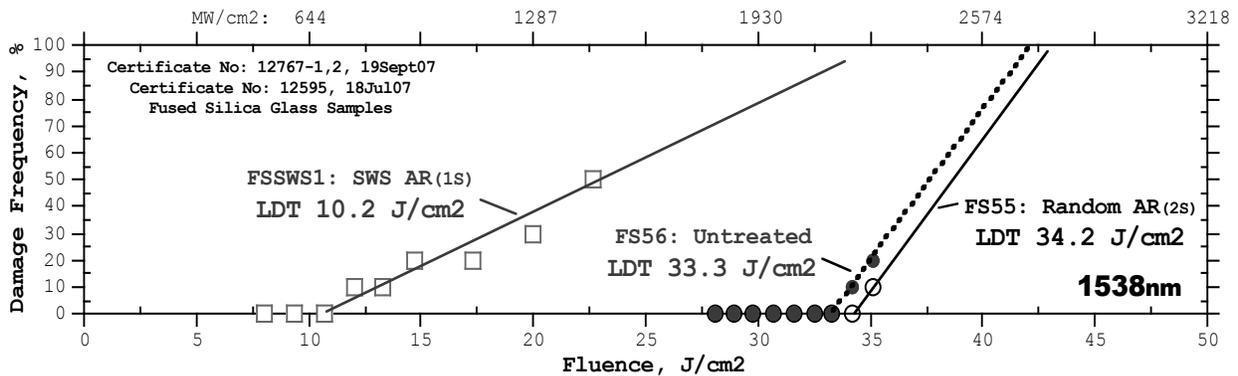


Figure 8: Results of a 1538nm LIDT test of fused silica glass windows with and without AR microstructures.

A third untreated fused silica window from the same lot as the FS55 window, was prepared as a control sample and designated FS56. All three parts were cleaned and sent to Big Sky Laser for NIR LIDT testing at 1538nm. Big Sky exposed 90 locations on each window to 9 different fluence levels using a 1538nm wavelength, linearly polarized, pulsed laser with a 14ns pulse length and a 0.31mm spot size ($TEM_{00} - 1/e^2$). The pulse repetition rate was 20Hz allowing 200 pulses at each location. Again, the criteria for damage was a permanent surface change as observed by visual inspection through a microscope. The results are shown above in Figure 8 where the damage threshold for the fused silica window with the AR microstructures is $34.2 J/cm^2$, and the damage threshold for the untreated window is $33.3 J/cm^2$. The damage threshold for the SWS AR microstructured surface is $10.2 J/cm^2$. This level, while at least 2

times higher than an equivalent performance thin-film AR coating^[12], is lower than anticipated from the previous test results. This may be explained by inadequate surface cleaning or material defects, but may also be fundamental owing to the nature of the SWS AR function which relies on interference effects similar to single-layer thin-film AR coatings.

3.5 LIDT Testing at 355nm

One sample of fused silica Random AR microstructures designed and fabricated for 1064nm operation, was prepared for damage testing at 355nm. Because the light scattering level of this first sample was expected to increase significantly for wavelengths below 400nm, a characteristic that may limit the measured damage threshold, a second fused silica window was fabricated with AR microstructures of reduced depth and increased packing density. This second window, designated FS53, was also chosen from highly polished fused silica stock that had been intended for deep UV applications (lowest absorption at 244nm). A third Schott Borofloat glass window containing Random AR microstructures designed for very high visible-band AR performance, was also prepared for 355nm damage testing. The goal was to obtain less than 0.3% reflection at 355nm. Figure 9 shows the measured reflection from the two fused silica samples and the borofloat glass window. As with the Figure 4 data, a grating-based spectrometer was employed with a white light source coupled to a fiber-optic reflection probe to deliver light and receive light reflected at near normal incidence. Again each sample was optically coupled to a broad-band absorber to eliminate back side reflections.

Fused silica window FS42 (solid light gray curve) was fabricated along with FS40 and FS44 described above, exhibiting the same broad-band AR performance with an average reflectance less than 0.4% over the VIS-NIR range. The reflectance of FS42 at 355nm was estimated to be less than 0.5% but with a potentially limiting level of scattering. Fused silica window S53 (solid black curve) shows significantly reduced AR performance at longer wavelengths but the short wavelength trend indicates that the reflectance level at 355nm may have been less than 0.2% (with little or no scattered light), a value that is typical for sol-gel AR coatings^[14]. The measured reflection of an untreated fused silica window is shown as the dark gray curve at the top of the figure. The reflection of the third sample of AR microstructures in a Schott Borofloat 33 glass window, BF110, is shown as the dashed black curve. The AR performance of BF110 is estimated to be less than 0.2% at 355nm with very little scattered light.

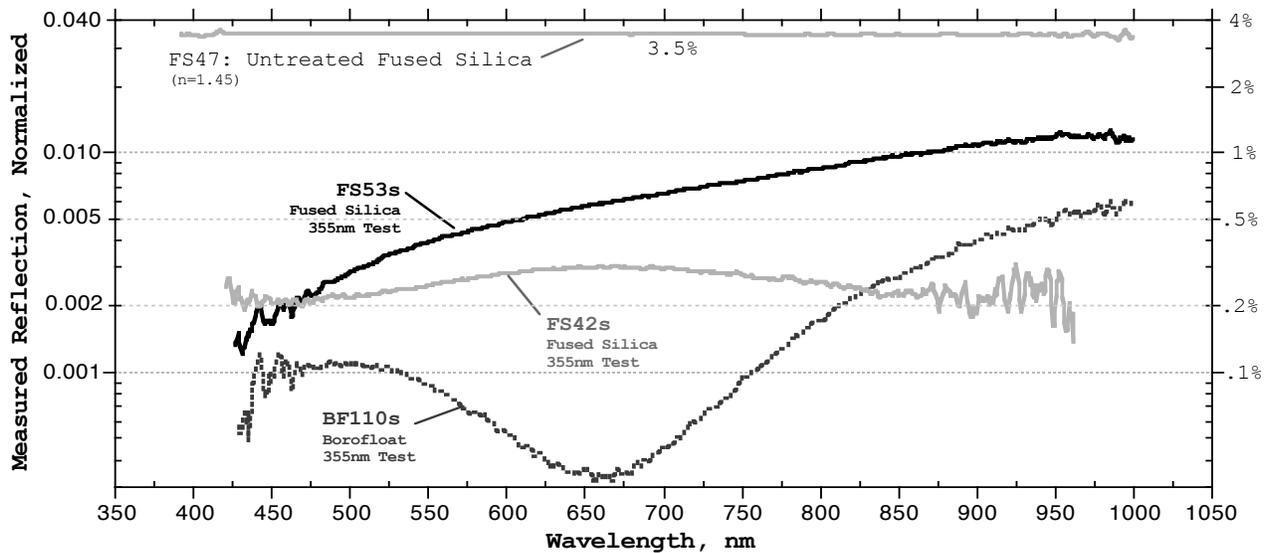


Figure 9: Reflection of VIS-NIR light from Random AR microstructures in glass submitted for 355nm LIDT testing.

All three AR micro-structured windows and two untreated fused silica windows (FS47 and FS54) were cleaned prior to the damage testing with a standard acid (H2SO4:H2O2) immersion and solvent rinse followed by a nitrogen blow dry. All parts were then shipped to Big Sky Laser for LIDT testing at 355nm. In the first test with AR micro-structured window FS42 and untreated window FS47, Big Sky exposed more than 100 locations on each window to 10 different fluence levels using a 355nm wavelength, linearly polarized, pulsed laser with a 10ns pulse length and a 0.28mm spot

size ($TEM_{00} - 1/e^2$). The pulse repetition rate was 20Hz allowing 200 pulses at each location. The criteria for damage was a permanent surface change observed by visual inspection through a microscope. The results, shown above in Figure 10, indicate a damage threshold for the fused silica window with the AR microstructures of $9.9 J/cm^2$, and a damage threshold for the untreated window of $22.4 J/cm^2$. According to information provided by Big Sky Laser, this threshold is about 20% greater than the best single-layer thin-film AR coatings they have tested in the past.

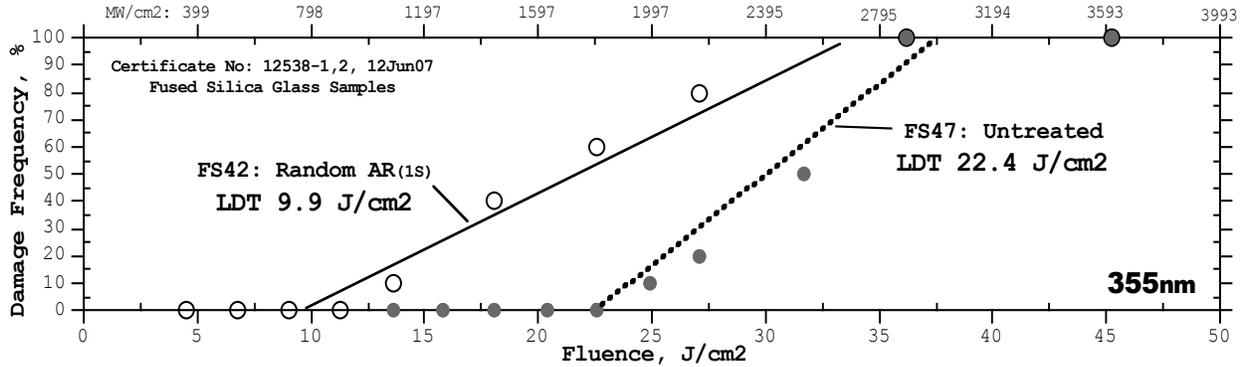


Figure 10: Results of a first 355nm LIDT test of fused silica glass windows with and without AR microstructures.

In the second test with AR micro-structured windows FS55 and BF110, and untreated fused silica window FS56, Big Sky exposed more than 100 locations on each window to between 3 and 8 different fluence levels using the same 355nm wavelength, linearly polarized, pulsed laser with a 10ns pulse length and a 0.29mm spot size ($TEM_{00} - 1/e^2$). The pulse repetition rate was 20Hz allowing 200 pulses at each location. The results, shown in Figure 11, indicate a damage threshold for the fused silica window with the optimized AR microstructures of $19.8 J/cm^2$, and a damage threshold for the untreated fused silica window of $21.3 J/cm^2$. A similarly high damage threshold of $21.3 J/cm^2$ is found for the borofloat glass window with the AR microstructures. Because such high thresholds were not anticipated before the test, the measurement was made at the limit of the system power calibration. A repeat of the test with a smaller spot size may yield an even higher damage threshold measurement. In any case, there appears to be no published data for any other type of AR treatment that rivals the damage threshold found with AR microstructures at 355nm.

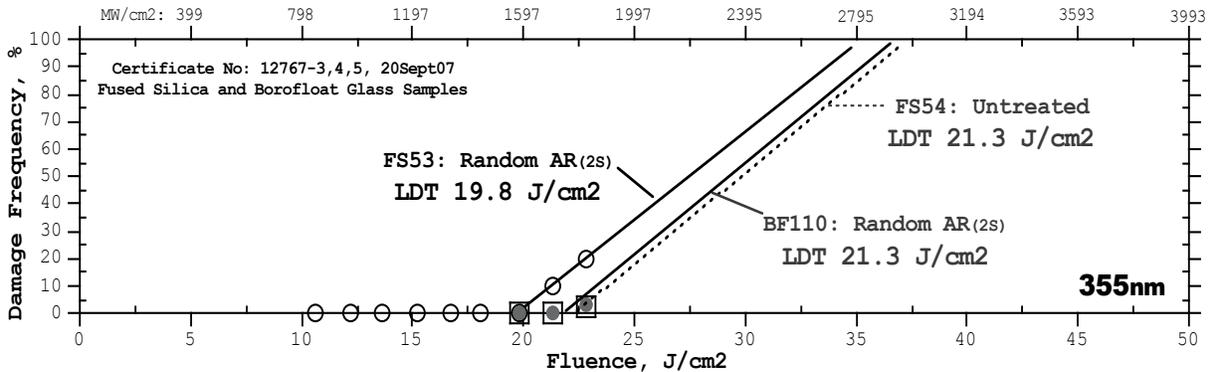


Figure 11: Results of a second 355nm LIDT test of fused silica glass windows with and without AR microstructures.

3.6 Short Pulse Length LIDT Testing at 351nm

For the laser fusion programs at both LLNL and Rochester's LLE, very short pulse length (0.5ns) lasers operating at 351nm are employed. To extrapolate the results of the 355nm, 10ns pulse length damage tests described above to a damage level for 351nm, 0.5ns pulse length laser operation, a scaling factor equivalent to the ratio of the square roots of the pulse lengths is often applied. This would reduce the damage thresholds found for fused silica windows FS42 and FS53 by a factor of about 4.5 yielding 2.2 J/cm^2 and 4.4 J/cm^2 respectively. These extrapolated levels do not compare favorably with the $12\text{-}14 \text{ J/cm}^2$ damage thresholds for sol gel AR coatings reported by the LLE^[14]. From discussions with other industry professionals, it was learned that the square root scaling law may be applicable only for longer pulse lengths. To sort this problem out, Dr. Semyon Papernov of Rochester's LLE was kind enough to conduct two separate laser damage tests on fused silica windows fabricated with AR microstructures in both surfaces using Rochester's 351nm, 0.5ns pulsed laser system.

Dr. Papernov conducted two types of tests described as single-shot 1-on-1, and multiple shot n-on-1 tests. In most respects the n-on-1 test is similar to the s-on-1 tests made by Big Sky Laser. The damage criteria was a permanent surface change as observed using dark field microscopy at 100X magnification. In the first n-on-1 test fused silica windows FS49 and FS50 containing AR microstructures with performance similar to the FS42 window, exhibited damage thresholds of 8.83 and 8.29 J/cm^2 respectively. This result is close to the 9.9 J/cm^2 found for FS42 tested at 355nm with a 10ns pulse length source indicating that the square root scaling law may not be applicable for short pulse lengths below 10ns. A second n-on-1 test with a fused silica window containing AR microstructures designed to produce less scattered light in the UV (FS63), resulted in a damage threshold of 10.44 J/cm^2 . (This test may have been limited by scattered light and microstructure defects on the back surface of the window due to processing errors.) With the potential for less environmental sensitivity than the fragile sol-gel coatings used today, these tests indicate that etched surface relief AR microstructures may prove useful for laser fusion applications.

4. SUMMARY

High performance AR microstructures have been fabricated in fused silica and Schott borofloat glass windows and tested for laser induced damage at five laser wavelengths ranging from the UV to NIR. High pulsed laser damage thresholds were found, particularly at 1064nm and 355nm. The results indicate a level of performance that is uncommon in the literature and may be unprecedented. Etched surface AR microstructures show a damage threshold at least two times greater than comparable single-layer thin-film AR coatings at 1064nm. In the near UV, both short (0.5nsec) and long (10ns) pulse length systems find damage thresholds near 10 J/cm^2 , a level that compares favorably with current fragile sol-gel thin-film AR coatings – but with the potential to yield more durable optics. Figure 12 is a chart summarizing the test results including some results from literature references. An initial test of the surface absorption of Random AR microstructures at 1064nm shows the potential for increased lifetime of optics in high power continuous-wave laser systems.

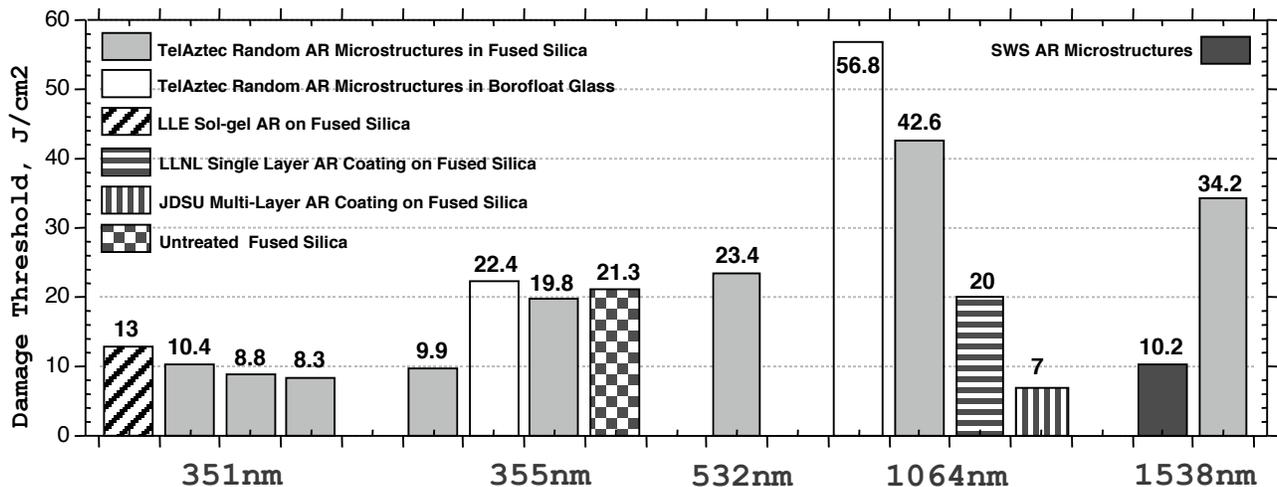


Figure 12: Summary of LIDT test results compared to results from references 2,3, 12, 13, and 14.

5. ACKNOWLEDGEMENTS

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