# Initial Erosion Studies of Microstructure-Based Anti-Reflection Treatments in Zinc Sulfide

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## ABSTRACT

Results are presented of a preliminary investigation into the mechanical durability of anti-reflecting (AR) microstructures built in the surface of zinc sulfide windows. The goal of the work was to advance the long held concept that the erosion resistance of AR microstructures will be comparable to or better than the resistance of the material of which the microstructures are built. Such durability of AR microstructures could yield windows that transmit more light over longer time periods when operating in abrasive environments, than any type of AR treatment based on thin-film coatings. Five different types of AR microstructures designed for high transmission in the long wave infrared region were fabricated in the surface of both regular grade zinc sulfide (ZnS) and multi-spectral zinc sulfide (ClearTran™) windows. Over 90 treated and untreated ZnS windows were exposed to increasing loads of sand particles traveling at high speeds, and to rain drops traveling at high speed for varying time periods. The calibrated exposures were made with the facilities of the University of Dayton Research Institute, UDRI. It was found that the transmission of several types of AR micro-textured windows degraded at an equal or lower rate than untreated windows for increasing levels of sand particle impacts. AR microstructures such as arrays of hole structures that contain some level of mutual support, were better able to maintain a high transmission than arrays of isolated post structures for a given sand load. In addition, the data indicates that the transmission reduction due to sand impacts of AR microstructures built in the harder ZnS windows, was less than the transmission reduction found with AR microstructures built in the softer ClearTran™ windows. With high speed rain drop impacts, the damage to a window or optic is typically found just below the polished external surface. The results of the rain drop impacts on the surface of windows incorporating AR microstructures shows that the textures may be effective at dispersing the force of the rain drop impact so as to minimize or eliminate sub-surface damage. ZnS windows with pyramidal profile Motheye AR microstructures traveling at 470MPH for 20 minutes through a rain field consisting of 2mm diameter drops falling at a rate of 25mm/hr, showed no sub-surface damage and only minor surface damage leading to a transmission loss of less than 2%. Based on this initial data, it is expected that AR microstructures built in hard materials such as ALON™, spinel, and sapphire, could combine the wide bandwidth and high transmission typical of AR microstructures with a greatly enhanced operational lifetime. Lastly, the merging of AR microstructure and hard coating technologies may expand the range of window material choices for cost sensitive applications.

**Keywords**: Antireflection, AR, Motheye, Microstructures, Mechanical Durability, Rain and Sand Erosion, LWIR

## 1. INTRODUCTION

The operational lifetime of windows employed in the protection of sensors and laser communications systems in military aircraft and ground equipment is limited due to damage introduced by the environment. In particular, rain and sand impacts cause an increase in light scattering from the windows that can rapidly reduce the image contrast in sensor systems or reduce the signal to noise ratio of laser communications systems. A further lifetime reduction is introduced by damage to the often less durable anti-reflection (AR) treatments needed due to the high reflectivity of most infrared transmitting windows – a reflectivity that increases dramatically when the window needs to conform to an aircraft body.

Single, or multiple layer thin-film interference coatings are currently employed to reduce reflections in many military windows. In abrasive environments, the needed performance of thin-film AR coatings degrades abruptly producing scattered light that can often reduce the visibility through the window to a level below that of an untreated window exposed to similar conditions. This problem has led to the elimination of thin-film AR coatings in many applications or the use of low performing single layer coatings of durable materials such as diamond like carbon, and metal oxides. As

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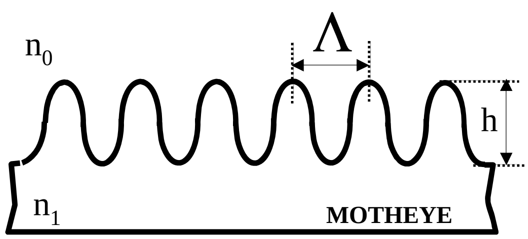
a consequence, there exists a tradeoff between AR performance, lifetime, and cost that cannot accommodate many critical applications, particularly windows used in long wave infrared (LWIR) imaging and communications systems.

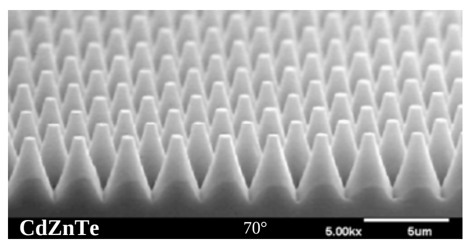
An AR treatment based on surface relief micro-structures built directly into a window material, has demonstrated superior AR performance with higher transmission over broader bandwidths than thin film coatings in many applications[1-6]. Specifically, AR microstructure technology offers significant advantages over thin-film AR coating technology for high power laser systems[7], and devices operating in environments with high levels of radiation[8]. An initial (2004) rain and sand erosion test of AR microstructures built in ALON™ windows, showed little to no damage or loss in transmission at levels well beyond the range where thin-film AR coatings failed. The goal of this work is to further define the potential for microstructure-based AR treatments to enhance the performance and survivability of windows and optics operating in abrasive conditions.

## 2. BACKGROUND - MICROSTRUCTURE BASED ANTI-REFLECTION TECHNOLOGY

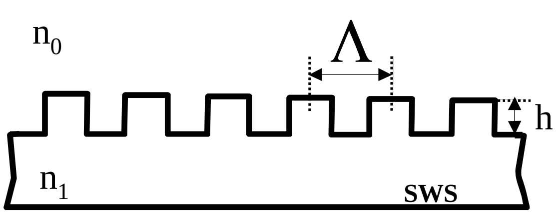
Three distinct types of surface relief AR microstructures, commonly known as Motheye, SWS, and Random textures are under development. The term Motheye is literally derived from the eye of nocturnal moths that have evolved an AR microstructure as shown in the SEM images on the right to avoid detection by their main predator, the owl[9]. Each type of structure has unique optical properties and mechanical characteristics that can be tailored for specific materials and applications. A brief outline of the three structure types is given here;

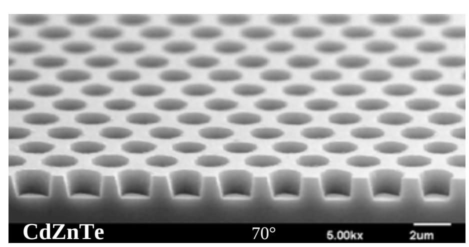
**1) MOTHEYE AR Structures:** The Motheye structure is an array of surface depressions or pyramidal protrusions that has been described extensively in the literature[10-13]. A tapered surface structure provides a gradual change of the refractive index for light propagating from air into the bulk optic material. Reflection losses are reduced to a minimum for broad-band light incident over a wide angular range. A typical Motheye texture profile is depicted below left where the height h and the spacing Λ are indicated. A scanning electron microscope (SEM) image of Motheye structures fabricated in the surface of a cadmium zinc telluride (CZT) window, are shown on the right below.



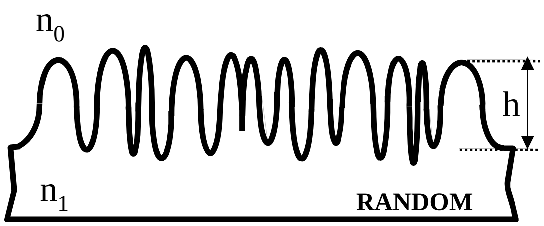



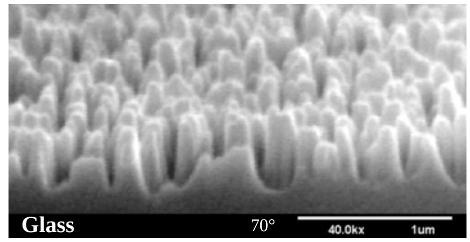

**2) SWS Effective Index Structures:** A Sub-Wavelength Structure, or “SWS” effective index texture is depicted in the profile diagram and SEM image below. An array of holes or posts provides an AR function that is equivalent to a single layer thin-film coating. The effective index is set to the optimum index for a particular optic or window material, which is the square root of the material index of refraction. This is accomplished by tailoring the texture fill factor - the proportions of solid and open areas in the surface. Structure height h is then set to one quarter-wave optical thickness at the effective index. At one wavelength reflections are completely eliminated, and over a narrow wavelength band reflections are suppressed to very low levels. Dual and triple-band AR performance can be obtained with deeper structures set at multiples of the quarter-wave optical thickness.



**3) Random Texture AR Microstructures:** TelAztec has developed a simple fabrication process for AR textures that have a random distribution of sub-wavelength sized surface features. The very small and dense features, as shown in the figures below, provide AR properties that are both extreme and broadband. Advantages of the Random AR texture include the cost driven benefit of eliminating the separate microstructure lithography step, and the ability to apply the structure conformal to challenging topologies, such as micro-lenses and MEMS. Random AR structures built in glass, plastic, and silicon windows exhibit extreme AR performance for visible and NIR light.




To achieve high performance AR with surface relief microstructures, optical phenomena such as diffraction and scattering must be avoided. This requires that the surface structures be fabricated with a feature spacing ($\Lambda$ in the figures above) smaller than the shortest wavelength of operation within the material for a given application. In addition, for Motheye and Random AR structures, the height and cross sectional profile of the surface features must be sufficient to ensure a slowly varying density change. In general, AR microstructures will exhibit characteristics similar to the material in which they are built with respect to mechanical durability, thermal issues, laser power handling capacity, and radiation resistance. The problems with thin-film AR coating adhesion, stress, off-axis performance degradation, durability and lifetime, are eliminated.

## 3. AR MICROSTRUCTURES – PROTOTYPE FABRICATION

In order to demonstrate a link between the mechanical durability of a window material and the mechanical durability of AR microstructures built from the same material, two types of window materials with different hardness levels were needed. Zinc sulfide (ZnS) and multi-spectral, or clear ZnS (ClearTran™) windows were chosen because of their similar chemical and optical properties and their extensive use in significant military and commercial products. Sixty ZnS windows each 25 millimeter (mm) round and 6mm thick were purchased from Phoenix Infrared of Lowell Massachusetts and polished to a surface figure specification of 80-50 scratch-dig and 1-5 waves RMS flatness. Rohm and Haas Company of Woburn Massachusetts provided 40 ClearTran™ windows with a similar surface polish. Pyramidal cross section Motheye textures and both post- and hole-type SWS AR textures were fabricated in one surface of the ZnS and ClearTran™ windows using custom interference lithography techniques and proprietary dry etch transfer processes[14-15]. The microstructures were fabricated on a honeycomb grid with a spacing of 2.7 micrometer (µm) that allowed for high performance AR over the LWIR spectral range from 7 to 14 µm. Figure 1 shows SEM (50° elevation, 5000X) images of the three microstructure variants. Motheye cone structures are etched to a height, h of 3.2 µm, whereas SWS-HOLE and SWS-POST structures are etched just 1.6 µm deep. A thin (0.3 µm) coating of yittrium oxide (Y2O3) was deposited in a conformal manner on half the microstructured window samples to form a second set of variants designated with a label prefix of P-. It was thought that the hard Y2O3 coating might provide additional durability by the rounding of sharp features and by filling in polycrystalline domain boundary surface cracks.

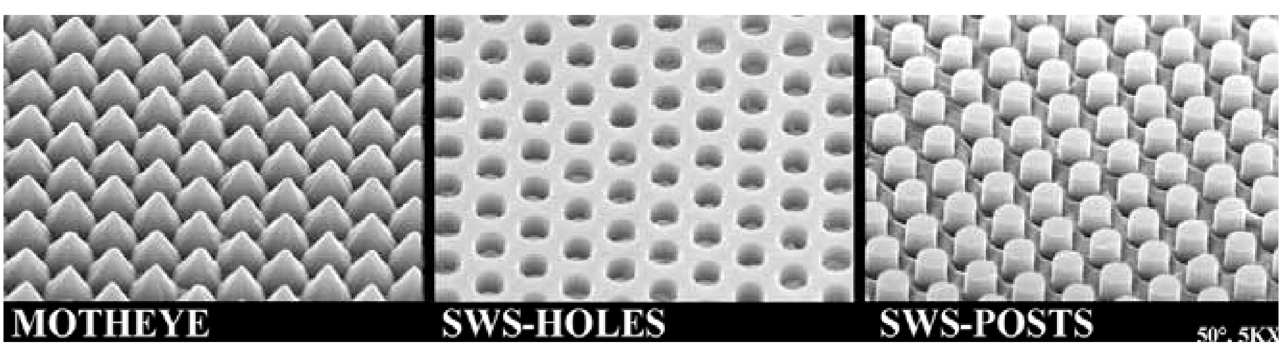



**Figure 1: SEM images of pyramidal and binary profile AR microstructures fabricated in the surface of ZnS windows.**

The infrared transmission of each of the windows was recorded over the spectral range of 2 to 16 μm using a Nicolet FTIR spectrometer. Figures 2 and 3 show the typical transmission of each of the variants where part-to-part transmission variation was less than 1%. In each of the figures the transmission of an untreated window is given and the scale has been set to indicate an estimate of the maximum transmission attainable through a window with an AR treatment in one surface only (78% for ZnS, 84% for ClearTran™). Also indicated in each figure is the wavelength below which transmission loss due to free space diffraction from the microstructures occurs. This was designed to fall near 6 μm to correspond to the natural ZnS absorption band.

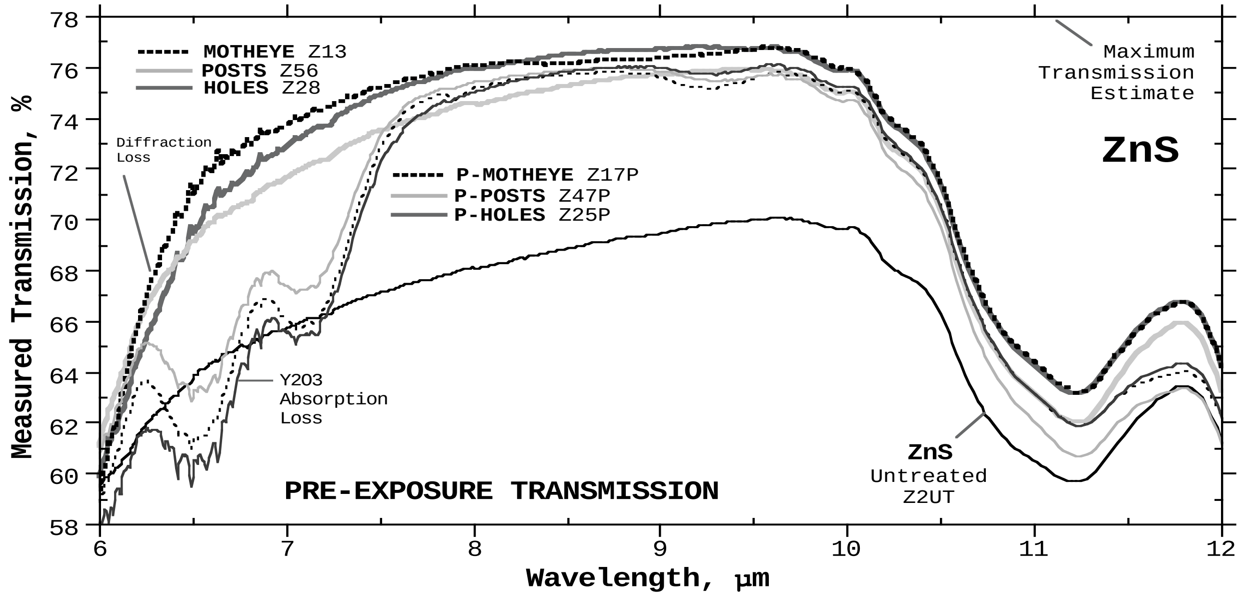



**Figure 2: Typical LWIR transmission of AR micro-structured ZnS window variants prior to erosion testing.**

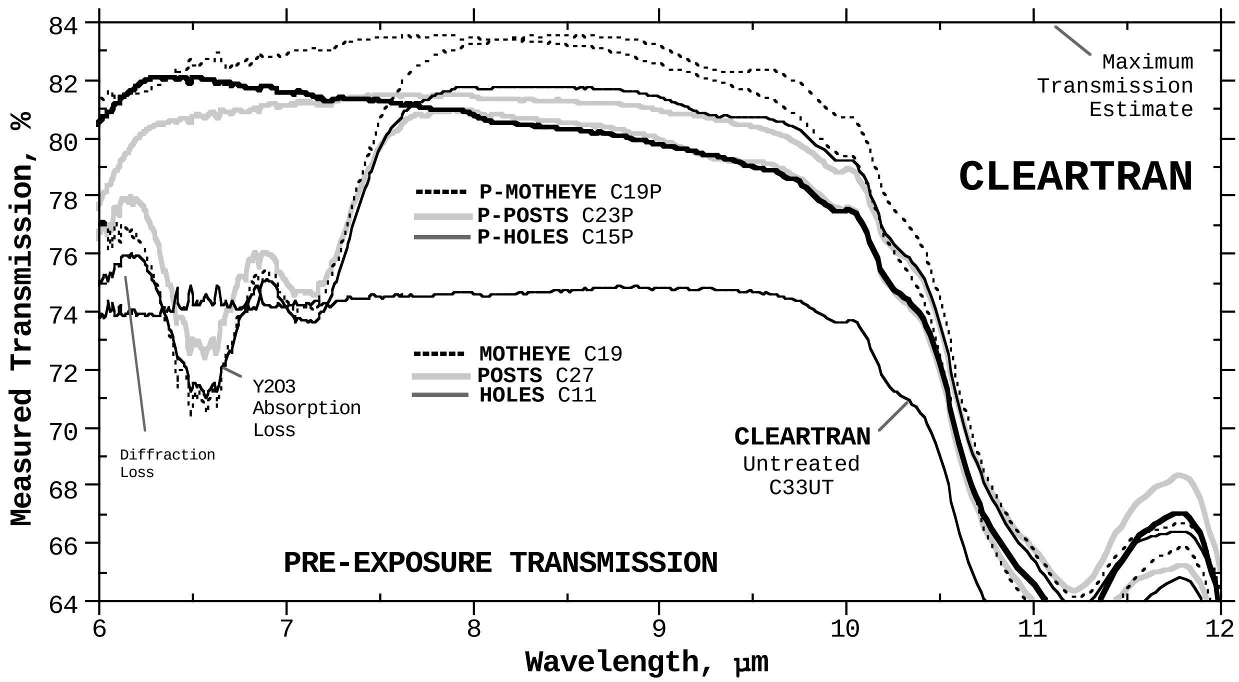



**Figure 3: Typical LWIR transmission of AR micro-structured ClearTran™ window variants prior to erosion testing.**

## 4. 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 flight conditions. Both rigs are described in detail in documents provided on UDRI's website[16-17]. A matrix of sand and rain conditions was developed for exposing each type of AR microstructure fabricated in both ZnS and ClearTran windows. The details are given below beginning with the sand erosion testing;

### SAND EROSION TESTING OF AR MICROSTRUCTURES

The particle erosion rig at UDRI, pictured here on the left, can be configured to deliver a number of different size silica sand particles with speeds that are relevant to conventional aircraft flight 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 such as with ground operations (taxi, takeoff, landing) or with high altitude cruising. The parameters chosen for the AR microstructure exposures were taken from the literature, in particular reference 18, with some modification as a result of the system calibration. Large sand particles with a diameter ranging from 125 to 177 µm, traveling at 135 meters per second (m/s, or 302 miles per hour, MPH) were designated as Sand Condition A and may be relevant to the conditions found during aircraft ground operations. Small sand particles with a diameter ranging from 38 to 53 µm, traveling at 270 m/s (605 MPH) were designated as Sand Condition B and may be relevant to the conditions found during flight. Sand particles were directed perpendicular to the window surface for both Conditions, and three levels of exposure were specified for each Condition, for a total of six exposures, or machine cycles. For Sand Condition A the dose levels were 10, 20, and 30 mg/cm$^2$, and for Sand Condition B the dose levels were 4, 8, and 12 mg/cm$^2$. With the UDRI equipment, sixteen 25mm diameter windows can be exposed during each machine cycle. An experimental matrix was generated where each of the AR microstructure variants was exposed to a single level. Untreated windows were included in each cycle. Figure 4 shows untreated ZnS and ClearTran windows for each exposure level, where the uniformity of the damage is indicative of a well calibrated apparatus. The visual appearance of the AR microtextured windows is similar.

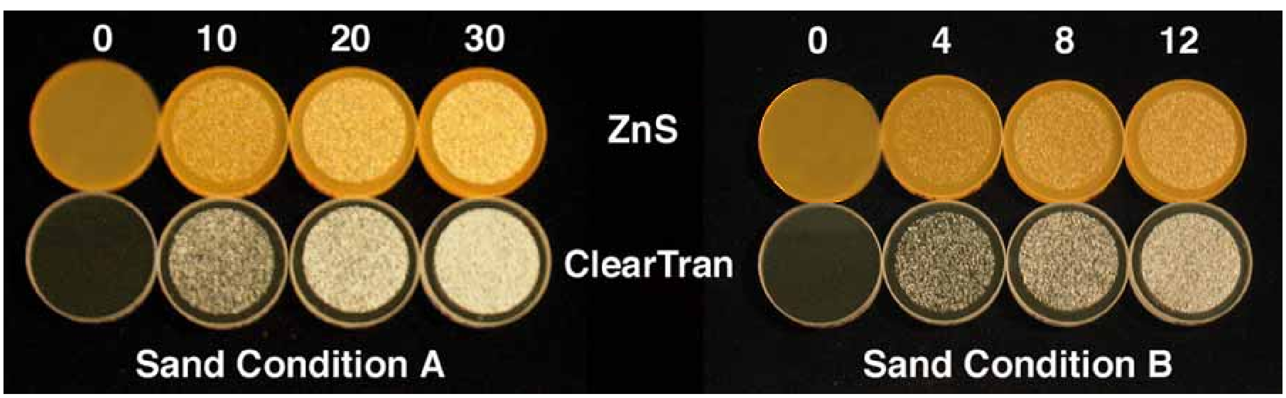
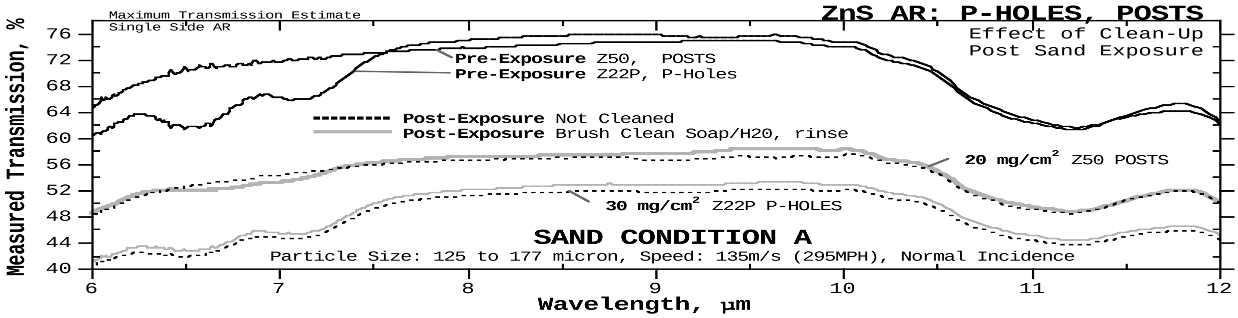
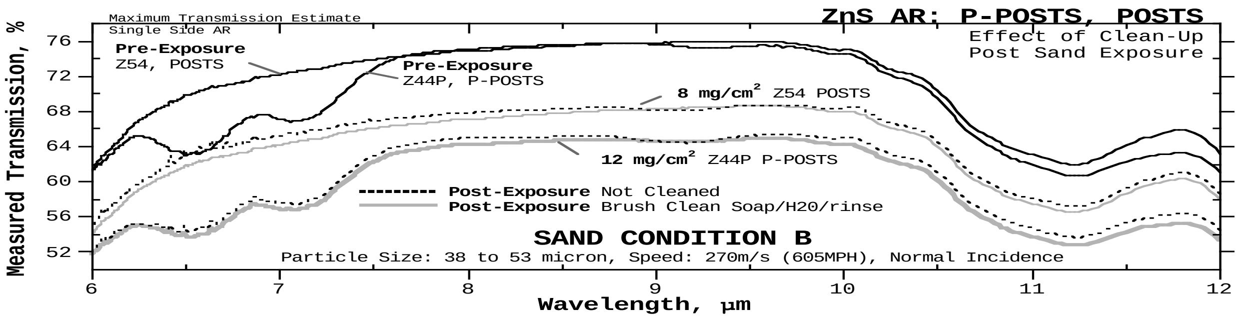



**Figure 4: Untreated ZnS and ClearTran windows after sand exposure in the UDRI particle erosion rig.**

With surface relief textures there has been concern that particle debris will become lodged in the microstructure valleys causing a loss in transmission. There is also a concern that the textures cannot be cleaned in a practical manner. To investigate these concerns the transmission of the samples exposed in the particle rig was recorded immediately after exposure, and again after an aggressive physical cleaning with standard dish soap in water and a nylon tipped toothbrush. Figures 5 and 6 show that there is little difference between the transmission of the damaged surfaces before and after cleaning, indicating that further damage to the microstructures is not introduced by practical cleaning methods, and that most debris from the sand impacts does not remain on the surface.




**Figures 5 (top) and 6: Transmission of AR micro-structured ZnS windows before and after cleanup from sand exposures.**

Figures 7, 8, and 9 depict the measured transmission of the untreated and AR microstructured windows exposed by the particle erosion rig. The plots are grouped by the sand condition and by the window material with Figure 7 showing the ZnS windows exposed to Sand Condition A, Figure 8 showing the ZnS windows exposed to Sand Condition B, and Figure 9 showing the transmission of the ClearTran™ windows for both sand conditions. Measurements of the window transmission before exposure are given in each case as the solid black curve at the top of each plot. The lowest exposure level is indicated by the solid gray curves in each plot and the dashed black curves indicate the mid-level exposure. The highest exposure level is typically at the bottom of each plot and is indicated again as a solid black curve. In general the measurements show a uniform drop in the transmission for increasing dust loads over the entire LWIR spectral range. This indicates that the primary transmission loss is due to light scattering that is consistent with their visual appearance. It was noted that transmission losses increased at shorter wavelengths in the near and mid-wave infrared region. This is also consistent with light scattering from the surface damage. SEM images of the surface damage are shown in Figures 10, 11, and 12.

Comparing the results of the two sand conditions, the largest transmission losses are with the large sand particles – Condition A – that seem to have produced impact damage covering a larger area (compare Figs 10 and 11). A second observation found with both sand conditions is that as the particle load increases, the transmission falls at a faster rate for the softer ClearTran™ windows than for the harder ZnS windows. Both results are consistent with the literature and the prevailing industry knowledge. Figure 12 shows SEM images of damage sites in ClearTran™ where one interpretation of the images suggests that a single impact may cause collateral damage by fracturing the surface along the polycrystalline grain boundaries. With a grain size that is 4 to 5 times larger than ZnS grains, fracturing along grain boundaries in ClearTran™ would produce larger damage areas that result in more scattered light loss. For the AR micro-textured ZnS windows it can also be observed that the transmission remains at a higher level than the untreated windows up to the maximum dust loads. In fact the rate of transmission loss for all the variants of AR microstructured ZnS windows is very similar to that found with the untreated windows. Figures 13 and 14 better illustrate the observed trends by plotting the measured transmission through the various windows as a function of particle load. In these plots an average transmission value was calculated for each variant over the spectral range of from 7.5 to 10 µm. Linear fits to the data were estimated and plotted as solid lines in the figures. Values for the untreated windows are plotted as open crosses along with a heavy black line representing the linear fit. *In the majority of AR microstructure variants tested, the linear fit curves run parallel to the untreated curves supporting the concept that an AR treatment based on microstructures can outperform any type of thin film AR coating for longer time periods in abrasive environments.* One notable exception is found with the ClearTran™ windows exposed to large sand impacts where presumably the softer material gives rise to more easily damaged microstructures.

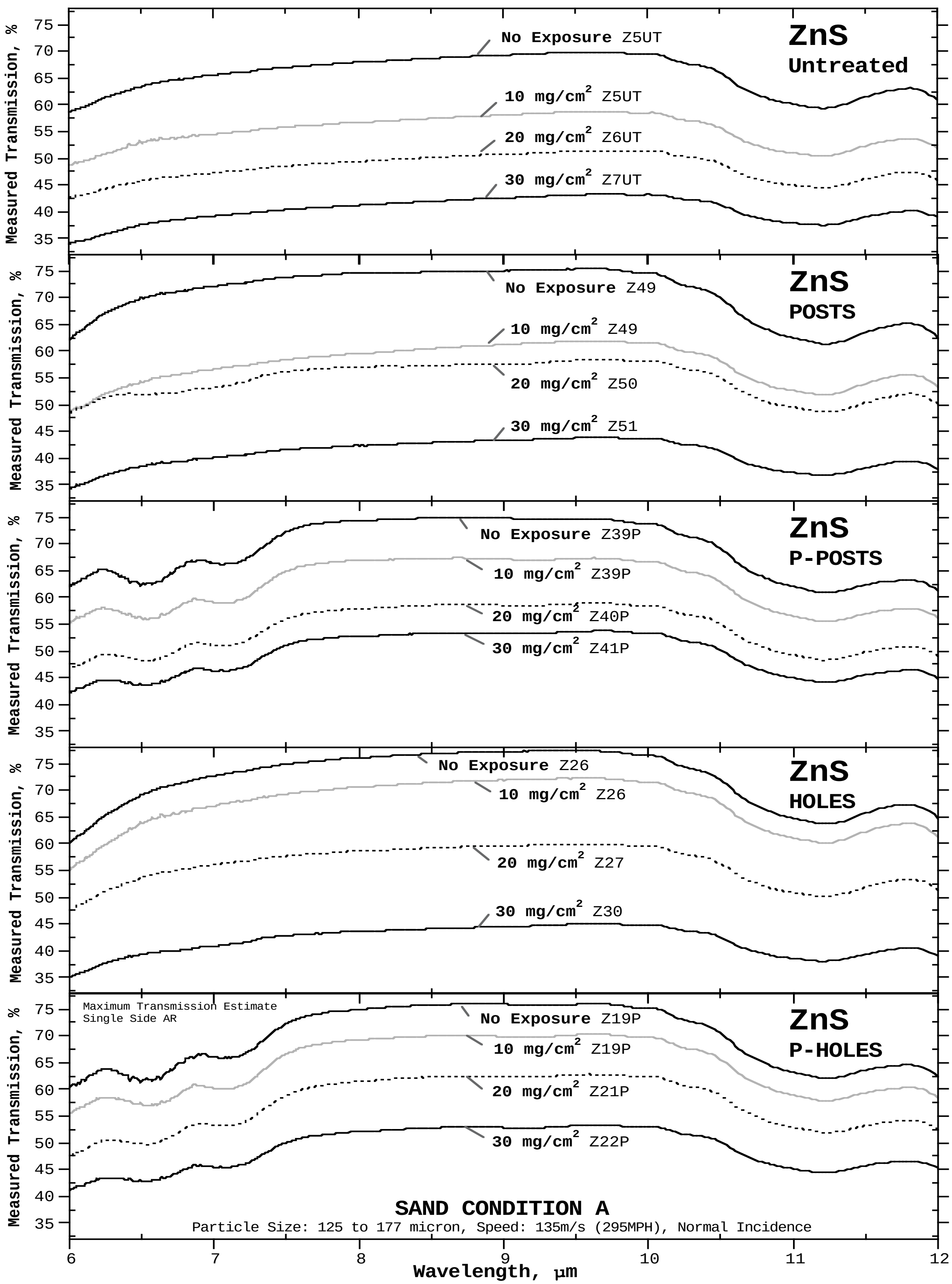



**Figure 7: Measured LWIR Transmission of AR micro-structured ZnS windows exposed to Sand Condition A.**

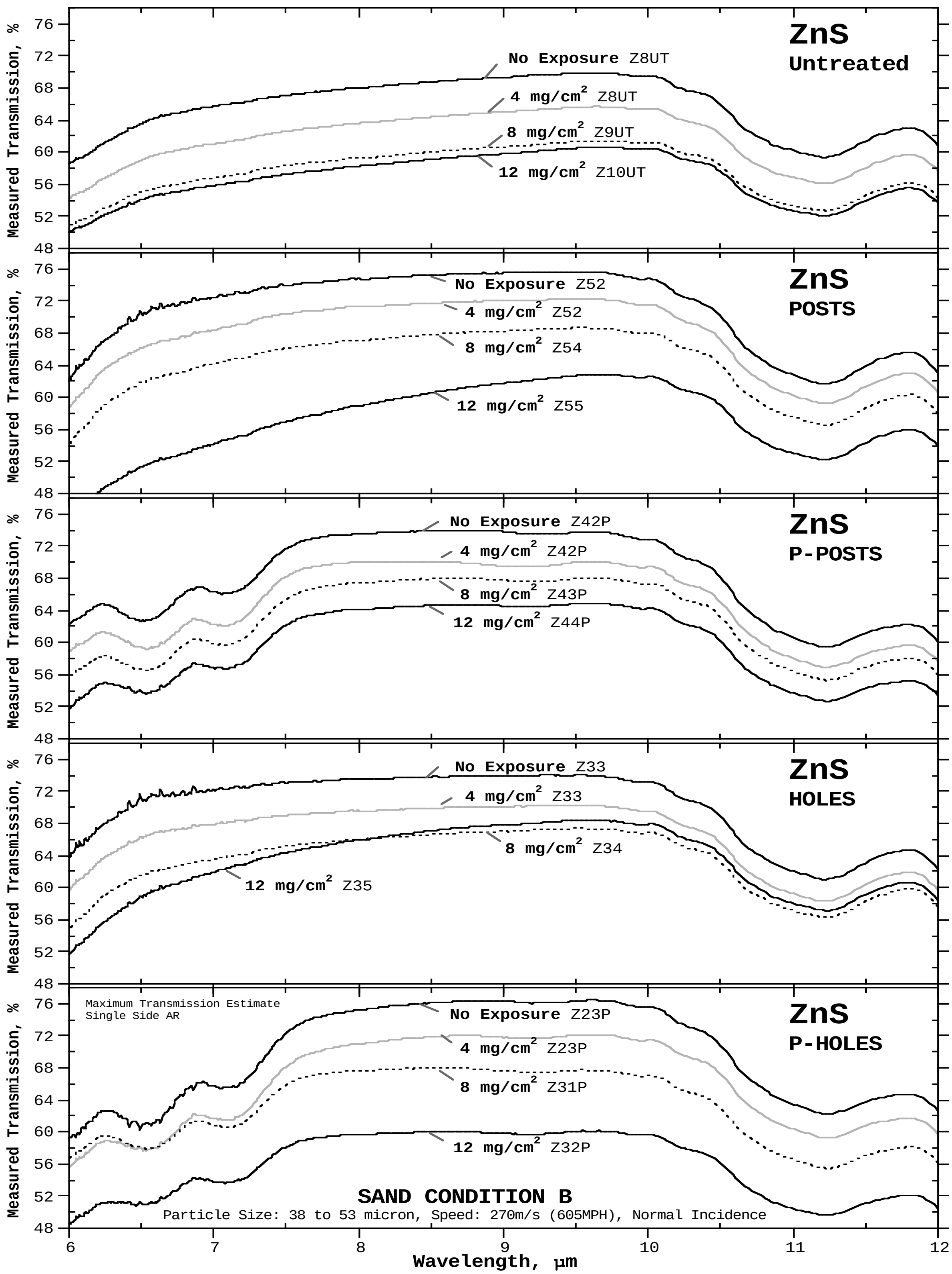



**Figure 8: Measured LWIR Transmission of AR micro-structured ZnS windows exposed to Sand Condition B.**

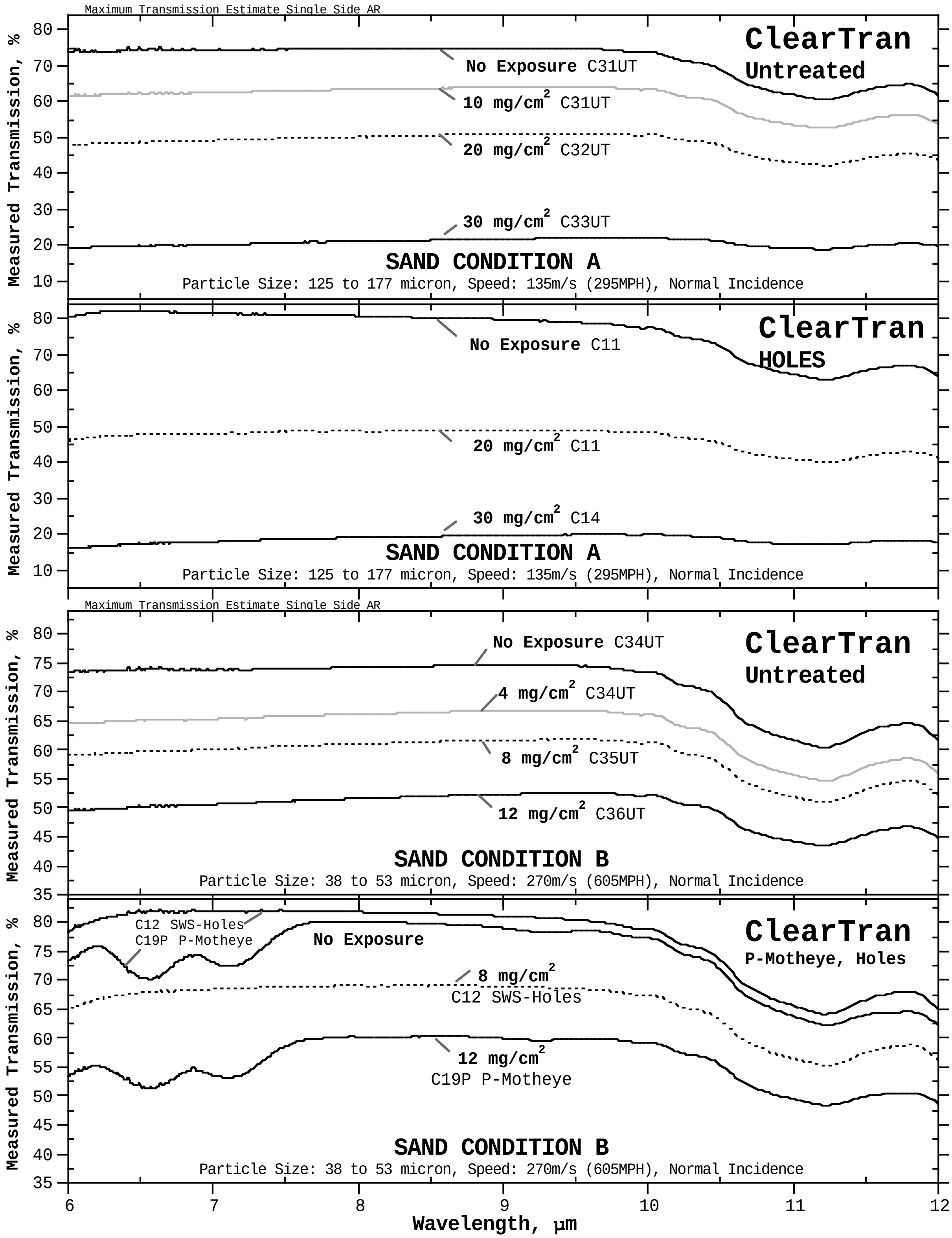



**Figure 9: Measured LWIR Transmission of AR micro-structured ClearTran™ windows exposed to Sand Conditions A&B.**

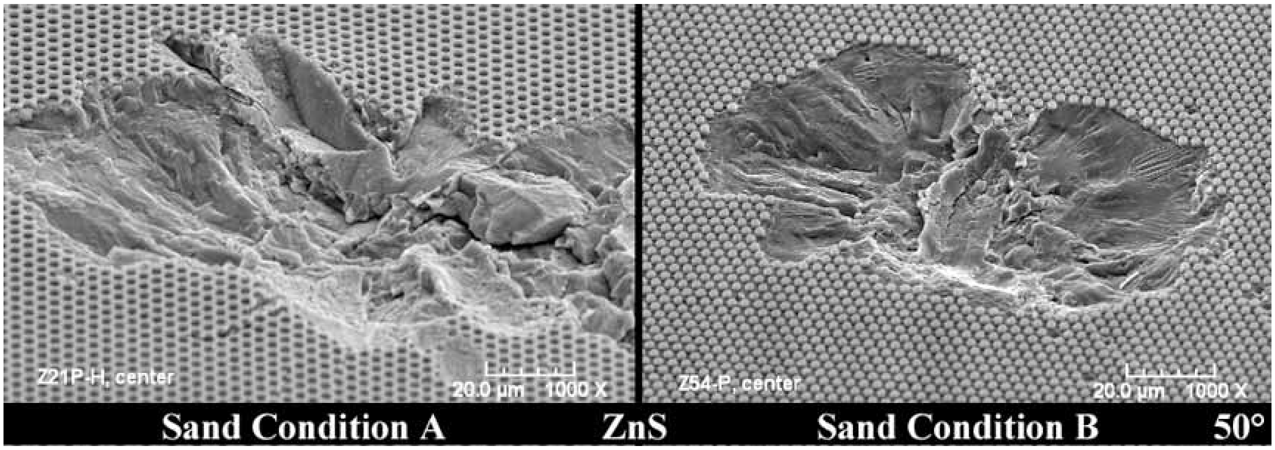



**Figure 10: Elevation views of isolated sand impact areas on AR micro-structured ZnS windows.**

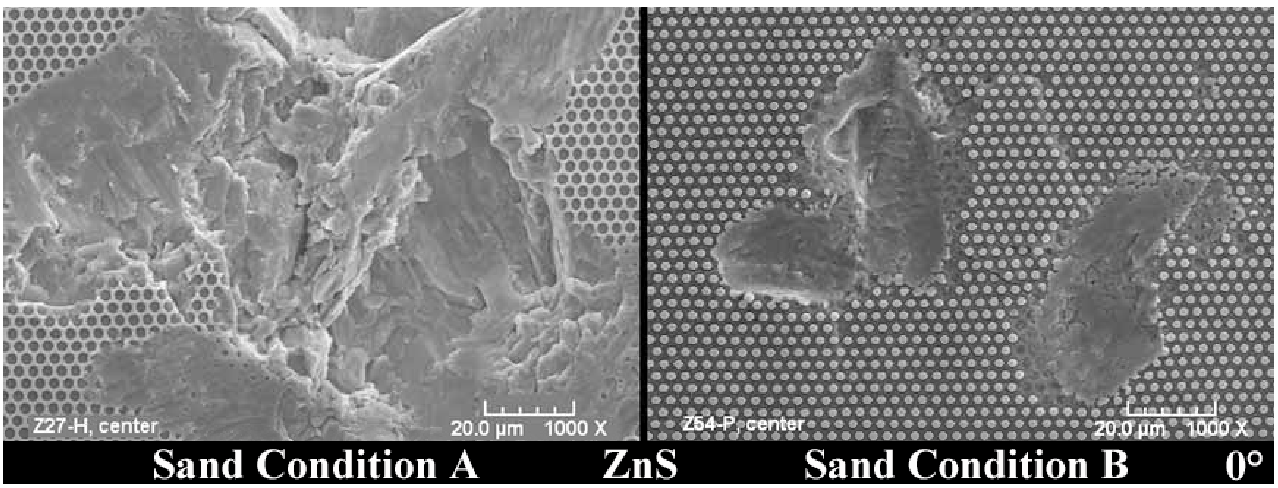



**Figure 11: Overhead views of isolated sand impact areas on AR micro-structured ZnS windows.**

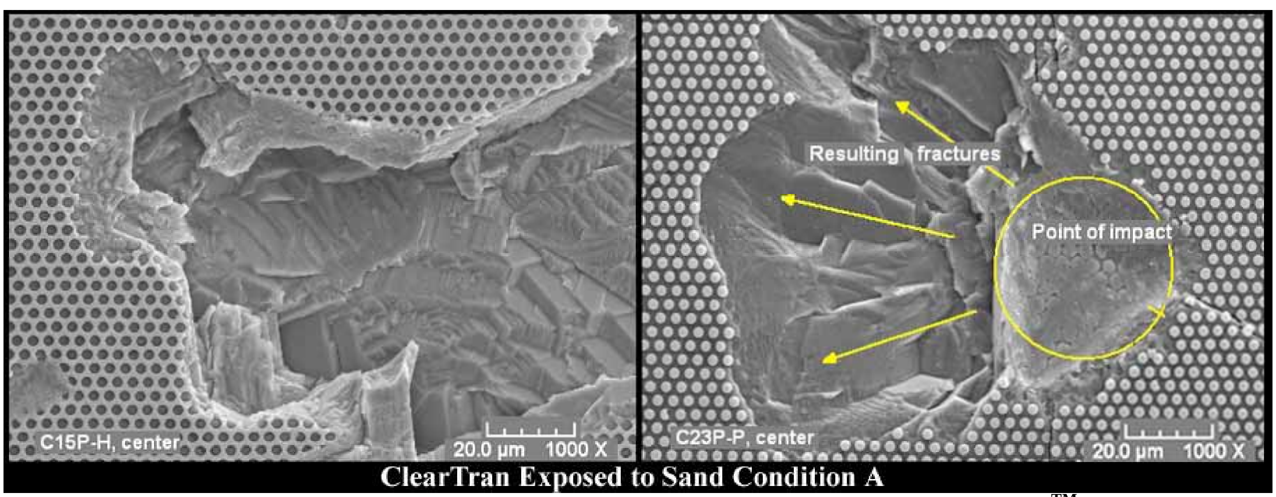



**Figure 12: Overhead views of isolated sand impact areas on AR micro-structured ClearTran™ windows.**

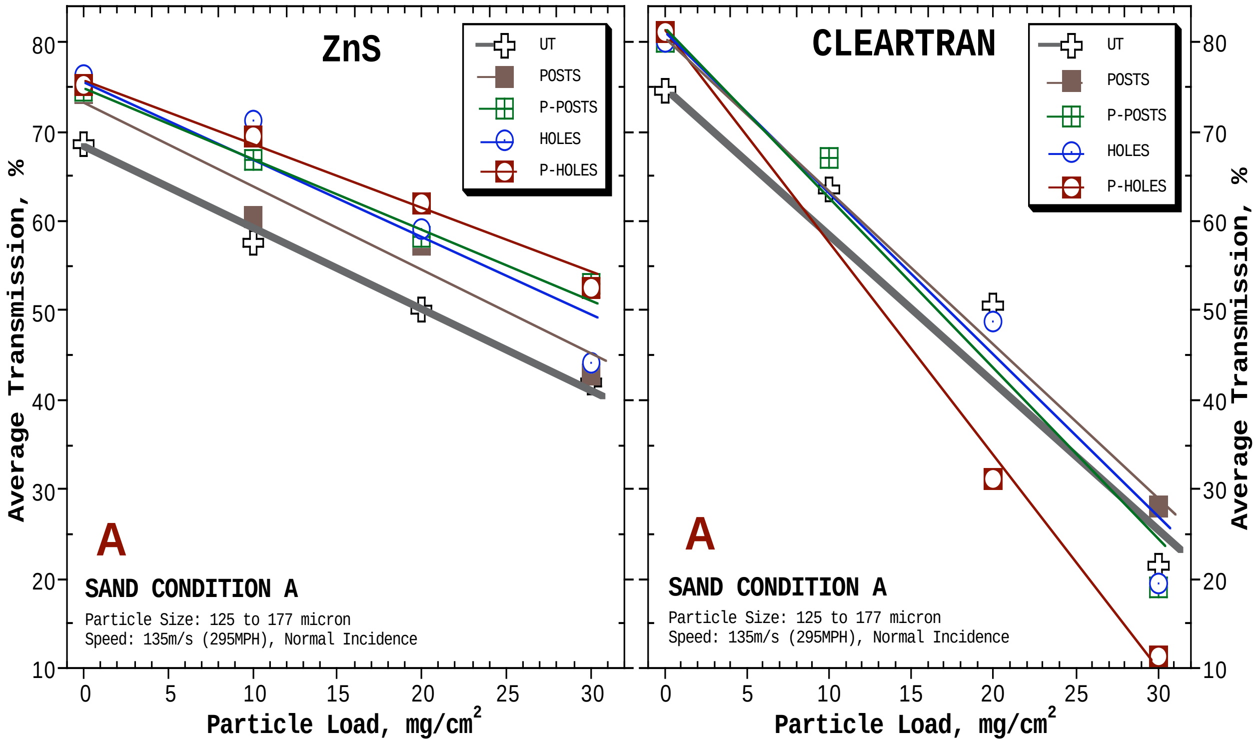



**Figure 13: Average LWIR transmission of AR micro-structured ZnS and ClearTran windows exposed to large sand.**

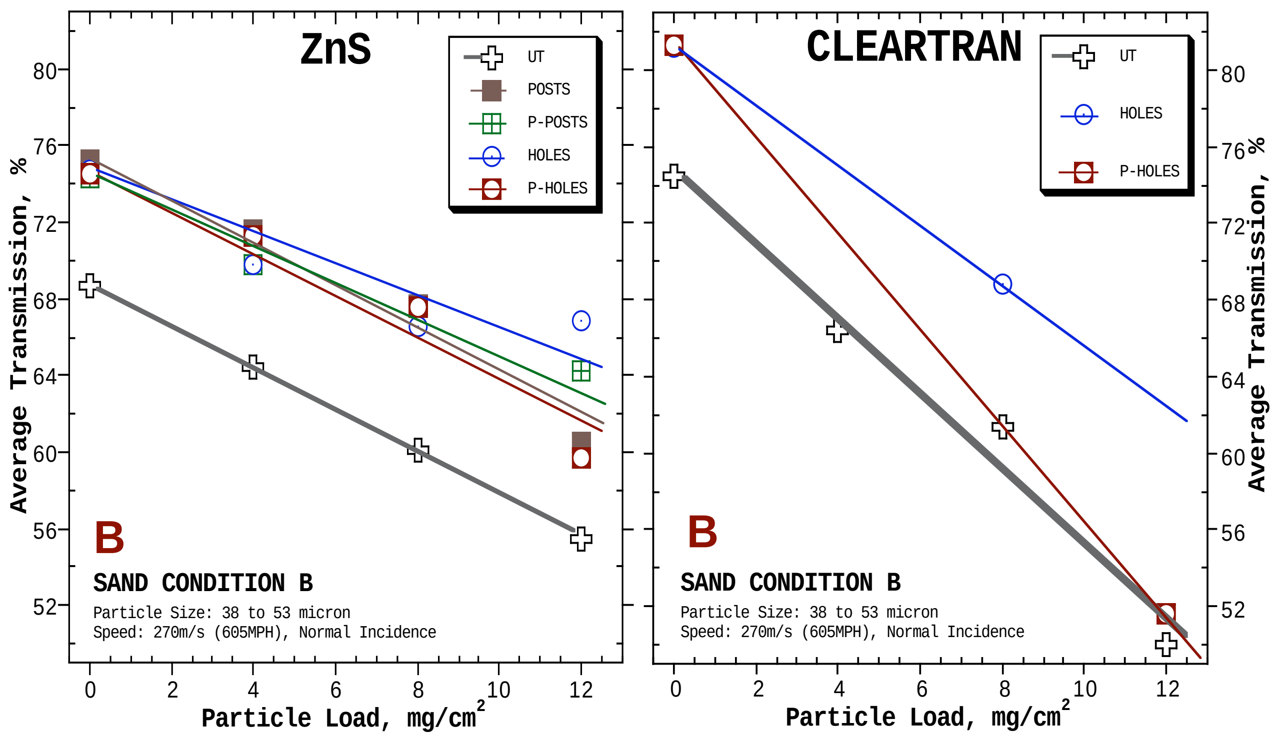
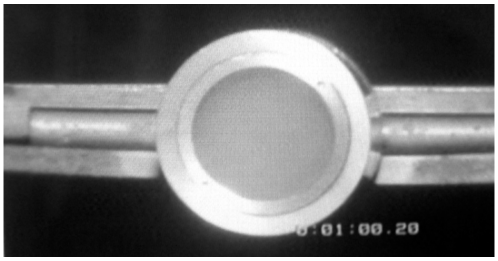



**Figure 14: Average LWIR transmission of AR micro-structured ZnS and ClearTran windows exposed to small sand.**

## RAIN EROSION TESTING OF AR MICROSTRUCTURES




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 speeds that are relevant to conventional aircraft flight conditions 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 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 AR microstructure exposures where an impact angle normal to the sample surface was also chosen. At these settings, the industry expectation, as described in reference 18, 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. An experimental matrix was generated where each of the AR microstructure variants was exposed for two different durations. Untreated windows were included in the testing to provide baseline information.

The nature of the damage caused by a rain drop impact is quite different than the damage caused by sand particle impacts. For untreated ZnS and ClearTran windows, it appears that a rain drop impact on the window surface causes damage just below the window surface, in contrast to a sand impact that removes surface material leaving a crater or crack in the surface. Figure 15 shows untreated ZnS and ClearTran windows after exposure in the UDRI rain rig for varying durations where the internal damage is observed by transmitting white light through the parts using a light table. Light reflecting from the exposed window surfaces remains smooth and specular, and microscopic inspections reveals little to no surface damage. To prevent this type of sub-surface damage that would destroy an expensive window, much work has focused on durable thin-film coatings combined with an elastic, or compliant material layer that can serve as a shock absorber to dampen the force of the rain drop impact. This approach leads to a tradeoff between AR performance and durability due to the limited choice of suitable materials and material deposition and adhesion issues. One of the goals of this work is to test the idea that AR microstructures can prevent sub-surface damage by laterally diffusing the stress wave generated by a rain drop impact. Because high performance AR microstructures can be built in any material for any application, the performance-durability tradeoff with thin-film hard coatings would be eliminated.

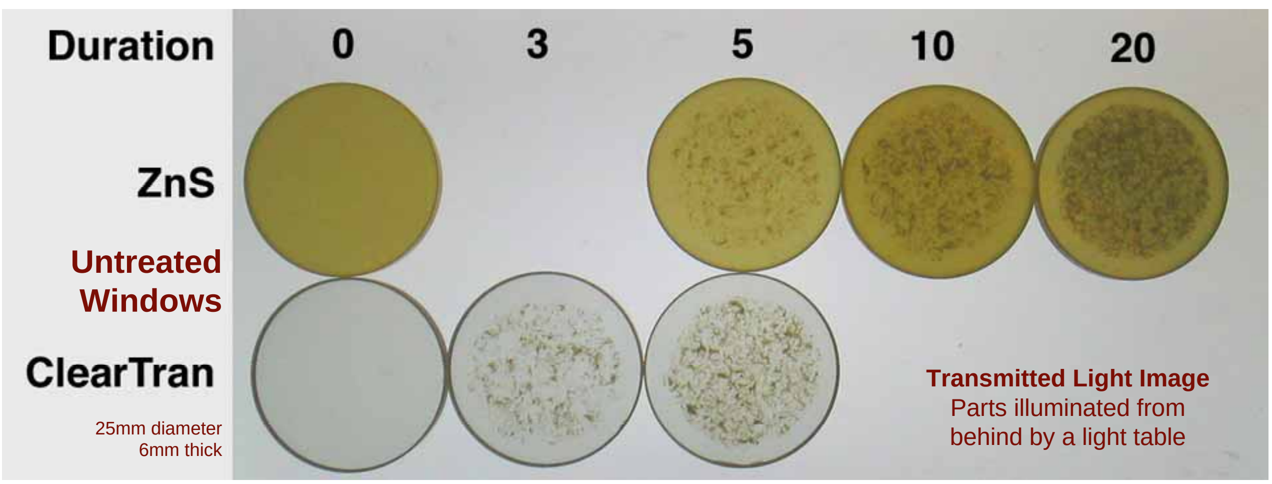



**Figure 15: Untreated ZnS and ClearTran windows after rain exposure in the UDRI whirling arm rig.**

Figures 16, 17, and 18 depict the measured LWIR transmission of the untreated and AR microstructured windows exposed in the rain erosion rig. As with the sand exposure transmission plots, the sample transmission before rain exposure is given as the solid light gray line, and the transmission after the maximum rain exposure duration (20 minutes for ZnS, 5 minutes for ClearTran™) is given as the solid black line. The dashed black lines represent an

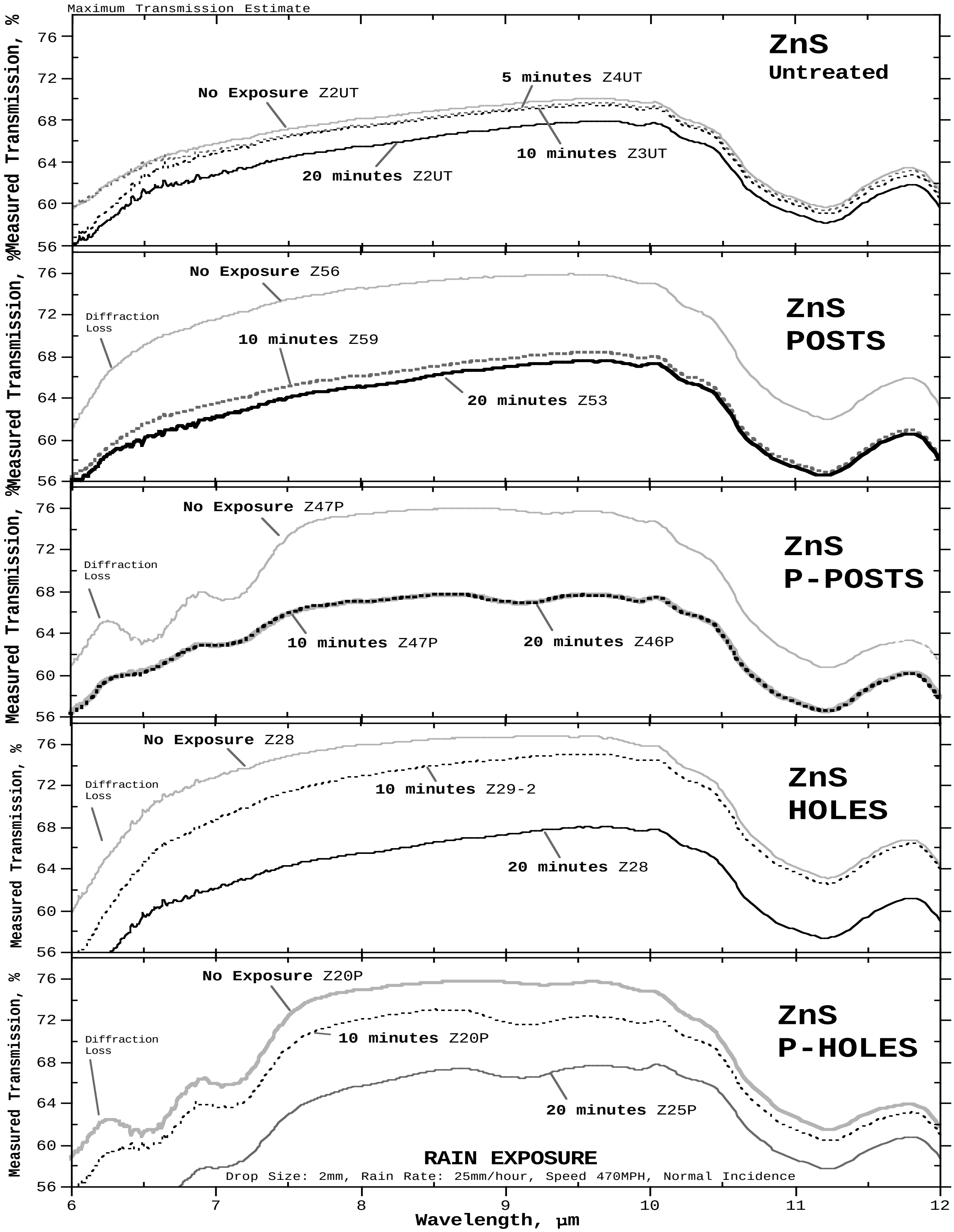



**Figure 16: Measured LWIR Transmission of AR micro-structured ZnS windows after Rain exposure.**

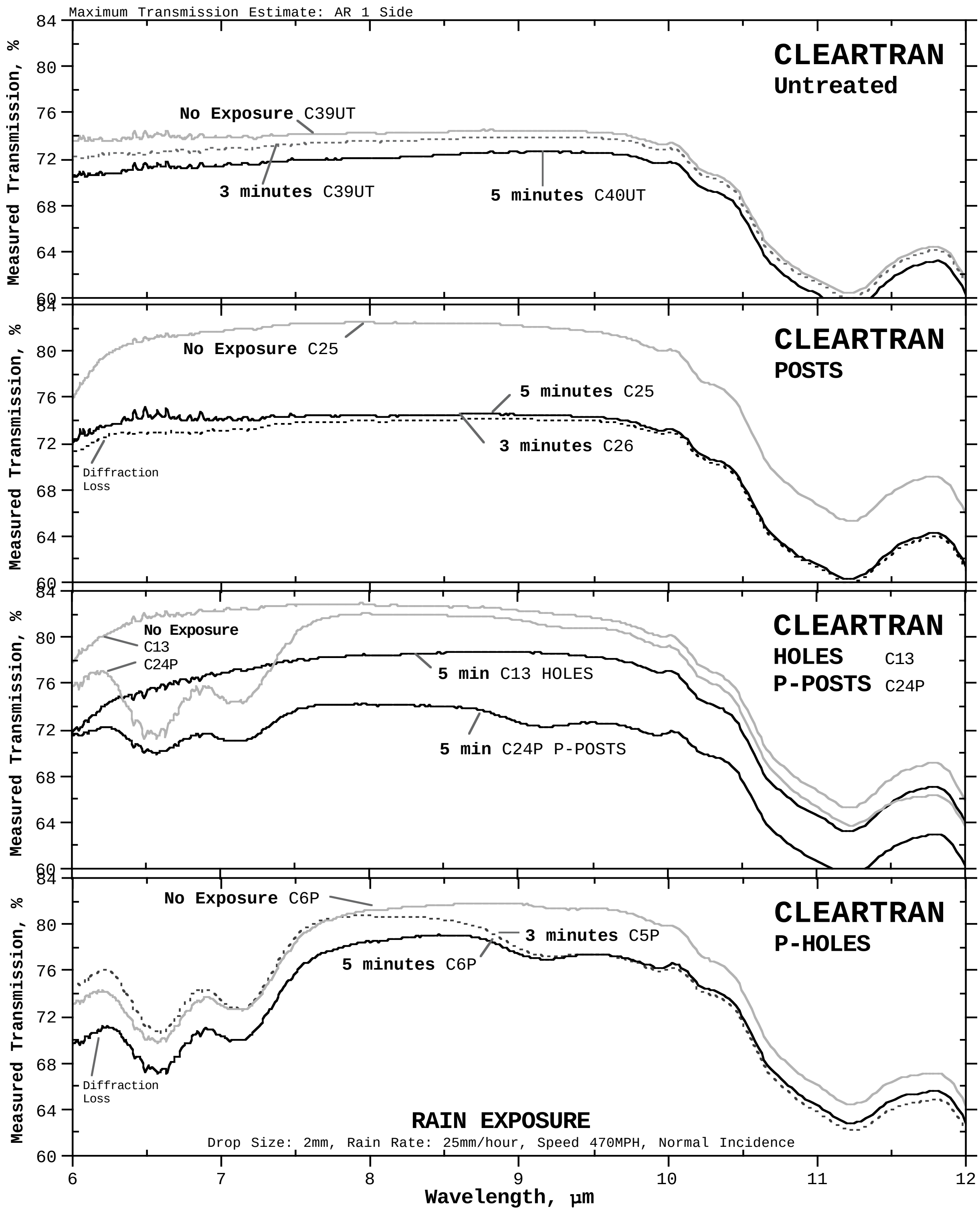



**Figure 17: Measured LWIR Transmission of AR micro-structured ClearTran™ windows after Rain exposure.**

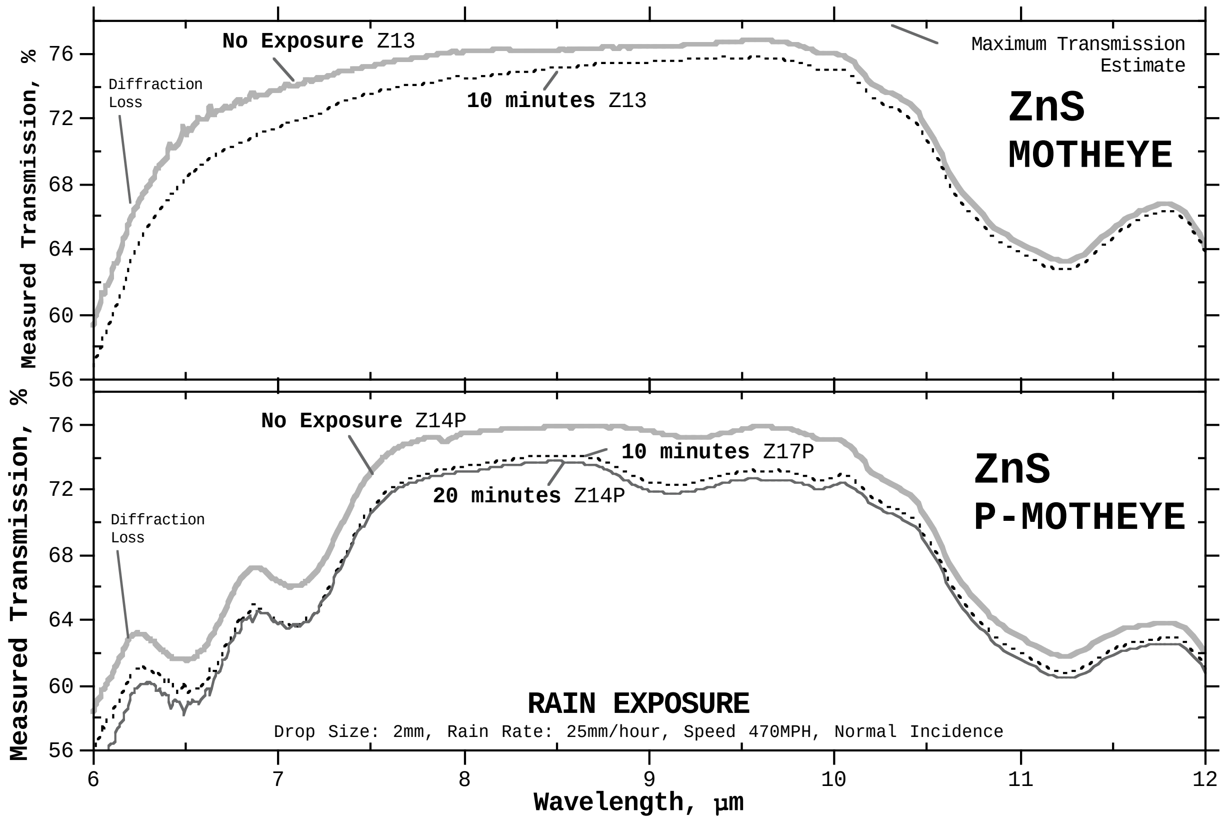
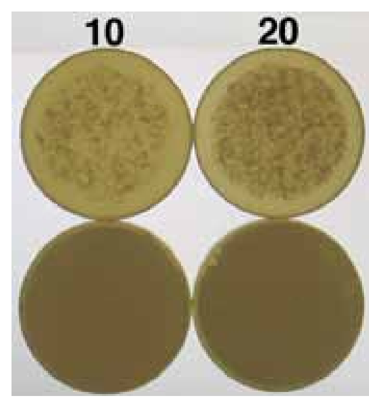



**Figure 18: Measured LWIR Transmission of Motheye textured ZnS windows after Rain exposure.**




intermediate exposure duration of either 10 minutes in the case of the ZnS windows, or 3 minutes for the ClearTran[TM] windows. The results in Figures 16 and 17 show a significant loss in transmission after rain exposure for the binary type SWS AR microstructures with and without the Y2O3 conformal coating. A visual inspection of these windows, two of which are shown at the top of the photograph on the right, shows the same level of internal damage that is observed with untreated windows. In addition, microscopic analysis of the surface of the hole and post structures after rain exposure shows considerable damage, with the post structures suffering the most damage. The SEM images in the bottom half of Figure 19 show that the post structures have been sheared off at the base and removed. This type of damage leads to a rapid loss in transmission early in the simulated life of the window due to the loss of the surface structure AR properties, followed by a more gradual transmission loss as the sub-surface damage accumulates. It appears that the binary type AR microstructures are not effective for diffusing the force of rain drop impacts and thereby preventing sub-surface damage.

In stark contrast, the results for the pyramidal profile Motheye AR microstructures are dramatic. There appears to have been no sub-surface damage in any of the three Motheye textured ZnS windows tested. The visual appearance of two of these windows is shown in the lower half of the photograph on the right above where white light illuminates the windows from behind. The cone structures in the window surface are more than 3 microns deep with a 2.7 micron spacing which leads to their dark appearance in the visible. The measured infrared transmission of these windows confirms that the loss in transmission due to rain exposure is minimal for exposure durations up to 20 minutes. SEM images, given in the top half of Figure 19, showing the Motheye microstructure surface before and after rain exposure suggests that the minimal transmission loss is due to scattered light from slight damage to the surface texture.

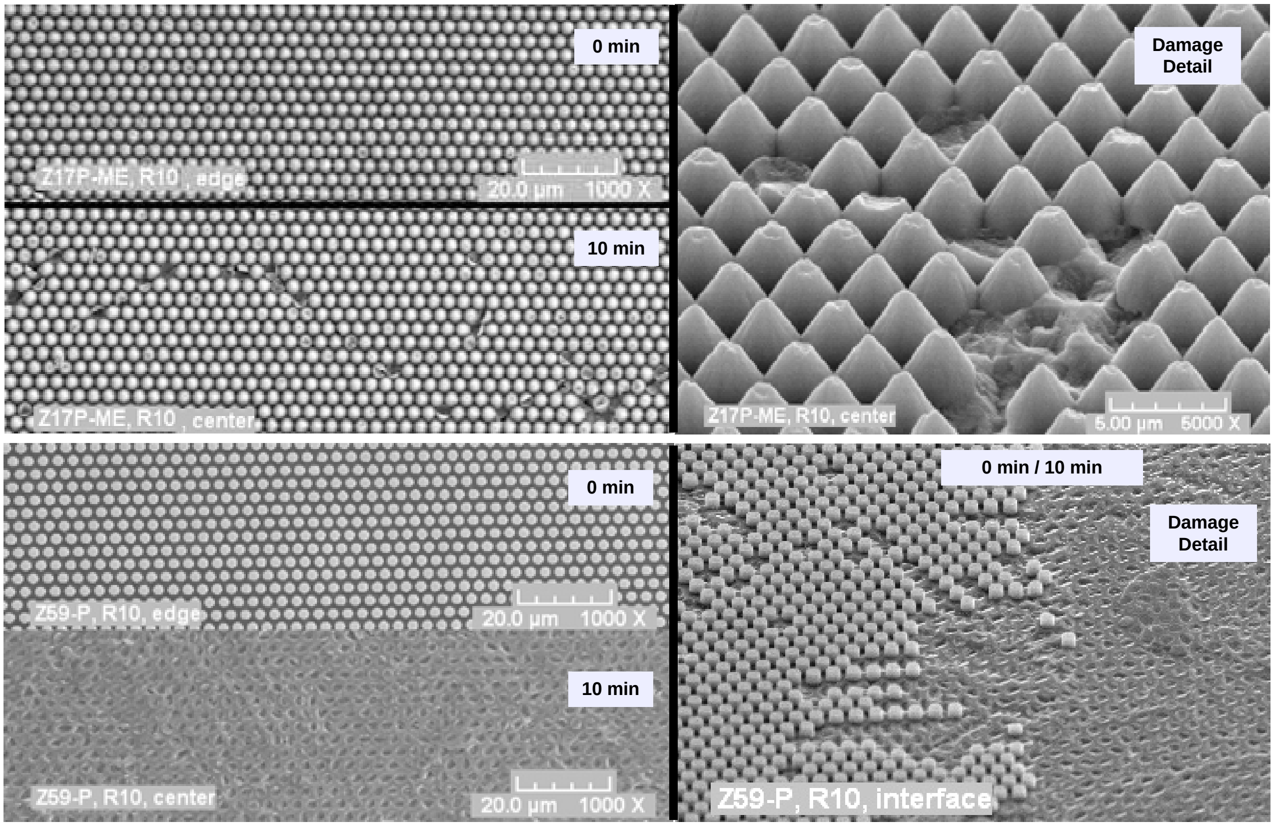



**Figure 19: SEM Images showing the damage found to the AR microstructures after Rain exposure. Top Motheye AR Structures after 10 minutes of Rain. Bottom – SWS Post Structures.**

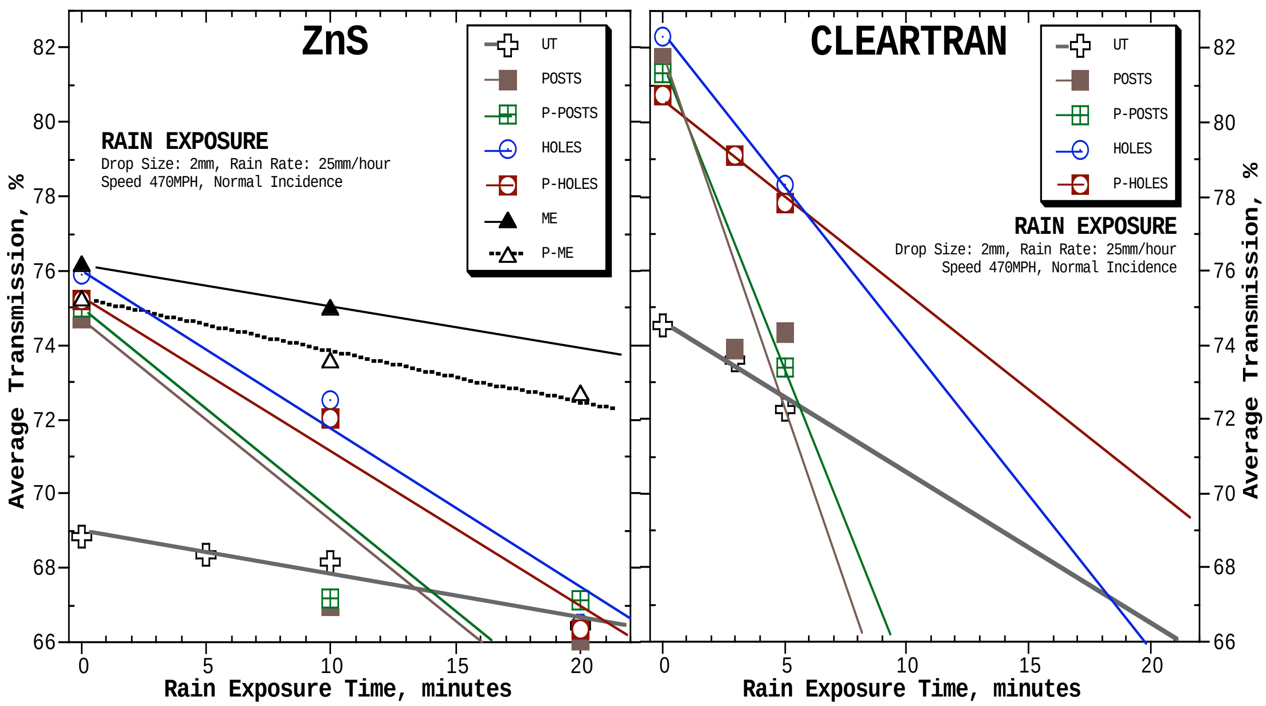



**Figure 20: Average LWIR transmission of AR micro-structured ZnS and ClearTran windows exposed to Rain.**

A compilation of the rain exposure results is shown in Figure 20 where as with the Sand exposure data, the average transmission of each window is calculated over the spectral range of 7.5 to 10 micron and plotted as a function of rain exposure duration. Separate plots for the ZnS windows (left) and ClearTran$^{TM}$ windows (right) are given side by side in the figure with equal axes limits. Data for untreated windows, shown as open crosses, is plotted together with data for each of the microstructure variants where open circles represent the hole array sample data, squares mark the post array microstructures, and triangles mark the Motheye microstructure data. Approximate linear fits to the limited set of data are drawn in as lines in each plot, with the solid black lines representing the untreated window data. The plots indicate that hole structures fared better than post structures likely due to the mutual support in the hole array texture. Data for these binary textures is scattered but seems to support the idea that surface damage to the microstructures causes rapid transmission loss early in the simulated window life, with a more gradual loss due to sub-surface damage occurring later. Again a significant difference is seen with the Motheye textured ZnS window data where only slight transmission loss is seen over the entire simulated life of the window.

## 5. SUMMARY

A preliminary investigation has been made of the durability of microstructure-based AR treatments in ZnS and ClearTran$^{TM}$ windows. Three types of AR microstructures were fabricated and exposed to rain and sand impacts using the facilities of the University of Dayton Research Institute (UDRI). Particle erosion testing showed that the rate of transmission loss with increasing sand load was the same for windows incorporating AR microstructures and untreated windows. AR microstructures in the harder ZnS windows were more effective than AR microstructures in the softer ClearTran$^{TM}$ windows as the dust load increased. This indicates that the performance benefits of a microstructured AR treatment could be combined with the long lifetime of hard materials such as sapphire, spinel, and ALON$^{TM}$.

The results of rain impact testing showed that Motheye AR microstructures have great potential for preventing sub-surface damage. High performance Motheye AR textures fabricated in ZnS windows showed less than 1% transmission loss after the maximum 20 minute duration in the UDRI rain rig operating at 470 MPH. No sub-surface damage could be found with the Motheye AR textured windows. Post type AR microtextures failed more rapidly in the rain exposure trials than hole-type AR textures, but neither of the binary textures was effective at preventing sub-surface damage.

This initial data indicates that AR microstructures can be an effective alternative to thin-film AR coatings allowing a window to maintain high transmission for longer time periods when operating in abrasive environments. Motheye AR textures may allow softer materials such as ClearTran$^{TM}$ and ZnS to meet the survivability requirements of a wider range of applications. Lastly, pyramidal microstructures similar to the Motheye textures, may provide enhanced rain erosion resistance for other applications such as missile and aircraft radomes.

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