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MISSE Scattered Atomic Oxygen Characterization Experiment

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Abstract

An experiment designed to measure the atomic oxygen (AO) erosion profile of scattered AO was exposed to Low Earth Orbital (LEO) AO for almost four years as part of the Materials International Space Station Experiment 1 and 2 (MISSE 1 and 2). The experiment was flown in MISSE Passive Experiment Carrier 2 (PEC 2), Tray 1, attached to the exterior of the International Space Station (ISS) Quest Airlock. The experiment consisted of an aperture disk lid of Kapton H (DuPont) polyimide coated on the space exposed surface with a thin AO durable silicon dioxide film. The aperture lid had a small hole in its center to allow AO to enter into a chamber and impact a base disk of aluminum. The AO that scattered from the aluminum base could react with the under side of the aperture lid which was coated sporadically with microscopic sodium chloride particles. Scattered AO erosion can occur to materials within a spacecraft that are protected from direct AO attack but because of apertures in the spacecraft the AO can attack the interior materials after scattering. The erosion of the underside of the Kapton lid was sufficient to be able to use profilometry to measure the height of the buttes that remained after washing off the salt particles. The erosion pattern indicated that peak flux of scattered AO occurred at an angle of approximately 45° from the incoming normal incidence on the aluminum base unlike the erosion pattern predicted for scattering based on Monte Carlo computational predictions for AO scattering from Kapton H polyimide. The effective erosion yield for the scattered AO was found to be a factor of 0.214 of that for direct impingement on Kapton H polyimide.

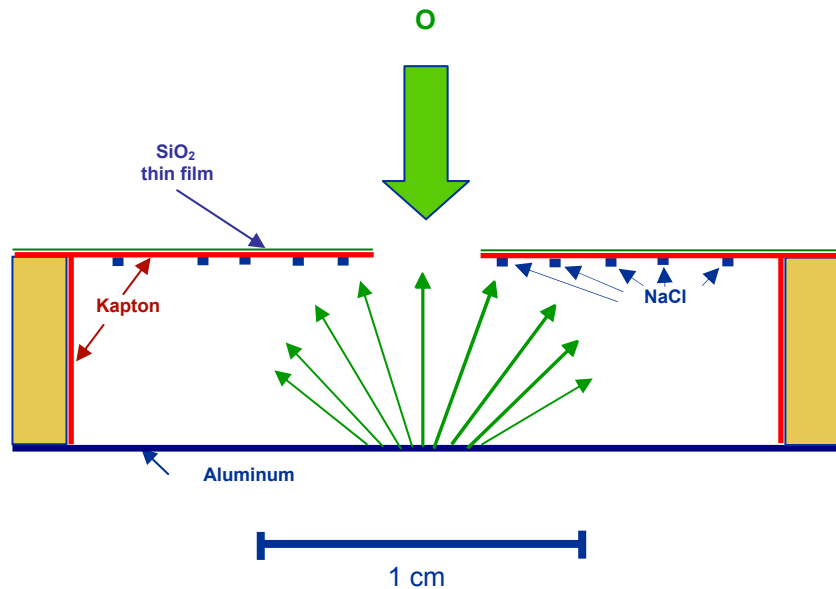
1. Introduction

Low Earth orbital (LEO) atomic oxygen (AO) has been well documented to be a durability concern for spacecraft polymers that are exposed to this environment (refs. 1 through 4). There have been relatively few investigations of the effects of scattered AO, with minimal actual in-space data. (ref. 5). Scattered AO is important for assessment of durability of polymers and metals that are not directly exposed to AO but may receive AO through secondary reflection processes. Materials that receive such scattered AO may be compromised if their function depends upon their structural, optical or surface conductivity properties. The objective of this effort was to measure the erosion characteristics of AO that is scattered from typical spacecraft surfaces. To make such measurements, a small AO scattering chamber was placed in a 2.54 cm diameter sample holder on the MISSE Passive Experiment Carrier 2 (PEC 2), Tray 1 (Sample Tray E5) on the exterior of the ISS Quest Airlock as part of the MISSE 1 and 2. The MISSE 2 Tray 1 was exposed primarily to direct ram AO exposure concurrent with solar exposure on-orbit. The sample was configured to scatter normally incident AO from an oxidized aluminum surface and allow it to react with Kapton H polyimide to measure the erosion as a function of ejection angle from the aluminum scattering surface which is representative of typical spacecraft materials.

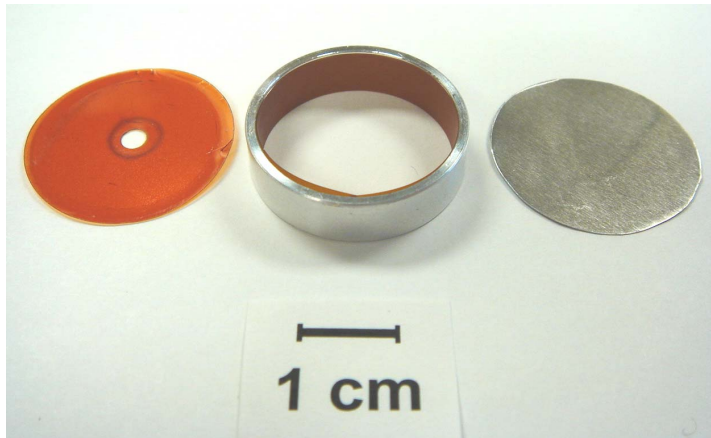
2. Flight Experiment and Experimental Procedures

2.1 Atomic Oxygen Scattering Chamber

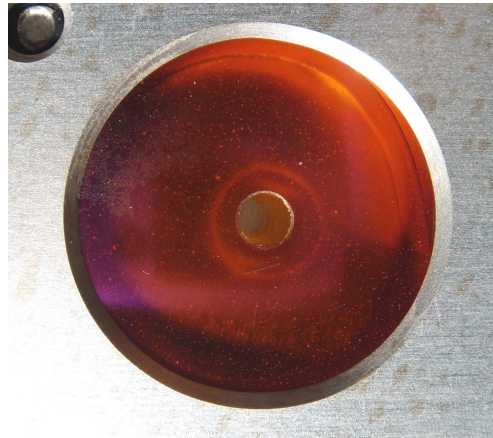
The experiment consisted of three components: a Kapton aperture lid, an aluminum cylindrical spacer and an aluminum base (see fig. 1). The aperture lid was a 2.54 cm diameter by 0.127 mm thick Kapton H polyimide disk, which was coated with ~1000 Angstroms of SiO₂ on the space exposed surface after ion beam precleaning its surface prior to coating. The SiO₂ was applied through ion beam sputter deposition. The aperture lid had a



(a) Section view drawing



(b) Photograph of component parts (left to right: Kapton aperture lid, Kapton-lined aluminum spacer and aluminum base)



(c) Photograph of scattering chamber in sample tray after retrieval

Figure 1.—AO scattering chamber.

3.05 mm diameter hole in the center of it to allow AO to enter and impinge at normal incidence upon the base of the chamber. Microscopic salt particles were deposited on the underside of the Kapton lid and were applied by repetitively sweeping the aperture disk through a fog solution of distilled water saturated with sodium chloride. The cylindrical aluminum spacer was 6.68 mm tall. The inside wall of the cylindrical spacer was lined with 0.127 mm thick Kapton H polyimide which was AO textured for the purpose of creating a rough surface texture that would enhance reaction with diffusely scattered AO to minimize additional scattering processes. The aluminum base disk was 0.125 mm thick aluminum. The aluminum base was not anodized but was exposed to AO in a plasma asher and hence had a relatively thick oxide on its surface.

2.2 Experimental Procedures

An Olympus SZH microscope operated with a Canon EOS D30 digital camera was used to document various features of the sample at magnifications on the order of 10 to 100X. Scanning electron microscope images were obtained using a Hitachi S-4700 field emission scanning electron microscope (FESEM) operated at an accelerating voltage of 6 kV. The AO erosion of the inside surface of the aperture lid was measured by first immersing the lid in distilled water to dissolve the salt particles, then using a Dektak 6M Surface Profiler to measure the height of pillars that were protected from AO erosion by the salt particles. The radial location of the measured pillars was determined by comparison with the optical microscope photographs of the pillars.

3. Results and Discussion

3.1 Leo Atomic Oxygen Exposure

The experiment was exposed to the LEO environment for 3.99 years from August 16, 2001 to July 30, 2005. During this exposure the orientation of the ISS was occasionally changed, however, the AO fluence impinging upon the face of the aperture disk was almost exclusively from a single direction which was measured to be approximately 8 degrees from normal incidence. The arrival angle was determined by measuring the inclination relative to the surface normal of undercut cavities at several small (~0.01 mm diameter) defect sites in the SiO₂ protective coating on the aperture disk. All the undercut cavities appeared as straight narrow cylinders which were parallel to each other indicating that the AO had arrived almost exclusively from a single direction. Because the aperture disk is recessed 0.762 mm below the surface of the sample tray (as were all adjoining samples) in the same tray, the AO arrival was limited to angles of up to 88 degrees from normal.

The AO fluence was measured using two Kapton H witness samples on the same PEC 2 sample tray (E5). The fluence was determined to be 8.43×10^{21} atoms/cm² based on a Kapton H polyimide density of 1.427 (as measured by a density gradient column) and an erosion yield for Kapton H to be 3.0×10^{-24} cm³/atoms (ref. 6).

3.2 Directed Atomic Oxygen Effects

There is a 2.37 mm wide ring around the outside of the aperture lid that was protected from the space environment. Microscopic inspection of the SiO₂ coating showed evidence of mud tile cracks across the exposed 2.065 cm central area of the disk, with no evidence of cracking in the protected region. The aperture lid was placed in distilled water for about 1 min to dissolve the salt particles. Immediately after the water was allowed to evaporate from the disk, it was observed that the SiO₂ coating had spalled in the exposed area of the disk as shown in figure 2(a). Figure 2(b) shows an electron micrograph of the spalled SiO₂ coating.

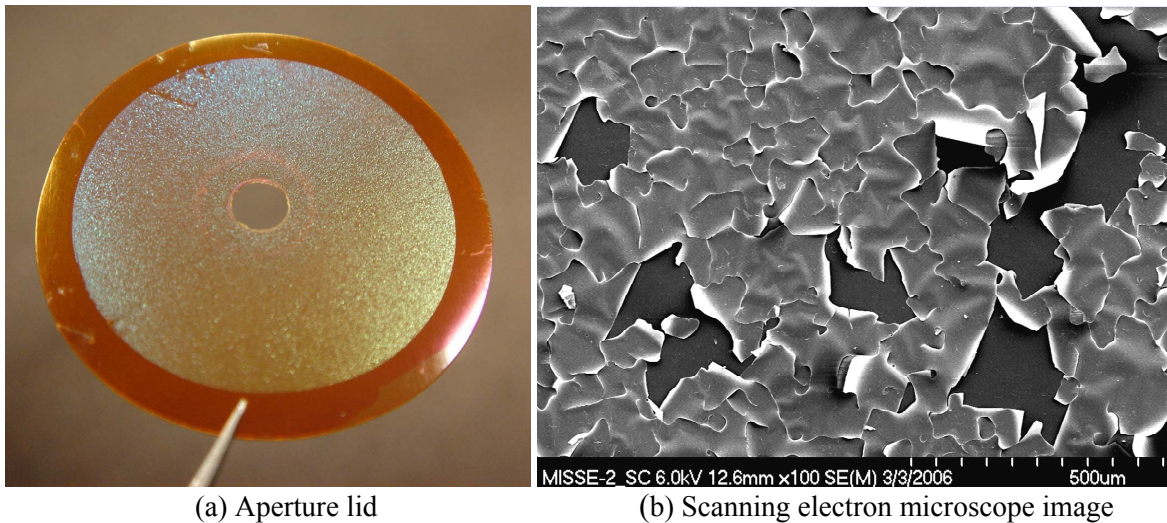


Figure 2.—Space exposed surface of aperture lid after salt removal in distilled water showing spalled SiO₂ coating in the space exposed region.

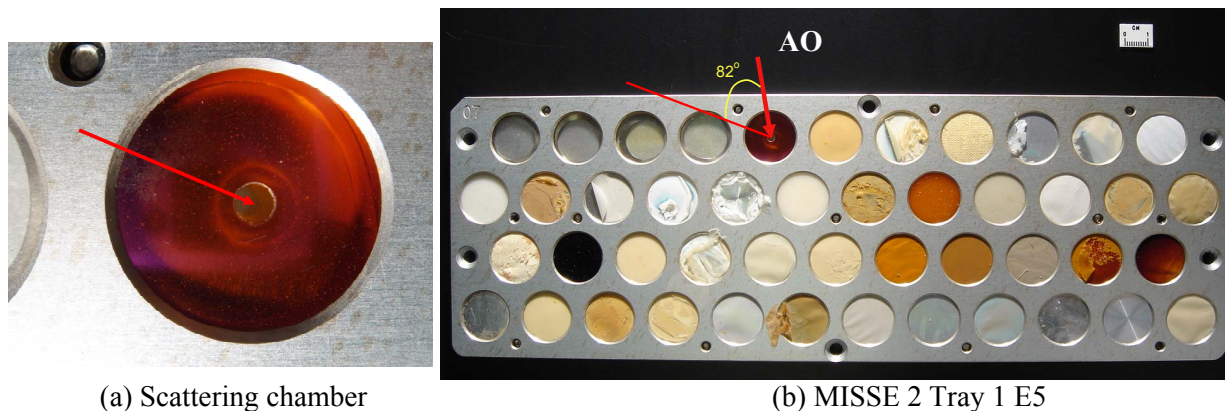


Figure 3.—Angle of arrival of AO on MISSE 2 Tray 1 E5 tray.

There were numerous small pin window apertures in the SiO₂ protective coating (defect sites) that allowed AO to etch all the way through the aperture lid. The holes etched into the aperture lid were well defined narrow tunnels indicating that the direction of AO attack on the scattering chamber was well aligned and unvarying. By microscopic alignment of several undercut tunnels below pin window defects, it was determined that the AO arrived was from an angle 8° from normal incidence and in a direction as indicated in figure 3. This arrival direction was also consistent with a slight amount of asymmetry of the dark scattered AO ring noticed on the inside surface of the aperture disk as seen in figures 3(a) and 4.

3.3 Scattered Atomic Oxygen Erosion

Figure 4 is a photograph that compares the inside surface of the flight sample aperture lid with a non-flow control aperture lid.

As can be seen from figure 4, the flight lid looks quite similar to the control lid with the exception of a dark ring surrounding the aperture of the flight lid. The salt particles are clearly visible in the photo. The actual exposed area of the aperture lid was the central 2.065 cm region of the disk.

Optical micrographs of the salt-sprayed inside surface of the flight aperture lid before and after salt particle removal are shown in figure 5.

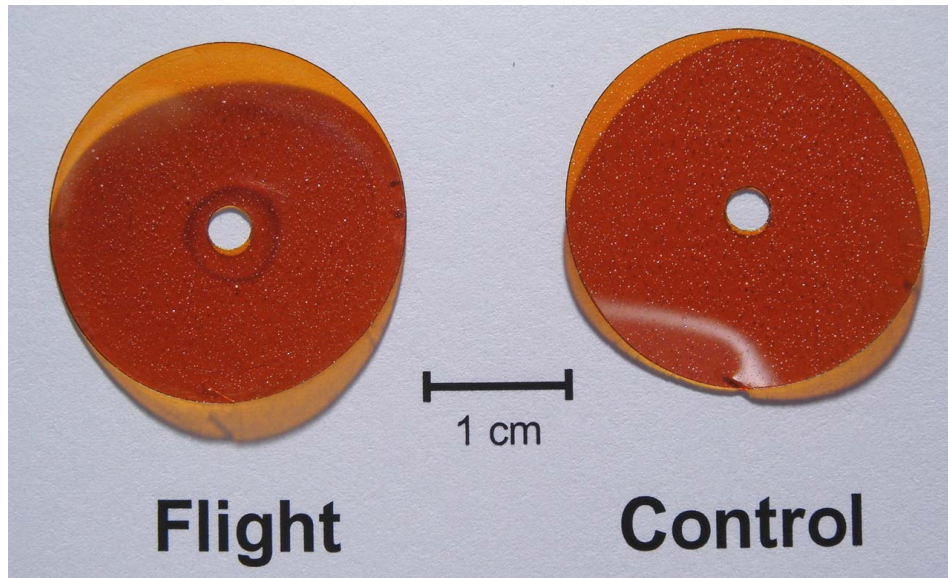
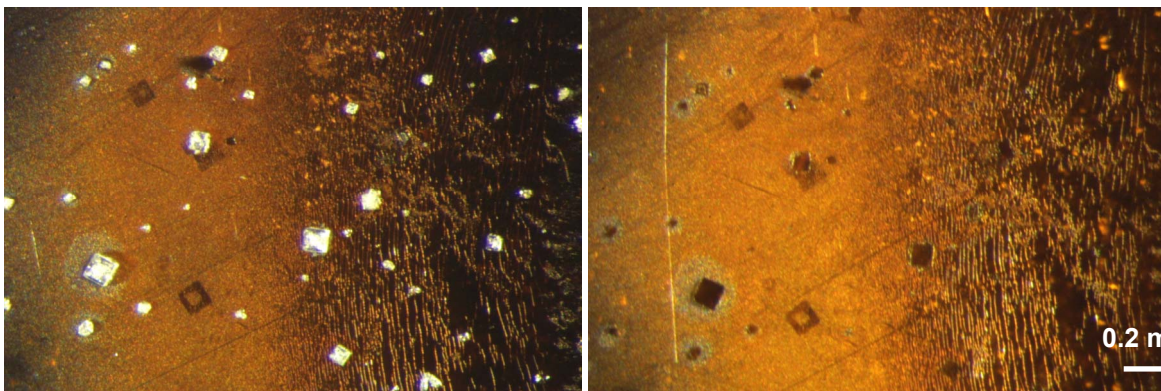


Figure 4.—Photograph of inside (salt dusted) surfaces of flight and pristine control aperture lids.

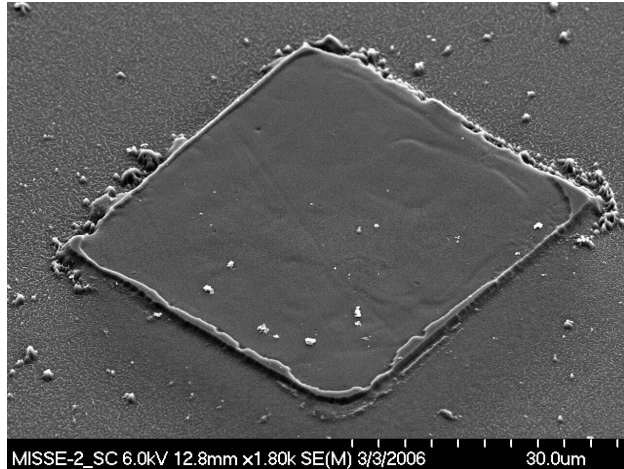


(a) Before salt removal

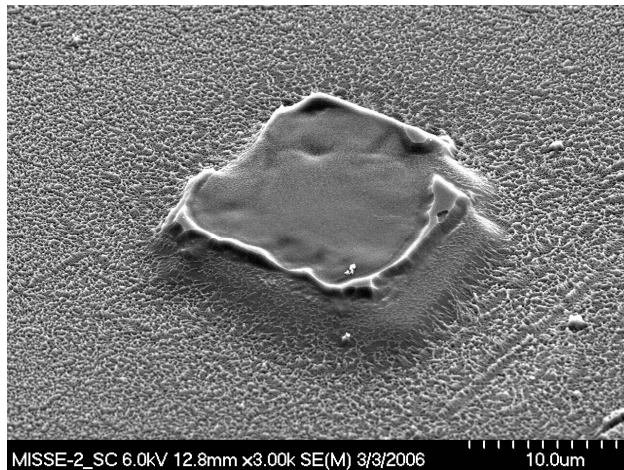
(b) After salt removal

Figure 5.—Inside surface of a portion of the flight sample aperture lid after AO exposure.

Scanning electron microscope images of two salt-protected pillars or buttes on the inside of the flight sample aperture lid are shown after salt-removal in figure 6. There appears to be a slightly elevated rim around most of the perimeter of the buttes. The reason for this is not understood at this time. Figure 6(a) is a butte located farther away from the aperture (outer circumference area), while the butte in figure 6(b) was located in the more textured and eroded “dark ring” area. The highly eroded areas show the expected texture in the Kapton H which results from directed high fluence AO attack.



(a) Low erosion area



(b) High erosion area

Figure 6.—Scanning electron microscope images of salt-protected buttes on inside surface of the flight aperture lid.

The inner plateau of the salt-protected buttes and the region external to this wall were used for profilometry comparison to measure the depth of AO erosion as a function of radius from the center of the aperture lid. Figure 7 shows a plot of the aperture lid AO erosion measurements as a function of radius from the center of the aperture. This figure also compares a photograph scaled to match. It is interesting that the dark ring observed around the aperture does not coincide with the radius of maximum erosion depth. What is immediately surprising from inspection of figure 7 is that the most eroded region was not located closest to the aperture disk as one would expect if the scattering behaved as a cosine distribution.

Integrating the erosion volume over the entire aperture disk predicts a scattered erosion volume of $4.04 \times 10^{-4} \text{ cm}^3$. This volume is 0.218 of the erosion volume which would occur if the aluminum target was Kapton H polyimide. Thus, some combination of a lower scattered AO flux and lower energy causes a reduction in the primary incoming AO effective erosion yield. The effective erosion yield for the scattered AO was found to be $6.42 \times 10^{-25} \text{ cm}^3/\text{atom}$ which is 0.218 of that for direct impingement on Kapton H polyimide. This reduction in effective erosion yield would probably be different for chemically different

base materials. To a first approximation, one might expect the variations in effective erosion yield for scattered AO to vary in a similar fashion to the differences between erosion yields in LEO and thermal energy RF plasma ashers because of a probable high degree of thermal accommodation for the scattered AO (which is discussed later in this paper).

A polar plot of this erosion distribution is shown in figure 8. This plot corrects the AO profilometry data for angle of incidence and the squared inverse distance flux relationship for the distance to the center of scattering site on the aluminum foil. The peak flux of the scattered AO occurs around 45° with very little flux scattering at less than 30° and at greater than 60° from the normal direction.

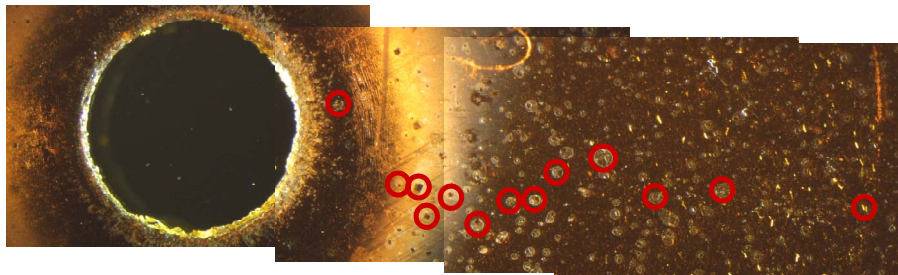
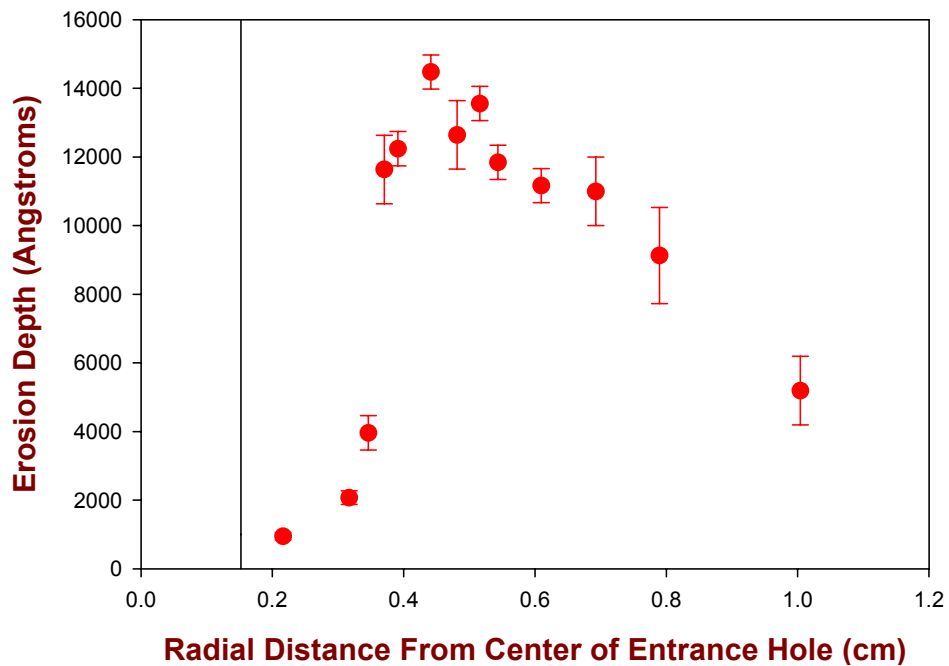


Figure 7.—Atomic oxygen erosion profile (depth vs. radius) of the inside surface of the flight aperture lid along with a photograph composite showing the X-axis of the plot and the specific locations of the pillars measured.

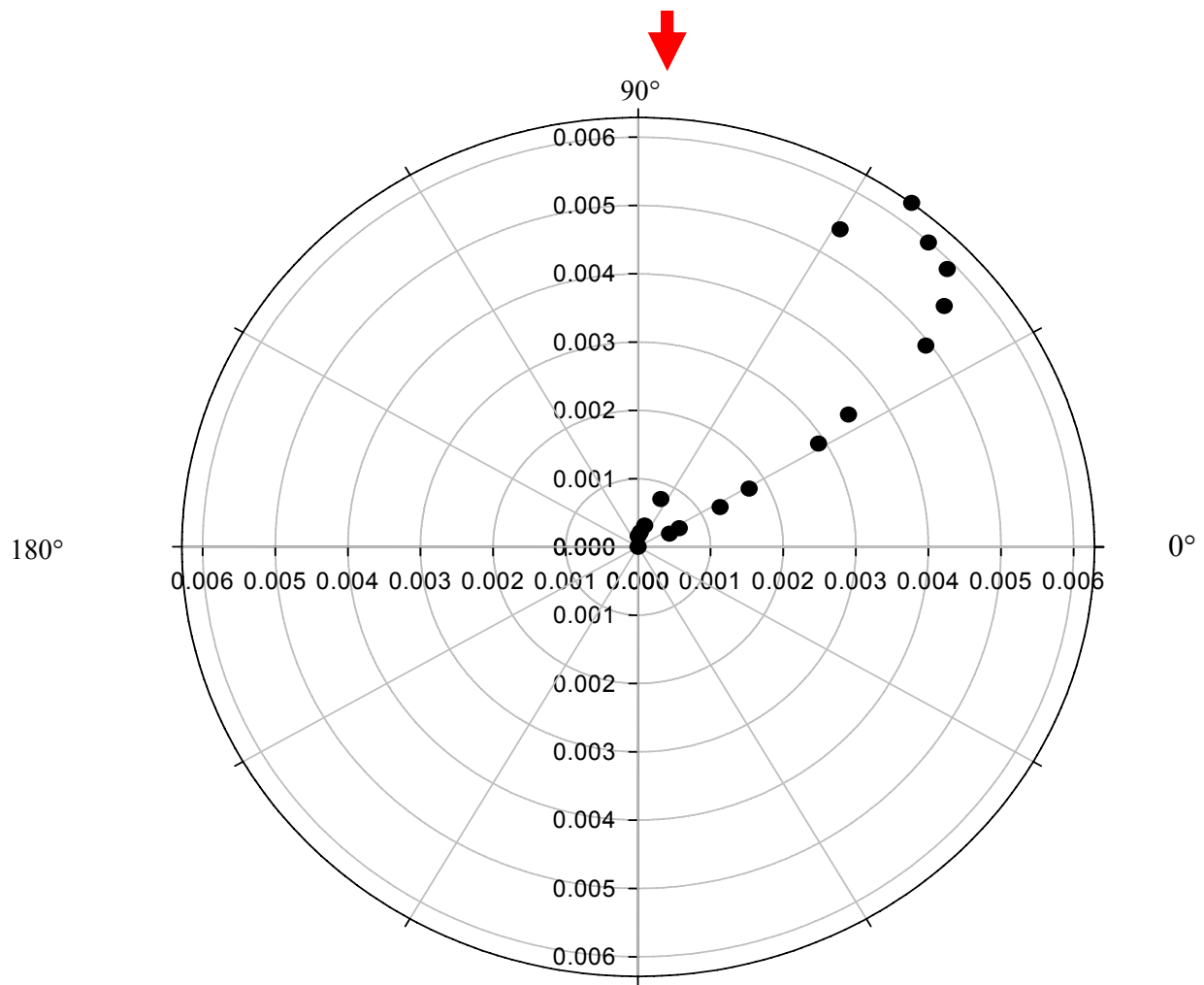


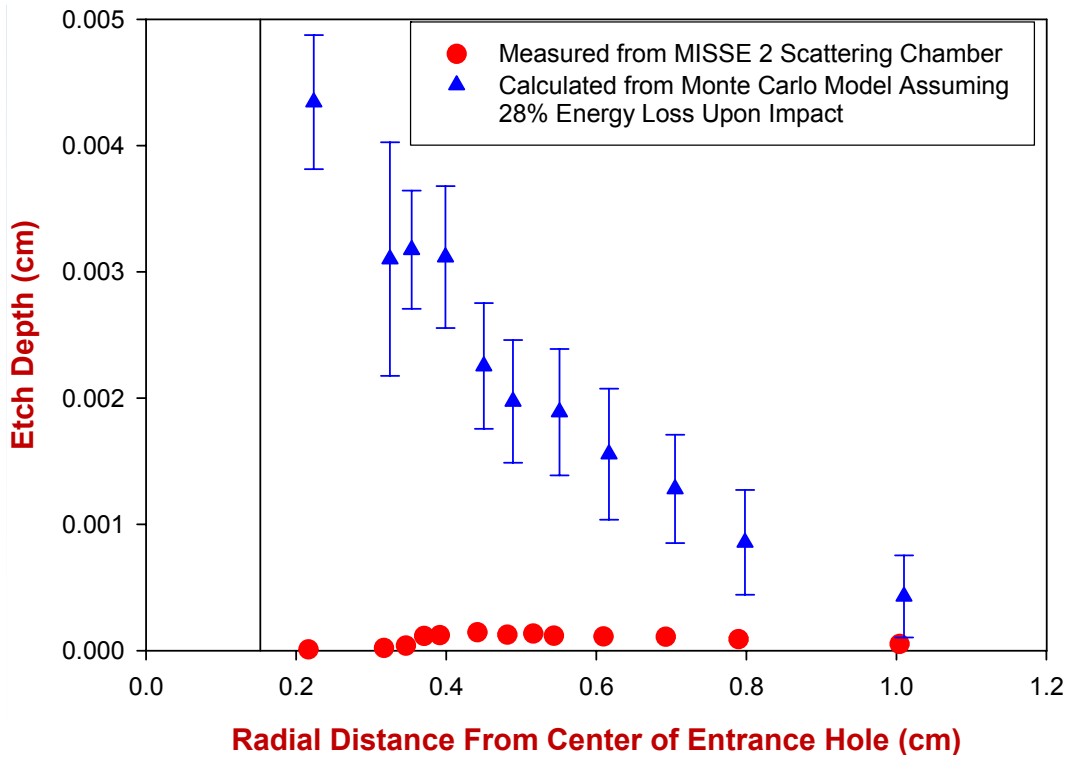
Figure 8.—Angular distribution of scattered atomic oxygen erosion rate relative to incident (arrow) atomic oxygen erosion rate.

3.4 Scattered Atomic Oxygen Erosion Modeling

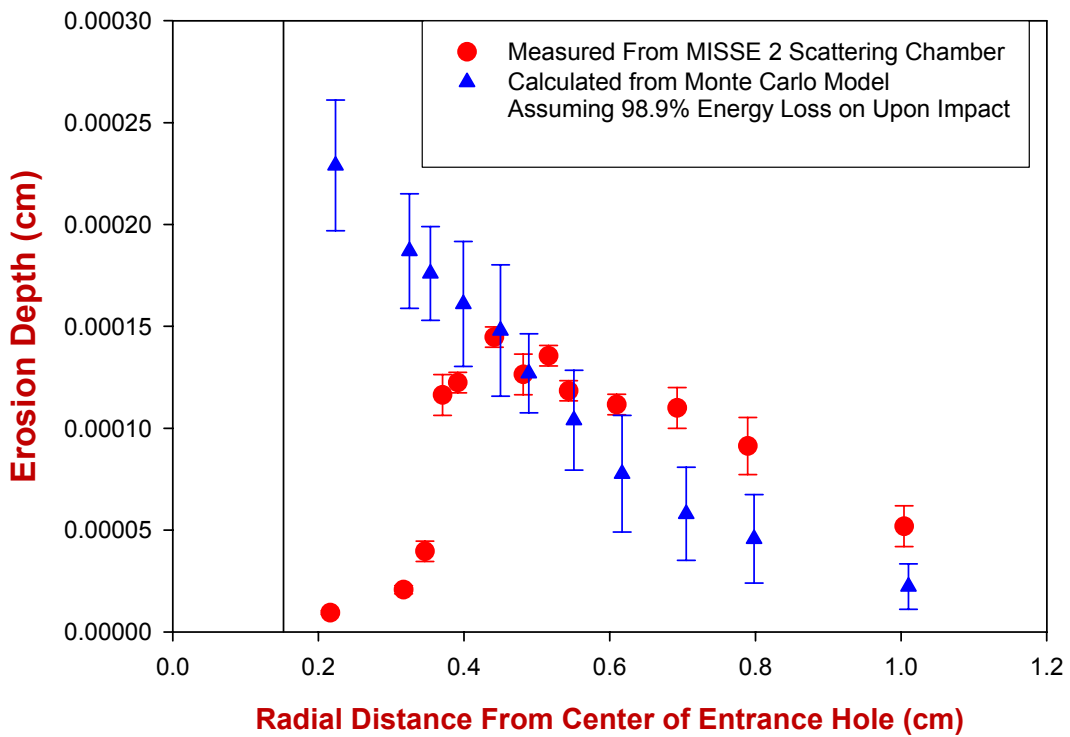
The angular scattering distribution off the oxidized aluminum base is a much different scattering distribution function than the cosine or Lambertian distribution that has been found to be optimal when using Monte Carlo computational modeling to predict erosion of AO scattering off Kapton H polyimide. Table 1 lists the computational modeling parameters which have been used in the past to replicate AO erosion of SiO₂ protected Kapton H (ref. 3). These parameters were used in the Monte Carlo model to predict scattered AO erosion that would occur on the inside of the flight aperture lid if the oxidized aluminum behaved as if it was SiO₂ with a cosine ejection distribution. The results of the erosion prediction are compared to the measured erosion from the flight sample in figure 9(a). The Monte Carlo computational modeling greatly over predicts the amount of AO erosion. By increasing the fractional energy loss of the AO upon impact in the Monte Carlo model from 28 to 98.9 percent (implying a high degree of thermal accommodation) there is closer erosion agreement as shown in figure 9(b). However, the angular distribution of the erosion is too intense at small scattering angles to properly replicate the erosion pattern. Thus, an angular ejection distribution which peaks at 45°, rather a cosine based distribution, may potentially improve the comparison between actual and Monte Carlo predicted erosion with Kapton H.

TABLE 1.—COMPUTATIONAL MODEL PARAMETERS AND REFERENCE VALUES FOR LEO ATOMIC OXYGEN INTERACTION

Parameter	Quantity
Atomic oxygen initial impact reaction probability	0.09
Activation energy, E _A , in eV for energy dependent reaction probability	0.26
Atomic oxygen probability angle of impact dependence exponent, n, in (cosθ) ⁿ angular dependence where θ is the angle between the arrival direction and the local surface normal	0.5
Probability of atomic oxygen recombination upon impact with protective coating	0.25
Probability of atomic oxygen recombination upon impact with polymer	0.35
Fractional energy loss upon impact with polymer	0.45
Fractional energy loss upon impact with protective coating	0.28
Degree of specularity as opposed to diffuse scattering of atomic oxygen upon non-reactive impact with protective coating where 1 = fully specular and 0 = fully diffuse scattering	0.45
Degree of specularity as opposed to diffuse scattering of atomic oxygen upon non-reactive impact with polymer where 1 = fully specular and 0 = fully diffuse scattering	0.035
Temperature for thermally accommodated atomic oxygen atoms, K	300
Limit of how many bounces the atomic oxygen atoms are allowed to make before an estimate of the probability of reaction is assigned	25
Thermally accommodated energy/actual atom energy for atoms assumed to be thermally accommodated	0.89
Initial atomic oxygen energy, eV	4.5
Thermospheric atomic oxygen temperature, K	1000
Atomic oxygen arrival plane relative to Earth for a Maxwell-Boltzmann atomic oxygen temperature distribution and an orbital inclination of 28.5°	Horizontal



(a) Assuming the oxidized aluminum surface acts like SiO₂ with a fractional energy loss of 28 percent upon impact.



(b) Assuming the aluminum oxidized surface causes a fractional energy loss of 98.9 percent upon impact (high degree of thermal accommodation)

Figure 9.—Measured and Monte Carlo predicted erosion profiles.

4. Summary

An experiment was conducted as part of MISSE 1 and 2 to measure the erosion depth profile and Kapton H effective erosion yield of AO scattering off an oxidized aluminum surface in LEO. The experiment was flown in the MISSE PEC 2, Tray 1, and was exposed to LEO AO for a period of almost four years on the exterior of the ISS Quest Airlock. The experiment consisted of an aperture lid of Kapton H polyimide coated on the space exposed surface with a thin AO durable film of silicon dioxide. The aperture disk had a small hole in its center to allow AO to enter into a chamber and impact a base disk of oxidized aluminum. The AO that scattered from the aluminum base could react with the under side of the aperture lid, which was coated sporadically with microscopic sodium chloride particles. The experiment was exposed to an AO fluence of 8.43×10^{21} atoms/cm² at 8° from normal incidence. The erosion pattern indicated that the peak flux of scattered AO leaving the oxidized aluminum base occurred at an angle of approximately 45° from the incoming normal incidence. The flight sample 45° peak flux dependence was found to be inconsistent with Monte Carlo model predictions for AO scattering off Kapton H (as a base), which indicate a cosine dependence of the scattering flux. The effective erosion yield of the Kapton H for the AO scattered off of oxidized aluminum was found to be 6.54×10^{-25} cm³/atom, which is 0.218 of the erosion yield for direct AO impingement on Kapton H polyimide in LEO.

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