Study of the Environmental and Optical Durability of AR Microstructures in Sapphire, ALON, and Diamond

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ABSTRACT

Data is presented for the erosion resistance and pulsed laser damage threshold of anti-reflecting (AR) microstructures built in the surface of the infrared light transmitting window materials sapphire, ALON, and diamond. It was found that the erosion resistance of AR microstructures (ARMs) in sapphire is comparable to the resistance of sapphire with no AR treatment. Such environmental durability, combined with the enhanced light transmission of windows incorporating ARMs, provides system designers with an effective solution for applications requiring high transmission over long mission times operating in abrasive environments. In addition, the optical power handling capacity of sapphire and ALON windows was investigated through pulsed laser damage threshold measurements with a laser source operating in the near infrared at a wavelength of 1573nm. As with prior results reported for ARMs in fused silica and borosilicate glass, the measured damage threshold of 19 J/cm^2 for ARMs treated sapphire windows is comparable to the damage level measured for untreated sapphire windows, and this level is at least two times higher than that found with the most durable thin-film AR coatings designed for fused silica. The damage thresholds measured for untreated and ARMs treated ALON windows was also comparable, but at a level more than four times less than the sapphire windows. Lastly, the long-wave infrared light transmission of high performance ARMs fabricated in clear diamond windows is presented. The Air Force Research Laboratoy's Laser Hardened Materials Evaluation Laboratory at WPAFB tested the damage threshold of the ARMs treated diamond windows along with untreated diamond windows using their pulsed CO2 laser setup operating at 9.56µm. Although the results of the tests using two different laser settings were quite variable and inconsistent due to the nature of the diamond material, the damage thresholds measured were in the 50 to 100 J/cm^2 range, a level much higher than can be achieved with thin-film AR coatings.

Keywords: Antireflection, AR, Motheye, Microstructures, Mechanical Durability, Laser Damage Threshold, Infrared

1. INTRODUCTION

In many commercial and military optical systems that must operate within harsh environments, the transmission losses due to light reflections from mechanically durable window materials such as sapphire, spinel, ALON, and diamond, cannot be tolerated. For these applications some form of anti-reflection (AR) treatment is required. The conventional AR treatment today relies on the deposition of one or more thin-film material layers over a window or optic surface. Such AR coatings cannot be made to resist erosion from rain and sand particle impacts when operating in an abrasive environment to a level equivalent to that of an untreated window.

An effective AR treatment is also critical in high power laser systems where back reflections can easily damage the laser source or sensor system. Here again, the practical problems associated with thin-film interference AR coatings often limit the lifetime and the amount of laser power that can be transmitted. A more durable AR treatment is needed when both high energy laser transmission and erosion resistance are specified.

An AR treatment based on surface relief micro-structures built directly into a window material, has demonstrated superior AR performance with higher transmission over broader bandwidths than thin film coatings in many applications[1-6]. Specifically, AR microstructure (ARMs) technology offers significant advantages over thin-film AR coating technology for high power laser systems[7], for devices operating in environments with high levels of radiation[8], and for windows subject to erosion by rain or sand particle impacts in abrasive environments[9]. The goal of this work is to further investigate the combination of mechanically durable window materials with the high performance and power handling capacity of microstructure-based AR treatments. (Three 1-inch sapphire windows incorporating ARMs designed for NIR operation are shown on the right above).

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Window and Dome Technologies and Materials XI, edited by Randal W. Tustison,
Proc. of SPIE Vol. 7302, 73020J · © 2009 SPIE · CCC code: 0277-786X/09/$18 · doi: 10.1117/12.818335

2. MICROSTRUCTURE BASED ANTI-REFLECTION TECHNOLOGY

An array of microstructure protrusions or depressions in the surface of a window or optic, commonly known as Motheye textures[10], can produce an effective AR function through the smooth gradation of the refractive index between the environment and the bulk window material. TelAztec has reported on the design and fabrication of many different types of AR microstructures in many materials and for many applications – all with unique and beneficial properties and exceptional performance[4-9,11]. Recently, ARMs have been designed and fabricated in clear crystalline diamond windows for long wave infrared (LWIR) applications. Figure 1 shows SEM images of the microstructures built in clear diamond window fragments supplied by sp3 Diamond Technologies and Lockheed Martin. Figure 2 shows the measured LWIR transmission of the ARMs treated windows (solid gray and black curves) compared to an untreated window. Also shown in Figure 2 is the goal performance for the SWS-type ARMs in diamond as predicted by a computer model based on rigorous coupled wave analysis.

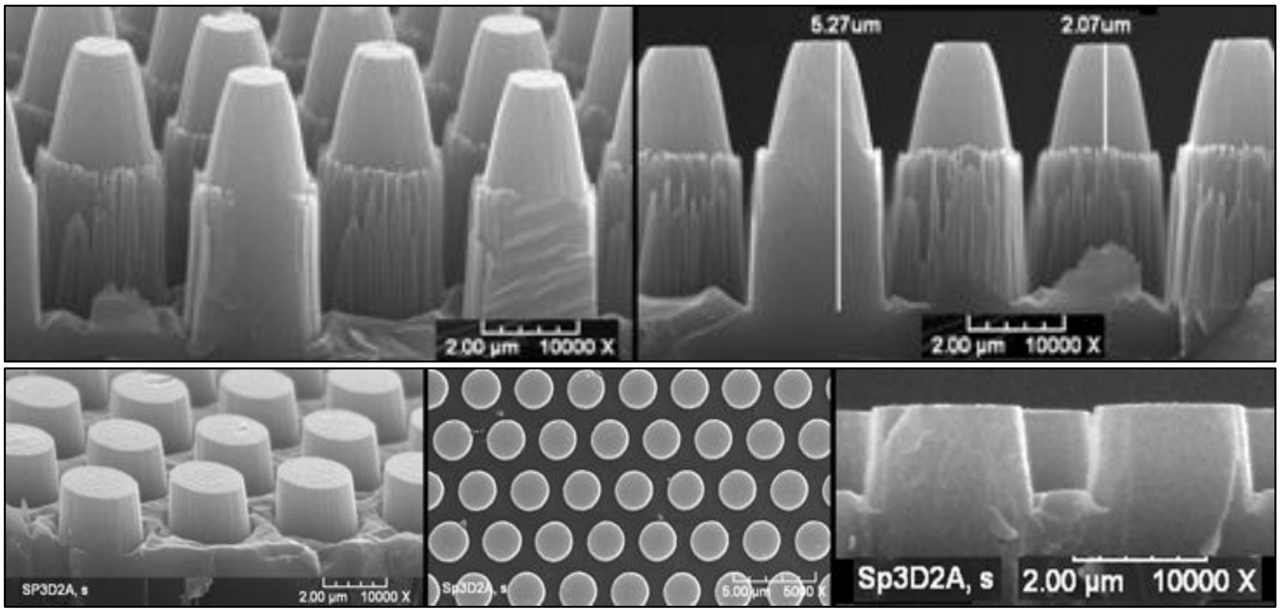


Figure 1: SEM images of cone-like (top) and binary profile (bottom) ARMs fabricated in the surface of diamond windows.

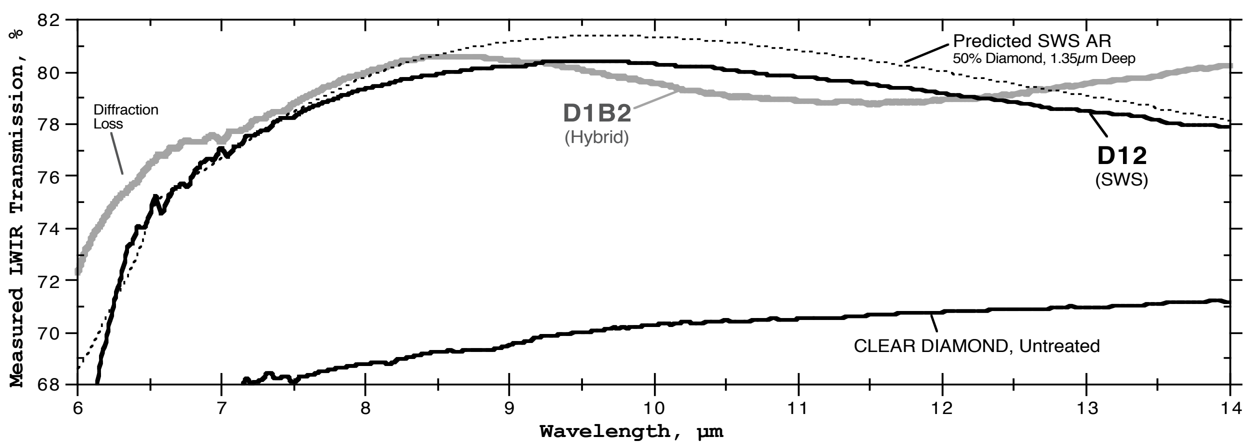


Figure 2: Measured LWIR transmission of untreated and ARMs treated clear diamond windows.

An improved fabrication process has been developed for producing ARMs in sapphire and ALON windows for mid-wave infrared (MWIR) applications. Figure 3 shows SEM images of Hybrid-type ARMs built in ALON windows acquired from Surmet Corporation. Figure 4 shows the measured MWIR transmission of the ARMs treated window (solid black curve) compared to an untreated ALON window (dashed black curve). With further process optimization to increase the texture fill factor and cone height, the measured transmission is expected to reach the theoretical single surface maximum of 92% indicated in the figure.

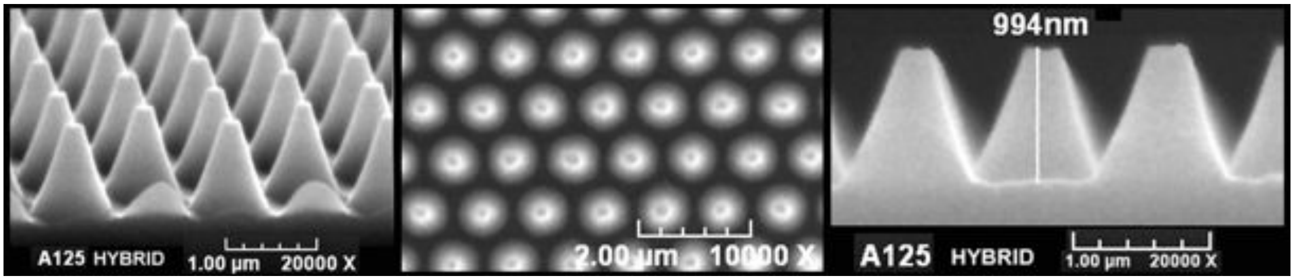


Figure 3: SEM images of HYBRID-AR cone-profile ARMs fabricated in the surface of an ALON window.

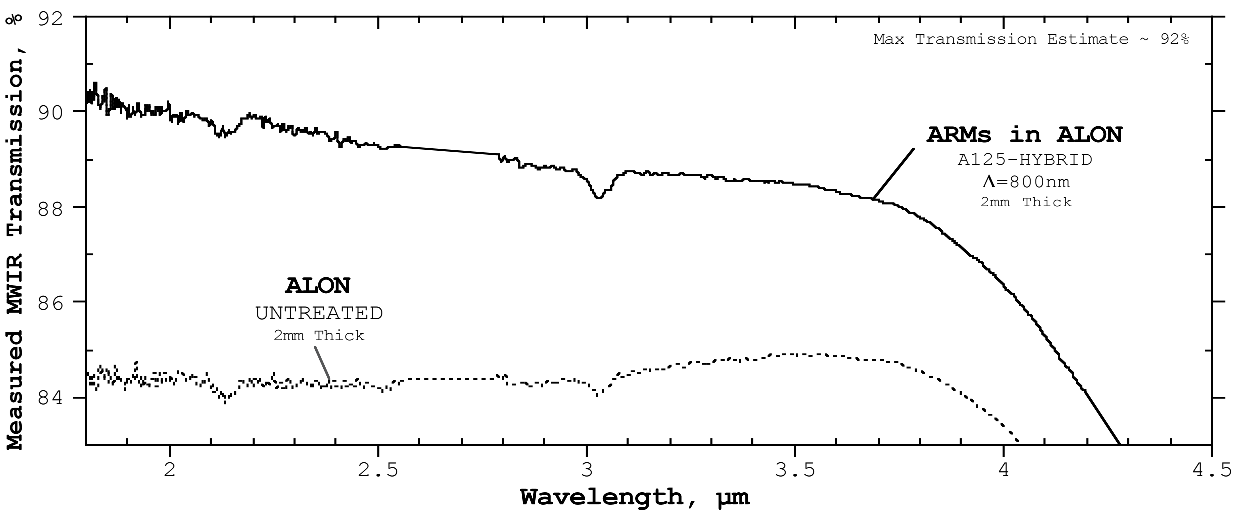


Figure 4: Measured MWIR transmission of untreated and ARMs treated ALON windows.

3. EXPERIMENTAL – WATER AND PARTICLE EROSION TRIALS

The Air Force Research Laboratories (AFRL) and the University of Dayton Research Institute (UDRI) have developed sophisticated apparatus to assess the survivability of windows and optics intended for military equipment that must operate in harsh environments. One rig is designed to expose an optic to a field of sand particles traveling at high speed. Another rig generates a curtain of water drops that fall in the path of an optic attached to a whirling arm spinning at speeds that are typical of aircraft or missile flight conditions. Both rigs are described in detail in documents provided on UDRI's website[12-13]. Building upon the experience gained from a 2008 investigation into the erosion resistance of ARMs in Clear ZnS and ZnS[9], a set of sand and rain conditions was developed for exposing both untreated and ARMs treated sapphire windows designed for enhanced NIR transmission. The expectations of this erosion trial were;

1) UDRI's Sand rig produces consistent exposure conditions day-to-day and run-to-run allowing the data collected as part of the current trial to be compared with data collected in an earlier trial as if all parts were exposed to the same conditions at the same time.

2) UDRI's Rain rig, although less consistent than the Sand rig at re-producing similar exposure conditions from day-to-day, is consistent enough to allow for comparisons of data collected in an earlier trial with the new cycle data as if all parts were exposed at the same time.

3) The transmission degradation of windows damaged by rain drop and sand impacts is sufficiently linear to allow for a reduced number of exposure levels for a given rain field or sand condition. Just two exposure levels along with the unexposed condition, yields an adequate linear fit. An exposure level can be accumulated on a single window, or multiple windows exposed to a single level can form a data set.

Further details are given below beginning with the sand erosion testing;

SAND EROSION TESTING OF AR MICROSTRUCTURES

The particle erosion rig at UDRI, pictured on the left, can be configured to deliver a number of different size silica sand particles with speeds that are relevant to specific environmental conditions. A user must select the particle size range, speed, and angle of impact, which in turn determines the number of impacts over a unit area in a given time. Expressed as the dose, or dust load in milligrams per square centimeter (mg/cm^2), the total load is chosen to simulate the lifetime of an optic or window in the intended environment. The parameters chosen for the ARMs exposures were taken from the literature[14-15], with some modification as a result of the system calibration and to be comparable to prior trial settings[9]. Large sand particles with a diameter ranging from 125 to 177 µm, traveling at 168 meters per second (m/s, or 376 miles per hour, MPH) were designated as Sand Condition A. Small sand particles with a diameter ranging from 38 to 53 µm, traveling at 262 m/s (586 MPH) were designated as Sand Condition B. Sand particles were directed perpendicular to the window surface, and two levels of exposure were specified for each Condition, for a total of four exposures, or machine cycles. For Sand Condition A the dose levels were 10 and 30 mg/cm^2, and for Sand Condition B the dose levels were 4 and 12 mg/cm^2. Each ARMs treated sapphire window was exposed to a single level. Untreated sapphire and ZnSe windows were included in each cycle as controls.

Figure 5 shows the one-inch round, 6mm thick window samples after exposure arranged by Condition and exposure level. The damage uniformity seen for the ZnSe windows is indicative of a well calibrated apparatus. All of the damage appears to be on the window surfaces, where a void or crater has been created from ejected material at each impact site. As widely reported in the industry, the amount of damage is directly related to the material hardness. Although it appears from the Figure 5 images that the untreated and ARMs treated sapphire windows have not been damaged, microscopic analysis does reveal a uniform distribution of damage sites with a higher density of damage sites found with the heavier dust loads. (The ARMs treated windows do not appear transparent due to visible light diffraction from the microstructure arrays that are designed for enhanced NIR transparency.)

Figure 5: Sapphire and ZnSe windows after sand exposure in the UDRI particle erosion rig.

Figures 6 and 7 show scanning electron microscope (SEM) images of the damage sites in the surface of the ARMs treated sapphire windows. The left hand images in each figure show the damage sites caused by the large sand particles of Condition A, and the right hand images show damage caused by the small sand particles of Condition B. Figure 6 shows overhead views of the damaged surfaces magnified 200 and 1000 times. As might be expected, the crater sites resulting from the 30mg/cm^2 Condition A exposure appear to be larger and more numerous than the crater sites found with the 12mg/cm^2 Condition B exposure. The top half of Figure 7 shows overhead views of the damaged surfaces magnified 5000 times where the microstructures appear to be both crushed into the surface and sheared at the base. The bottom half of Figure 7 details the crush damage with elevation views magnified 5000 times.

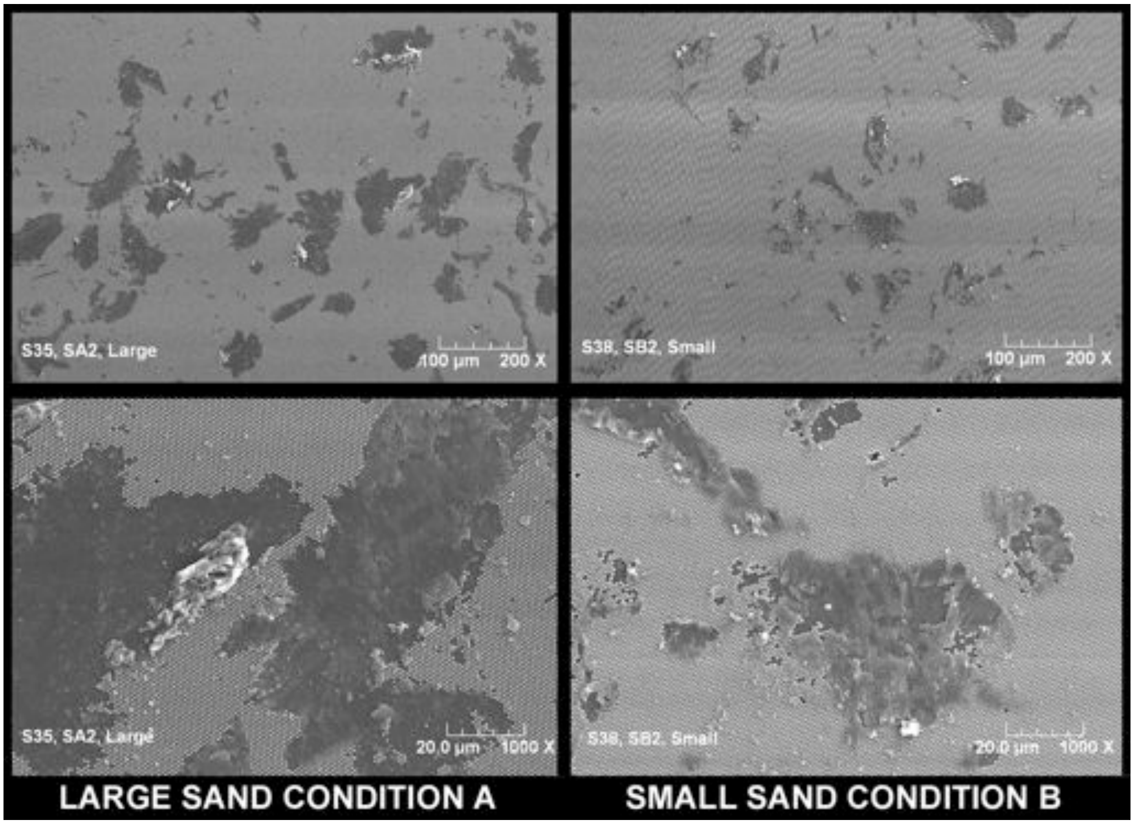


Figure 6: Overhead views (top:200X, bot:1000X) of sand impact damage on ARMs treated sapphire windows.

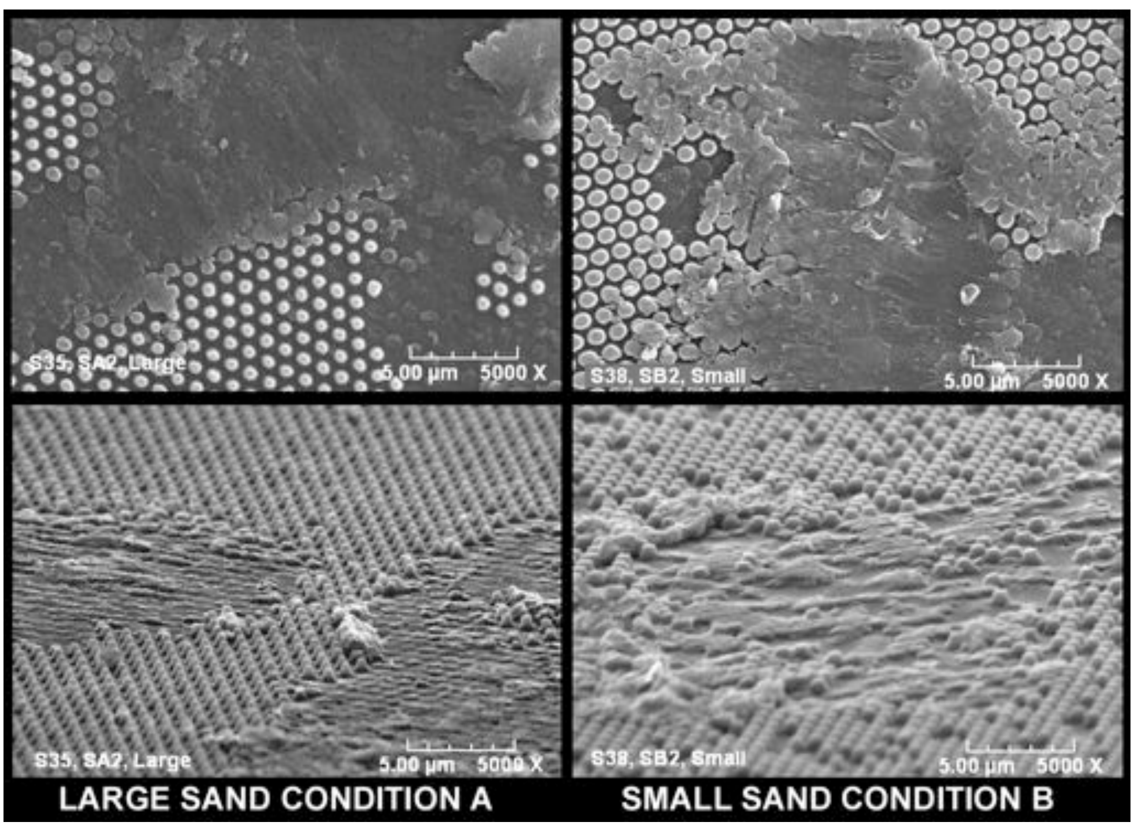


Figure 7: High magnification overhead (top) and elevation (bot) views of sand impact damage on ARMs treated sapphire.

Figures 8 and 9 depict the measured transmission of the untreated and ARMs treated sapphire windows exposed by the particle erosion rig over the near infrared (NIR) spectral range of 1300-1700nm. Spectral transmission was recorded with an accuracy of about 0.5% using a grating-based InGaAs spectrometer system with a tungsten-halogen light source transmitted and received through multimode fiber optic cables (Ocean Optics Model NIR512). The plots are organized by sand condition with Figure 8 showing the sapphire windows exposed to Sand Condition A, and Figure 9 showing the sapphire windows exposed to Sand Condition B. Measurements of the window transmission before exposure are given in each case as the thick solid black and solid gray curves. The low exposure level is indicated by the dashed black curves in each plot, and the high exposure level data is plotted as a thin solid black curve. Each curve is an average of multiple scans at multiple locations over the sample area made using a 3-4mm diameter light beam. In general, the measurements show a uniform drop in the NIR transmission for the samples exposed to the largest dust load. The transmission of the samples exposed to the low dust load remains unchanged within the measurement error of the system. This indicates that the low dust load settings cause too little damage for observation of transmission loss due to light scattering over the NIR wavelength range. Note that transmission loss does increase slightly at shorter wavelengths, a result that is also consistent with light scattering from the surface damage and their visual appearance. One exception to these observations is found with the low exposure Condition B sample where the higher than expected transmission loss may be due to the poor microstructure uniformity realized with this sample (randomly scattered voids in the ARMs array may have enlarged to cause additional scattered light loss after exposure.)

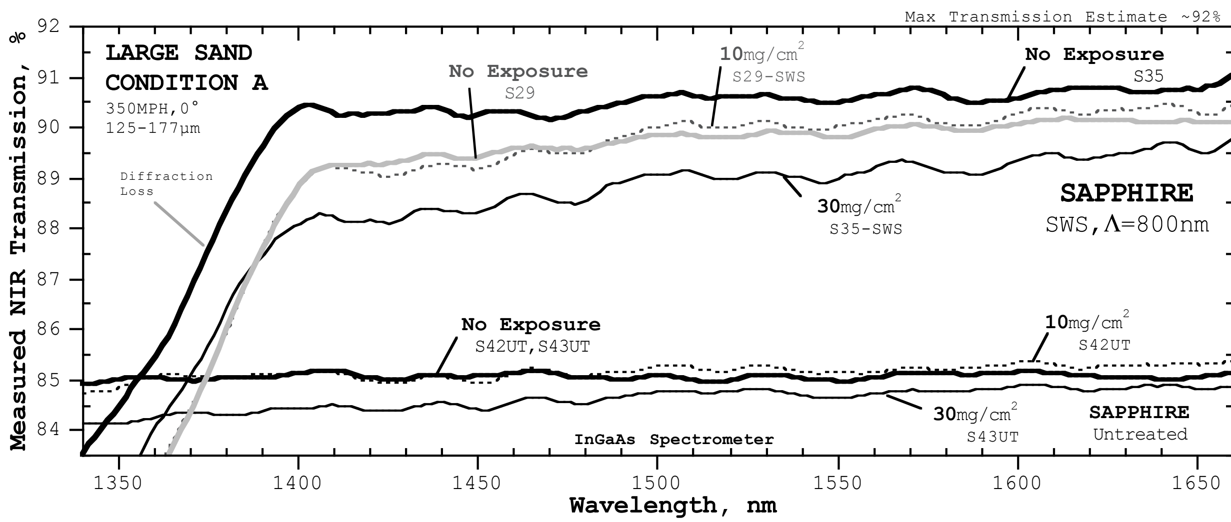


Figure 8: Measured NIR transmission of untreated and ARMs treated sapphire windows exposed to large sand condition A.

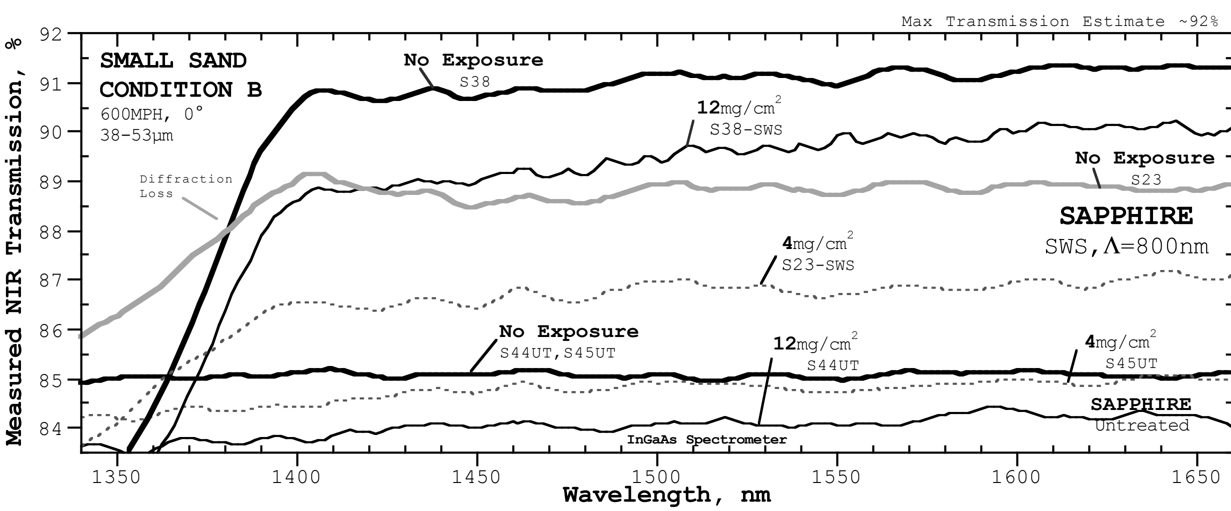
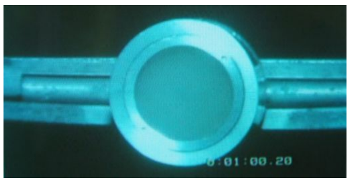


Figure 9: Measured NIR transmission of untreated and ARMs treated sapphire windows exposed to small sand condition B.

RAIN EROSION TESTING OF AR MICROSTRUCTURES IN SAPPHIRE

The nature of the damage caused by a rain drop impact is quite different than the damage caused by sand particle impacts. Typically the impact force from a rain drop creates a compression wave that cracks the material internally leaving the surface smooth and undamaged. Shearing forces over the surface as the water disburses can also cause damage to conventional thin film AR coatings. Internal cracking is catastrophic and readily observed with softer window materials such as ZnS, ZnSe, and ClearTran. Thin-film hard coatings can help – but not prevent this internal cracking, and coating failure due to delamination and surface cracking has been a major limitation[19]. Such concerns lead to the choice of untreated sapphire windows for most applications. In 2008 it was shown that high performance ARMs in ZnS were effective at preventing internal cracking[9], possibly by the lateral diffusion of the stress wave generated by a rain drop impact. The application of ARMs to sapphire windows may enable an enhanced transmission without degradation of the expected window lifetime.



The whirling arm rain erosion rig at UDRI can be configured to produce a curtain of calibrated size water droplets through which a sample is moved at high speeds by rotation of a non-lifting propeller. A user must select the water droplet size, rate of droplet production (rain rate), speed of droplet impact, and angle of impact. The impact of rain drops is of primary concern to missiles and aircraft traveling at high speed and moderate altitudes, a condition which guides the choice of parameters. Much of the work found in the literature refers to the testing of optics at a speed of 470MPH with 2mm diameter rain drops falling at a rate of 25mm each hour. This configuration was chosen for the ARMs exposures with an impact angle normal to the sample surface. At these settings, the industry expectation[14] is that a window should survive with little damage for a duration of 20 minutes. With the UDRI equipment just two 25mm diameter windows, loaded at opposite ends of the arm as shown in the picture on the left above, can be exposed during each machine cycle. One untreated window and one ARMs treated sapphire window were exposed during two cycles with durations of 10 and 20 minutes. A third machine cycle was run for 20 minutes at a higher speed of 550 MPH and a higher rain rate of 50mm/hr.

Figure 10 shows untreated ZnSe and sapphire windows along with ARMs treated sapphire windows after exposure in the UDRI rain rig for varying durations where any internal damage is observed using white light transmitted through the parts by a light table. Light reflecting from the exposed window surfaces remains smooth and specular, and microscopic inspections of the surface reveals little to no surface damage. As evident in the figure, extensive, catastrophic internal damage is found for the softer ZnSe windows, but no internal or surface damage is observed with either of the ARMs treated or untreated sapphire windows. (Again the ARMs treated sapphire windows appear gray due to diffraction of visible light from the microstructure array designed for enhance NIR transmission).

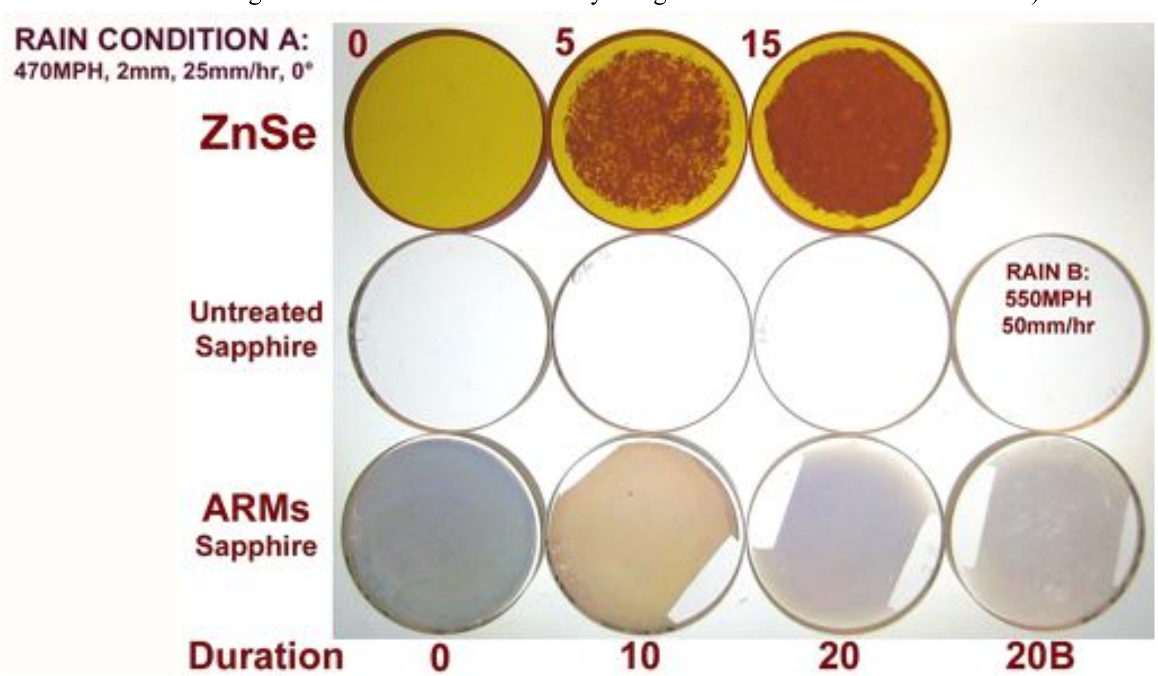


Figure 10: Untreated ZnSe, sapphire, and ARMs treated sapphire windows after rain exposure in the whirling arm rig.

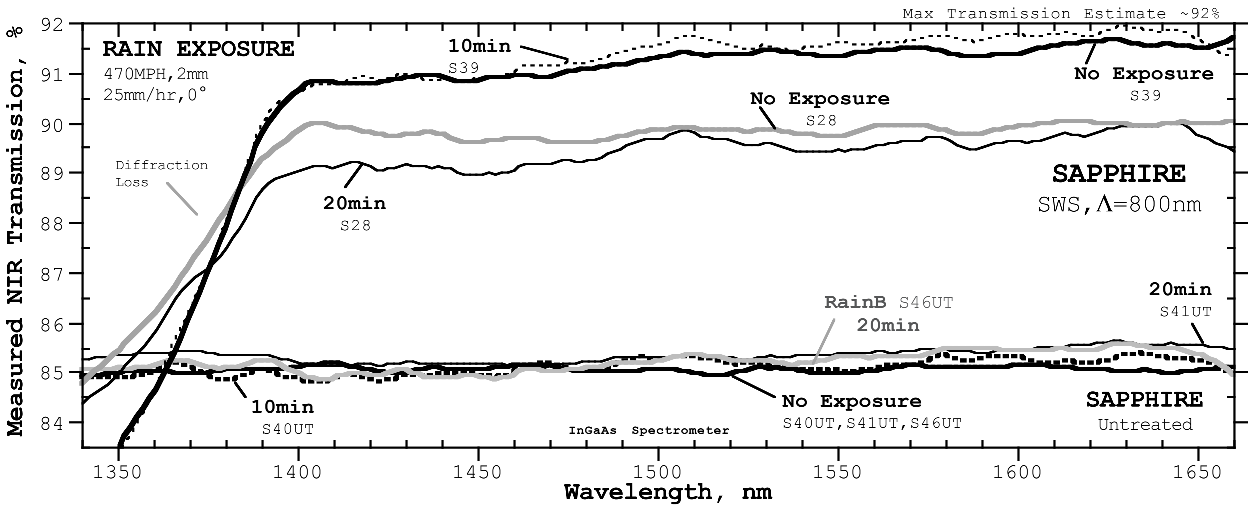


Figure 11: Measured NIR Transmission of untreated and ARMs treated sapphire windows after Rain exposure.

Figure 11 shows the NIR transmission of the sapphire windows before and after the rain exposures. Each curve represents the average of multiple scans at multiple locations over the window surface. Within the measurement system accuracy, no change in the measured transmission is found for the 5 samples tested after the rain exposures, a result that is consistent with the visual appearance of the windows. Further characterization of the window transmission over the 2 to 5 micron wavelength range using an FTIR spectrometer, also show no change in transmission due to rain exposure.

4. EXPERIMENTAL – PULSED LASER DAMAGE THRESHOLD TESTS

ARMs treated fused silica and glass windows have been shown to be capable of transmitting high energy laser light without damage at power levels up to five times higher than windows treated with thin-film AR coatings. There are many high power laser systems operating at NIR wavelengths that are required to perform in harsh environmental conditions. ARMs treated sapphire windows may offer the combination of high transmission, environmental durability, and high laser damage threshold needed for laser communications, laser radar, and laser weapon systems.

NIR PULSED LiDT TESTING AT 1573nm

Both binary SWS-type and pyramidal-profile Motheye-type ARMs textures were fabricated in sapphire and ALON windows. The ARMs textures were designed for broad-band NIR transmission with peak performance near 1550nm. Using a laser system operating at 1573nm, calibrated, NIST traceable, standardized pulsed laser induced damage threshold (LiDT) tests were conducted by Jeff Runkel at Quantel / Big Sky Laser, Inc. in Bozeman Montana on two ARMs treated sapphire windows, two ARMs treated ALON windows, and two untreated windows of each material.

Figures 12 and 13 show the transmission of each of the test windows over the NIR spectral range of 1350 to 1700nm. The measurements were made with the InGaAs spectrometer system described above. Overhead and elevation views of the ARMs treated windows taken by SEM are inset in the figures. In each plot the dashed black curves give the untreated window transmission, the solid gray curves give the transmission of SWS ARMs treated windows (mesas), and the solid black curves give the transmission of the Motheye ARMs treated windows (cones). The vertical gray bar in each plot marks the laser wavelength for the damage tests. All of the transmission curves represent an average of multiple scans taken at multiple locations over the sample surface. The three sapphire windows are 25mm round by 6mm thick supplied by Crystal Systems of Salem, Massachusetts. ALON samples were acquired from Surmet Corporation where the ARMs treated ALON windows are 25mm round by 2mm thick, and the untreated ALON window is 10mm square by 2mm thick.

Because surface preparation is critical for attaining adequate laser damage thresholds for materials in high power laser systems, all of the untreated and ARMs treated sapphire and ALON windows were cleaned prior to the damage testing with a standard acid (H_2SO_4:H_2O_2) immersion and solvent rinse followed by a nitrogen blow dry to remove any surface contaminants introduced during the fabrication or characterization processes. All parts were then shipped to Big Sky Laser for LiDT testing at 1573nm. Big Sky exposed more than 100 locations on each window to 10 different fluence levels using a 1573nm 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 (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.

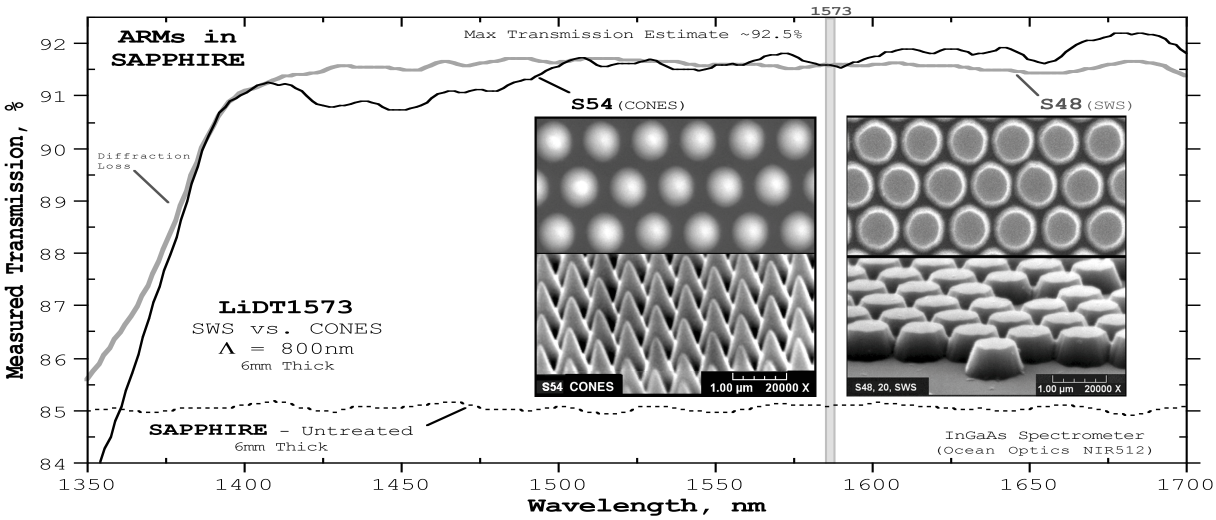


Figure 12: Measured NIR transmission of ARMs treated sapphire windows intended for LiDT tests at 1573nm.

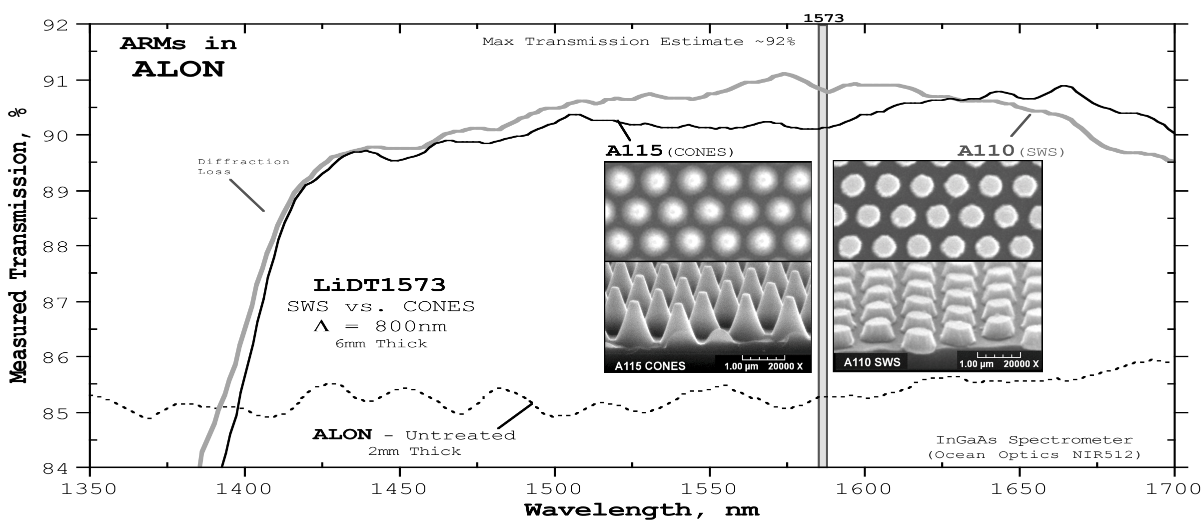


Figure 13: Measured NIR transmission of ARMs treated ALON windows intended for LiDT tests at 1573nm.

The results, plotted in Figure 14, show the percentage of sites damaged (the damage frequency) as a function of the applied laser fluence (the laser intensity on the sample surface is also given on the top axis). Each data point is a compilation of the data from 10 sites over the sample surface, and are marked as triangles for the cone ARMs treated samples, squares for the SWS ARMs treated samples, and diamonds for the untreated samples. The lines are a linear fit to the data with solid black lines representing the cone ARMs treated windows, solid gray lines indicating the SWS ARMs treated windows, and the dashed black lines the untreated windows. Damage thresholds for the SWS and Motheye ARMs treated sapphire windows are 18.8 and 19.5 J/cm^2 respectively, and 26.3 J/cm^2 for the untreated sapphire window. These thresholds compare favorably to the 33 J/cm^2 values found for untreated and ARMs treated fused silica windows[7], and are a factor of 2 greater than catalog data found for thin-film AR coatings advertised as high damage threshold coatings. Surprisingly, the damage thresholds found for all three ALON window variants follows the same trend as the sapphire window variants, but is more than four times less than that found with the sapphire windows. This indicates some fundamental difference between the two material's properties as opposed to a sample preparation or ARMs fabrication issue.

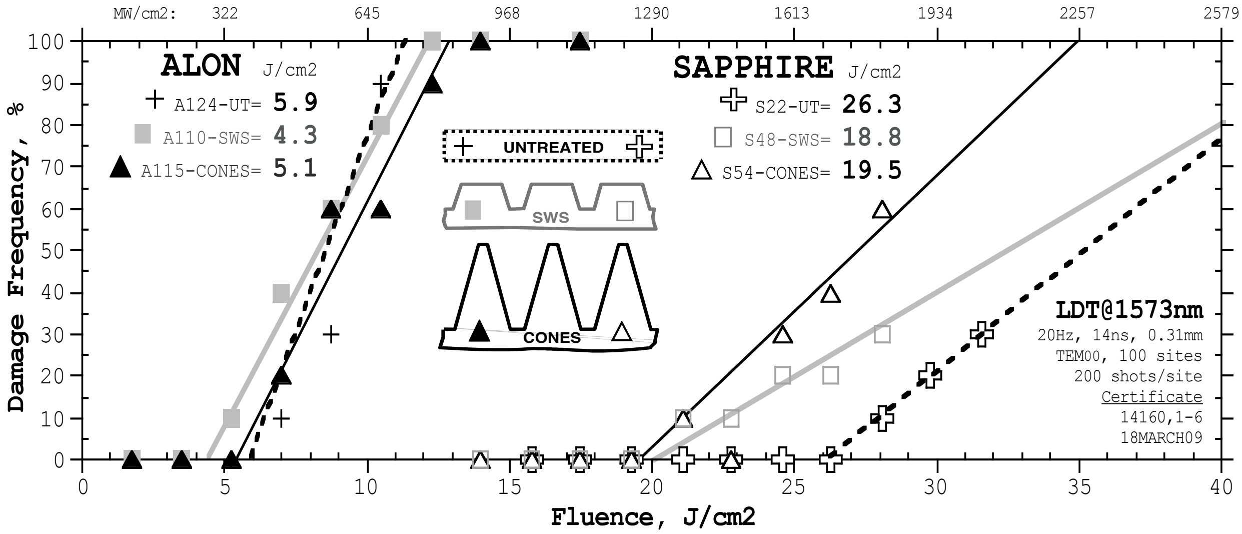


Figure 14: Measurement of the LiDT for untreated and ARMs treated sapphire and ALON windows at 1573nm.

Figure 15 shows overhead views of typical damage sites in both the ARMs treated ALON (left hand images) and sapphire (right hand images) windows resulting from the pulsed laser exposures. As might be expected from the damage threshold results, the nature of the surface damage is quite different with the two materials. At an exposure energy just one quarter the level of the sapphire exposures, the extent of damage is much larger for the ALON windows possibly due to failure at the polycrystalline grain boundaries (compare the top row images at 100X magnification). At a magnification of 1000X (bottom row images), jagged edges and smooth underlying surfaces are found with the ALON damage, whereas the sapphire surface damage has a fused or melted appearance.

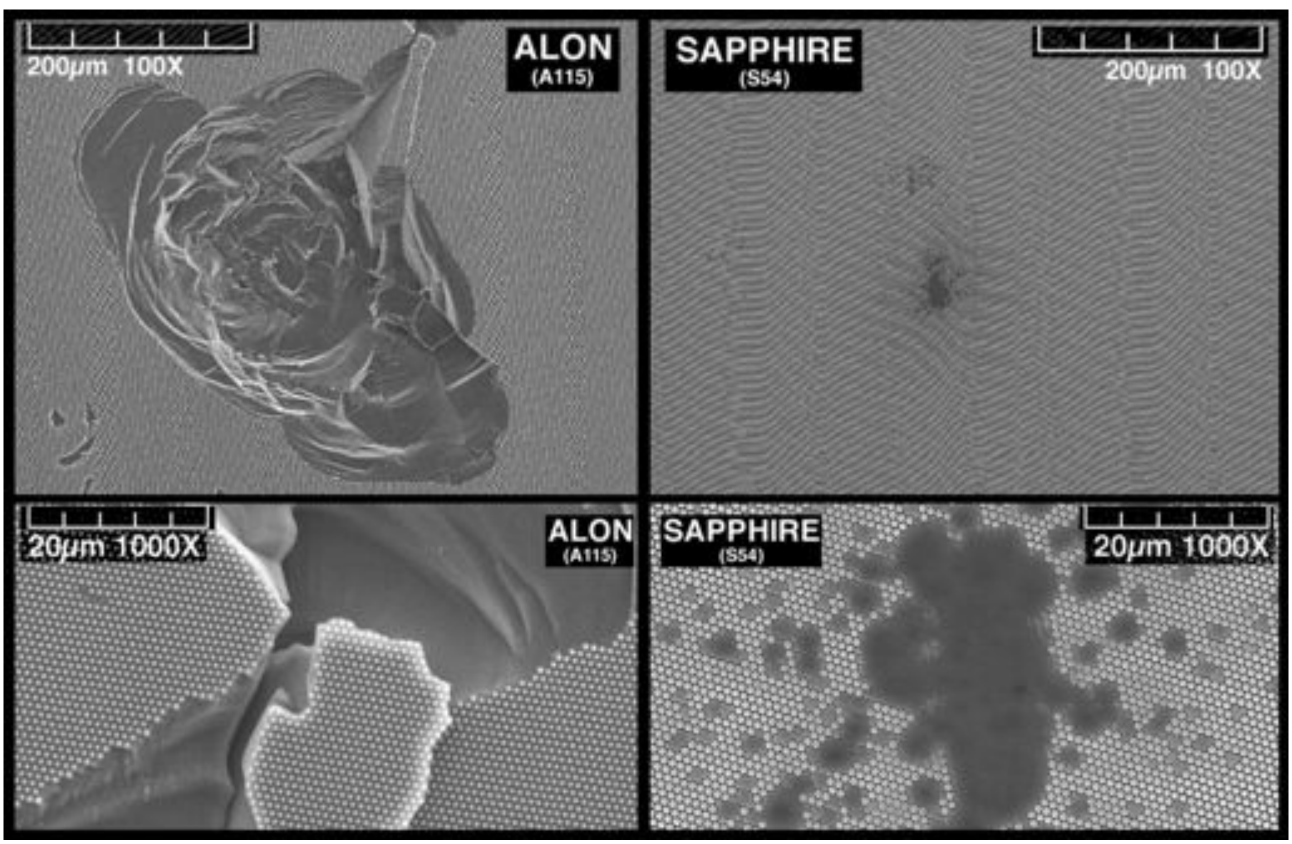


Figure 15: SEM images of the surface damage caused by the high energy laser exposures.

LWIR PULSED LiDT TESTING AT 9.56µm

The favorable thermal, mechanical, optical and radiation resistant properties of clear diamond make the material attractive for use in high power laser systems operating at long wave infrared wavelengths. With the recent development of an ARMs fabrication process for diamond, the potential to realize enhanced laser damage threshold diamond windows incorporating ARMs textures can be investigated. Small window fragments of clear diamond material were provided by Lockheed Martin, sp3 Diamond Technologies, and BAE Systems. Two types of ARMs textures designed for broad-band LWIR performance peaked at a wavelength of 10 µm, were fabricated in one surface of three diamond window fragments. Both post-type SWS and hybrid-cone type ARMs textures similar to those shown in Figure 1 above, were fabricated. Figure 15 shows the transmission of the three ARMs treated diamond windows (solid gray and black curves) along with an untreated window (dashed black curve) over the LWIR wavelength range of from 6 to 15 µm. Measurements were made using a Nicolet Model 550 FTIR spectrometer.

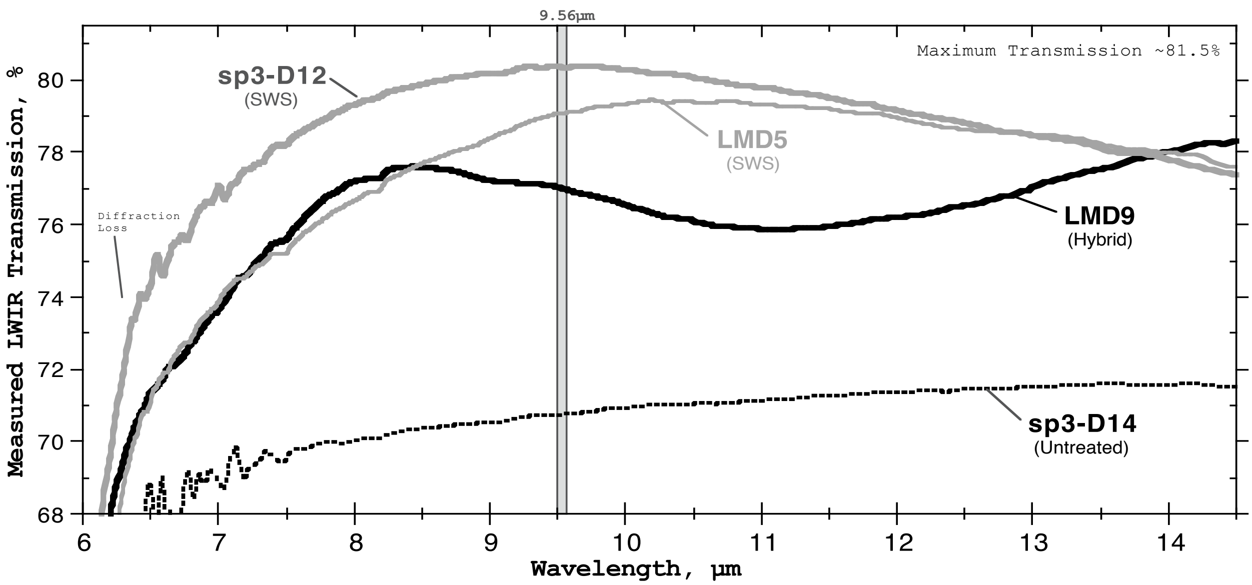


Figure 15: Measured LWIR transmission of ARMs treated diamond windows intended for LiDT tests at 9.56µm.

Under the direction of Dr. Shekhar Guha, AFRL's Laser Hardened Materials Evaluation Laboratory located at Wright Patterson Air Force Base in Ohio, operates a pulsed carbon dioxide laser system for testing the damage threshold of optical materials. All of the diamond window samples were cleaned by an acid bath immersion followed by solvent rinsing and an oxygen plasma cleaning cycle prior to shipment. Leonel Gonzalez configured and calibrated the test setup for a 9.56µm wavelength, 100ns pulse width, 4Hz repetition rate, and a beam spot diameter that was varied between 49 and 94 µm. Typically the test involves exposing up to 100 locations on each sample to as many as 10 fluence levels to determine an accurate damage threshold. Because of the small area of the diamond samples (each was less than 10x10mm), the number of fluence levels applied and the number of locations exposed needed to be reduced. In addition, it proved to be quite difficult to damage the diamond materials at the fluence levels attainable with the usual system configuration (spot size), a problem that required several calibration cycles that used a portion of the sample area. As a result, the number of exposed locations on each sample was limited to less than 20, with only three locations exposed to the same fluence. Two test cycles were required since the untreated diamond samples used for calibration could not be damaged at the highest fluence level using the initial spot size. The final results, given as a range of threshold values in Table 1, show a great amount of variation due to these limitations. However, the minimum values listed in the table are very high relative to other materials like silicon tested in a similar manner, and demonstrate the potential for ARMs textures in diamond to provide a highly durable window or optic.

Table 1: Pulsed Laser Damage Threshold Data for ARMs in Clear Diamond.

DIAMOND@9.56µm	Spot Size	Untreated - D14	SWS AR - D12	SWS AR - LMD5	HYBRID AR - LMD9
LiDT (J/cm2)	94µm	50-75	50-75	70-140	50-75
LiDT (J/cm2)	49µm	300-400	170-220	110-170	80-110
Window Source:		sp3	sp3-BAE	Lockheed Martin	Lockheed Martin

5. SUMMARY

An investigation of the optical and environmental durability of microstructure-based anti-reflection treatments applied to the important window materials sapphire, ALON, and diamond, has been made. It was found that the enhanced transmission attained from AR microstructures in sapphire can be expected to be maintained for a lifetime equivalent to that expected for an untreated sapphire window. A pulsed laser damage threshold two times higher than typical thin-film AR coatings, was measured for AR microstructures in sapphire at a wavelength of 1573nm. Unexpectedly, the pulsed laser damage threshold of untreated ALON windows and ALON windows incorporating AR microstructures, was measured at a level four times less than that found for sapphire. Finally, the pulsed laser damage threshold for clear diamond windows at a wavelength of 9.56µm in the LWIR, was investigated. While the results were quite variable, the levels found were very high indicating great potential for the use of AR microstructures in diamond for high power military and industrial lasers.

6. ACKNOWLEDGEMENTS

The authors would like to thank Charles Blair of UDRI for his helpful advice and careful maintenance and operation of both the particle erosion and whirling arm rain rigs. Jerry Zimmer of sp3 Diamond Technologies, Mike Evans of BAE Systems, and Kelly Choban of Lockheed Martin all provided clear diamond window fragments for the laser damage testing. AFRL's LHMEL facility at WPAFB, managed by Leo Gonzalez and Dr. Shekhar Guha, provided the laser damage testing of the ARMs treated diamond windows at 9.56µm. Derek Upchurch and Jacob Barnes calibrated the laser exposure system and collected the data. Jeff Runkel of Quantel / Big Sky Laser, Inc. provided the certified, NIST traceable, pulsed LiDT testing at 1573nm. Several dozen microstructure prototypes were fabricated with hard work from Ernest Sabatino, III. SEM analysis was performed as a service by John Knowles at MicroVision Laboratories, Inc., (978-250-9909).

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