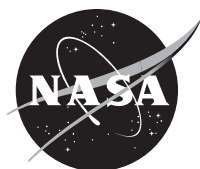


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# Effects of Sample Holder Edge Geometry on Atomic Oxygen Erosion Yield of Pyrolytic Graphite Exposed in Low Earth Orbit

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## Abstract

The measurement of the erosion yield (volume lost per incident atom) of polymers and carbon using low Earth orbit (LEO) atomic oxygen exposure has typically involved placing small samples in holders that have chamfered edges. The chamfered edges have sometimes caused the samples to tear by their perimeters. This paper reports an evaluation of the erosion profile of a LEO atomic oxygen exposed pyrolytic graphite sample to determine the extent to which the erosion yield is altered by the chamfer and what the expected erosion yield would be for large area samples. The results indicate that the durability of large area samples would be ~5.2 percent lower than predicted based on mass loss of small samples with exposed diameters of 0.838 in. (2.13 cm) and chamfered sample holders.

## Nomenclature

$F_O$	Atomic oxygen ram fluence that directly hits the sample at normal incidence, atoms/cm <sup>2</sup>
$F_D$	Atomic oxygen fluence that is diffusely scattered from the chamfered surface on to the sample surface, atoms/cm <sup>2</sup>
$F_S$	Atomic oxygen fluence that is specularly scattered from the chamfered surface on to the sample surface, atoms/cm <sup>2</sup>
$F_T$	Total atomic oxygen fluence arriving on the sample surface, atoms/cm <sup>2</sup>
$\phi$	Angle the diffusely ejected atomic oxygen fluence makes with the sample surface upon impact, radians
$D$	Diameter of the exposed area of the sample, cm
$E_y$	Atomic oxygen erosion yield, cm <sup>3</sup> /atom
$E_{yM}$	Atomic oxygen erosion yield based on mass loss for small samples, cm <sup>3</sup> /atom
$E_{yL}$	Atomic oxygen erosion yield based on profilometry for large samples, cm <sup>3</sup> /atom
$E_{yP}$	Atomic oxygen erosion yield based on profilometry for small samples, cm <sup>3</sup> /atom
$K$	Ratio of the erosion yield based on mass loss to erosion yield based on profilometry
$R$	Radius from the centerline of the sample to the perimeter of the exposed area, cm
$R_U$	Radius from the centerline of the sample to the maximum radius of the eroded area of the sample, cm
$r$	Radius from the centerline of the sample to an arbitrary radial location, cm
$r_i$	Radius from the center of the sample to the $i^{\text{th}}$ location, cm
$r_c$	Radius of the central flat eroded area of the sample, cm
$\Delta r$	Increment radius used for computation of erosion volume, cm
$z$	Erosion depth, cm
$z_i$	Erosion depth at location $r_i$ from the center of a circular sample, cm

## Introduction

The atomic oxygen durability of polymers has typically been estimated using dehydrated mass-loss measurements of circular or square samples that have been exposed to low Earth orbit ram atomic oxygen during experiments such as the Materials International Space Station Experiments 1-8 (MISSE 1-8) (Refs. 1 to 4). The most important measure of atomic oxygen durability of a material is the atomic oxygen erosion yield,  $E_y$ , which is the volume lost (through oxidative erosion) per oxygen atom that arrives at the surface. The  $E_y$  is calculated using the measured dehydrated mass loss ( $\Delta M$ ) due to atomic oxygen erosion of the sample of interest divided by its density ( $\rho$ ), the exposed area ( $A$ ) of the sample, and the atomic oxygen fluence ( $F$ ) and is given by

$$E_y = \frac{\Delta M}{\rho A F}$$

For such experiments the  $E_y$  typically assumes that the atomic oxygen (AO) fluence is uniform across the samples. However the MISSE-2 ram atomic oxygen fluence was sufficiently high and nonuniform enough to cause some polymer samples to erode completely or partially through at their perimeters as shown in Figure 1.

The unsymmetrical erosion of the samples on the sample tray, which was similar in orientation to that shown for cellulose acetate and ally diglycol carbonate in Figure 1, appears to be a result of enhanced AO erosion at specific locations around the perimeter of the samples. Furthermore, analysis of the angle of atomic oxygen arrival through pin windows in the SiO<sub>2</sub> coated Kapton covering the atomic oxygen scattering chamber indicated that the AO was arriving at 8° off normal (Refs. 3 and 4). Thus, it appears

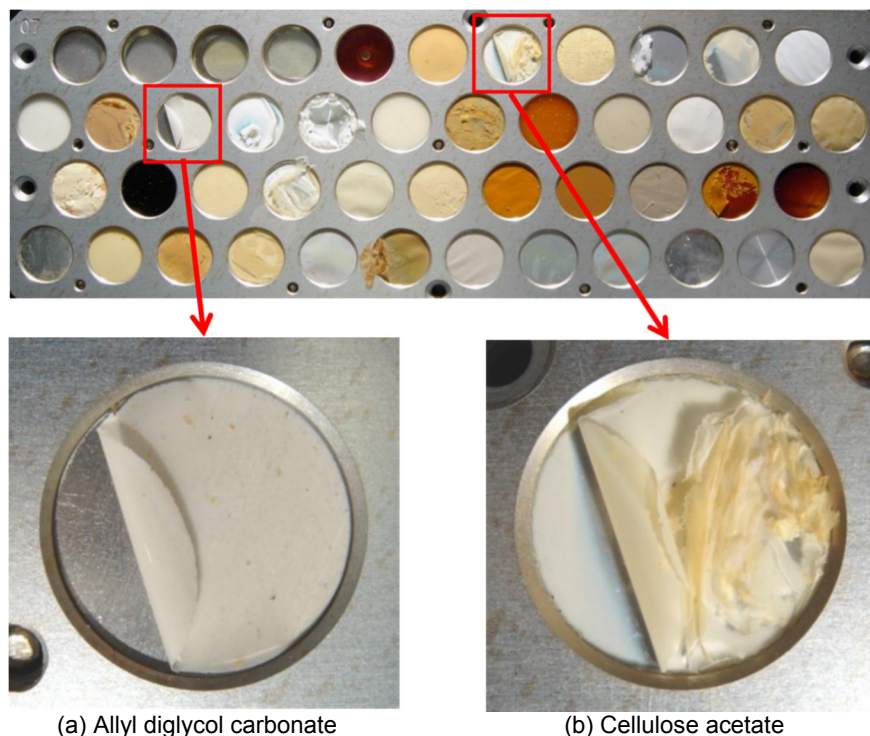


Figure 1.—The MISSE-2 sample tray after being exposed to 4.5 years in low Earth orbit to an AO fluence of  $8.43 \times 10^{21}$  atoms/cm<sup>2</sup> (Ref. 3). (a) and (b) are enlarged views showing erosion through the samples at the edge of the holder causing them to tear and curl.

that there was an additional AO fluence arriving on the perimeter of the samples caused by off-normal incidence AO scattering off the 45° chamfered sides of the 0.030 in. (0.0762 cm) thick anodized aluminum sample holders. This fluence varied around the perimeter of the samples which caused varying erosion in excess of the central areas of the samples. Although the magnitude of the off-normal incident direction was measureable there was no clear indication of the arrival direction due to the fact that the orientation of the SiO<sub>2</sub> coated Kapton circular samples was not known. One can only suspect that the enhanced erosion which resulted in the partial tearing of the two samples was due to a higher concentration of AO exposure arriving on that side due to the off normal arrival incidence. Although some AO scattering effects have been quantified by the scattering chamber test on MISSE-2 (Ref. 5) and from Monte Carlo computational modeling (Ref. 6), no quantitative information is known to be available to determine the effects of AO scattering from the 45° chamfered sample holders.

Because the  $E_y$  measurements assume a uniform AO fluence across the exposed face of the samples, the actual AO fluence is probably different than that which has been used for  $E_y$  calculations. This is a result of the increased AO arrival at the near the edges of the samples due to scattering off the surfaces of the chamfered sample holders and a decreased fluence in the immediate vicinity of the edges. As a consequence, the actual  $E_y$  for large area samples would most likely be different than estimated from small area samples due to the scattering near and attenuation at the chamfered perimeters of the sample holders.

The objective of this investigation is to examine potential effects of scattered AO from the chamfered sides of sample holders on the erosion yield through modeling and measurements of a pyrolytic graphite sample exposed as part of the MISSE-2 experiment. This investigation was conducted for the purpose of determining the magnitude of incorrect estimation of the  $E_y$  due to the modified contributions from the perimeter for small diameter holders and samples. Pyrolytic graphite was chosen for this analysis because it remains flat under the thermal, AO, and UV environments of LEO and thin polymers tend to distort. However, the results are probably relevant to polymers.

## Background and Approach Considerations

Scattering chamber tests performed on MISSE 2 indicated that ejection of AO from normally incident atoms is predominantly at 45° from normal (Ref. 4). If this is true, then for oxygen atoms arriving normal to the sample surface would impact the chamfer at 45°. They would then be scattered parallel to the sample surface and not have any impact on the measured  $E_y$ . However, if there is off normal arrival of AO, specularly scattered AO near the edge of the chamfer would contribute to enhanced local erosion, at least on one side of the sample. Diffusely scattered AO would have a portion of its flux contributing to the erosion of the samples over a broader area. Monte Carlo modeling of AO scattering from nonreactive surfaces of SiO<sub>2</sub> has shown that 33 percent of the atoms recombine upon impact while the rest are scattered. Of the AO that is scattered, half are ejected in a cosine distribution and the other half in a specular manner (which again would not impact the sample surface) (Ref. 5). Figure 2 shows the geometry of the sample and chamfered holder that was used to understand the AO erosion.

Assuming that there is only direct fluence and scattered (diffuse and/or specular) fluence impinging upon the sample, then the total arriving fluence would be  $F_T = F_O + F_D + F_S$ . Not knowing the fraction of fluence that is being scattered in a specular or diffuse manner, it is difficult to estimate the degree to which the measured  $E_y$  is artificially enhanced by the effects of the chamfer. In addition, there would be a loss in  $E_y$  caused by off-normal AO impact with the sample that is proportional to  $\cos(\pi/2 - \phi)^{0.5}$  from impact at an angle,  $(\pi/2 - \phi)$  onto the sample based on results from the Long Duration Exposure Facility (LDEF) (Ref. 6). Lacking sufficient information to estimate the difference in the erosion yields in this manner, stylus profilometry across the diameter of a circular flight sample can be used to determine the volume,  $V$ , of oxidative erosion of the sample. Figure 3 shows two potential erosion profiles, one which is dominated by specular scattering of AO and the other dominated by diffuse scattering of AO. Because of the magnification in the vertical direction, the chamfers appear steeper than the actual 45°.

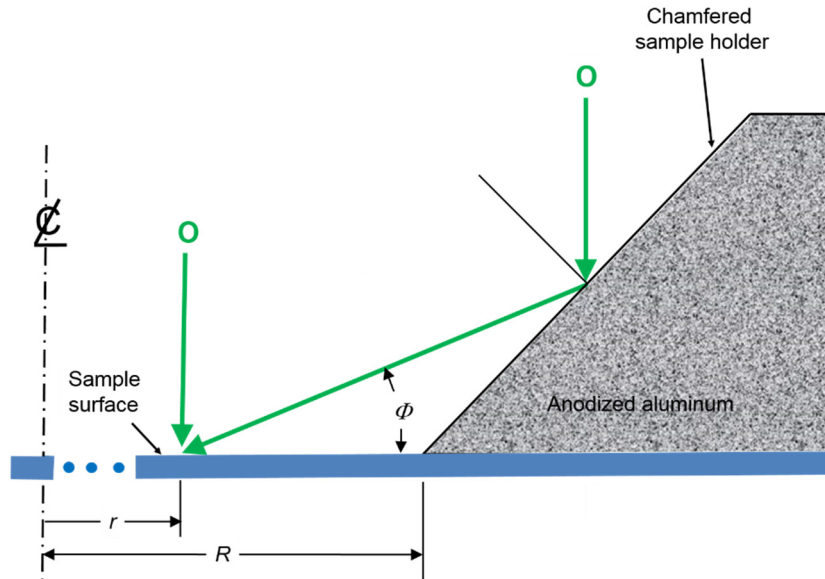


Figure 2.—Geometry of the sample and chamfered sample holder used to understand AO erosion near the perimeter of the samples.

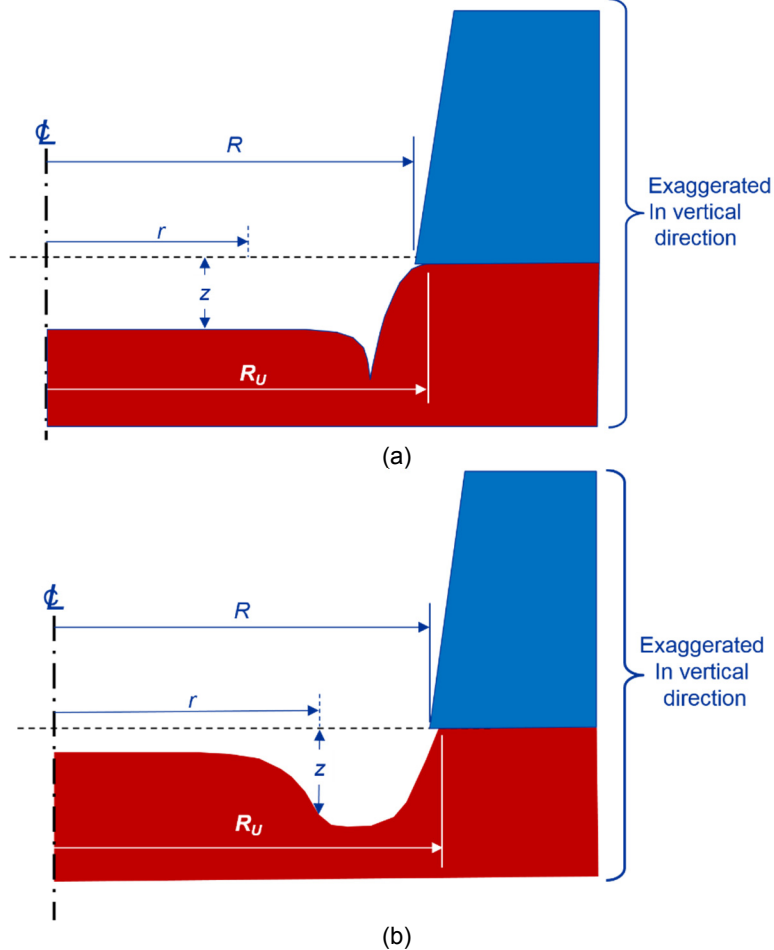


Figure 3.—Potential erosion crosssections of a circular sample showing additional AO erosion near its perimeter. (a) Dominated by specularly scattered AO. (b) Dominated by diffusely scattered AO.



The erosion yield based on profilometry measurements,  $E_{yP}$ , is given by

$$E_{yP} = \frac{V}{\pi R_U^2 F_T}$$

Where the volume,  $V$ , from stylus profilometry would be given by

$$V = 2\pi \int_0^{R_U} zr dr$$

By taking incremental measurements of  $z$  as a function of  $r$  in  $30^\circ$  azimuthal scans across the face of the sample, an average depth,  $z_{ave}$ , at each radius,  $r_i$ , can be used to calculate the volume of erosion by

$$V = \sum_{i=0}^{R_U} 2\pi r_i z_{ave} \Delta r$$

Thus, 
$$E_{yP} = \frac{\sum_{i=0}^{R_U} 2\pi r_i z_{ave} \Delta r}{R^2 F_T}$$

However, stylus profilometry cannot fully account for the mass loss of material eroded between the cone structures on the textured surface of the sample because the stylus tends to ride on or near the top of the cones on the textured surface. Figure 4 shows scanning electron microscope images of the pyrolytic graphite samples exposed to various space ram AO fluences.

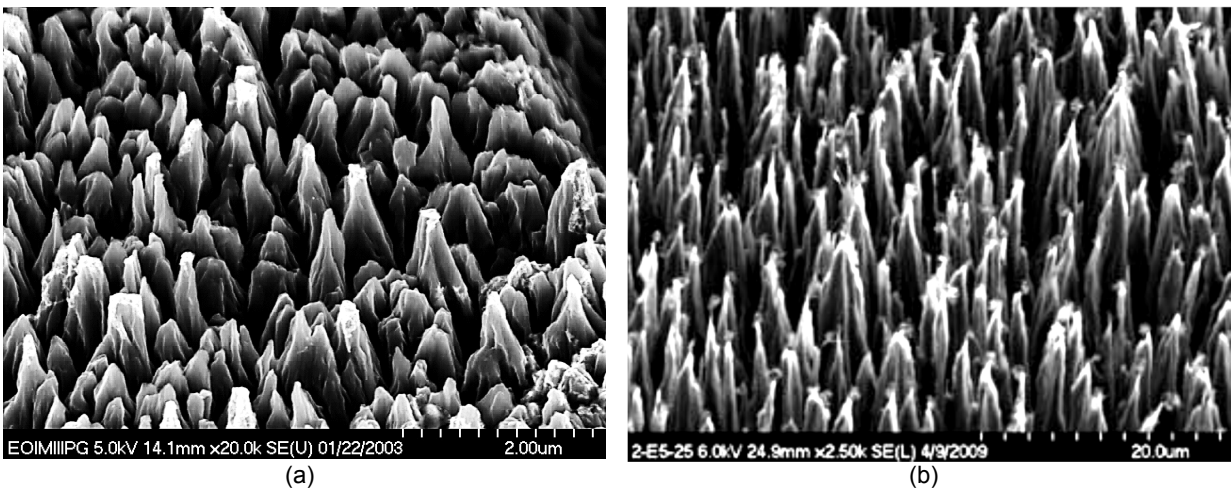


Figure 4.—Scanning electron microscope images of pyrolytic graphite samples exposed to various ram AO fluences  
 (a) Fluence of  $2.3 \times 10^{20}$  atoms/cm<sup>2</sup> on environmental oxygen interaction with materials shuttle experiment.  
 (b) Fluence of  $8.43 \pm 0.10 \times 10^{21}$  atoms/cm<sup>2</sup> on MISSE 2 (Ref. 4).

Thus the  $z_i$  values need to be proportionally increased so that the  $E_y$  values match with mass loss derived values. Such that

$$K = \frac{E_{yM}}{E_{yP}}$$

where  $E_{yM}$  is the erosion yield based on mass loss and  $E_{yP}$  is the erosion yield based on profilometry. Thus

$$E_y = \frac{K \sum_{i=0}^{R_U} 2r_i z_{ave} \Delta r}{R^2 F_T}$$

By using the corrected  $z_i$  values in the central flat eroded plateau of radius,  $r_c$ , one can calculate the erosion yield,  $E_{yL}$ , of a sample if there was no additional scattered fluence from the chamfered sample holder where

$$E_{yL} = \frac{K \sum_{i=0}^{r_c} 2r_i z_{ave} \Delta r}{r_c^2 F_T}$$

The ratio of these two calculations for erosion yield,  $E_{yP}/E_{yL}$  can then be used to determine the fractional overestimation of  $E_y$  due to AO scattering from small samples in chamfered holders. For the circular sample holders used to generate most of the  $E_y$  measurements from many of the MISSE missions the exposure diameter,  $2R$ , was 0.838 in. (2.13 cm) in diameter with the thickness of the 45° anodized-aluminum chamfer being 0.30 in. (0.762 cm) thick.

For this paper, pyrolytic graphite was used for profilometry because of its shape stability in the low Earth orbit environment and the fact that many of the thin polymer materials did not maintain a flat surface resulting in a curved shape which compromised the profilometry results. The exposed diameter of the particular sample holder used for the pyrolytic graphite sample analyzed in this paper was found to be  $2.13 \pm 0.0050$  cm, based on 10 measurements (Ref. 3). The ram AO fluence for MISSE 2 was measured to be  $8.43 \pm 0.10 \times 10^{21}$  atoms/cm<sup>2</sup>.

A Bruker DektakXT® stylus profiler with a stylus tip 12.5 μm in diameter with a 3 mg mass applied was used to measure the depth of atomic oxygen erosion every 30° across the face of a 1 in. (2.54 cm) diameter pyrolytic graphite sample. The erosion profile measurements were made every 30° around the sample with the intention of averaging out the effects of the ~8° misalignment of the MISSE sample plate. As a result of the significant roughness of the surface, it was not clear from the six profiles, taken every 30°, as to the direction of the ~8° misalignment.

## Results and Discussion

The results of the average of the six azimuthally different depth profiles representing 13,548 measurements each across the face of the pyrolytic graphite sample are shown in Figure 5.

Because the MISSE 2 samples were inclined to ~8° from normal incidence, the maximum depth of erosion found near the outer perimeter of the sample varied slightly around the perimeter of the sample resulting in slighter greater erosion in a narrow region near the right side of the erosion profile as shown in Figure 5. This may explain the reason some of the MISSE samples were almost completely eroded on one edge which allowed them to tear as observed for the allyl diglycol carbonate and cellulose acetate samples shown in Figure 1. Even multilayer Kapton H samples in square chamfered sample holders, both with and without spacing between the Kapton H layers, flown on MISSE 6 and exposed for 1.45 years in LEO, to a ram atomic oxygen fluence of  $1.90 \pm 0.05 \times 10^{21}$  atoms/cm<sup>2</sup>, show indications of enhanced erosion adjacent to the chamfered edges as shown in Figure 6 (Ref. 6).

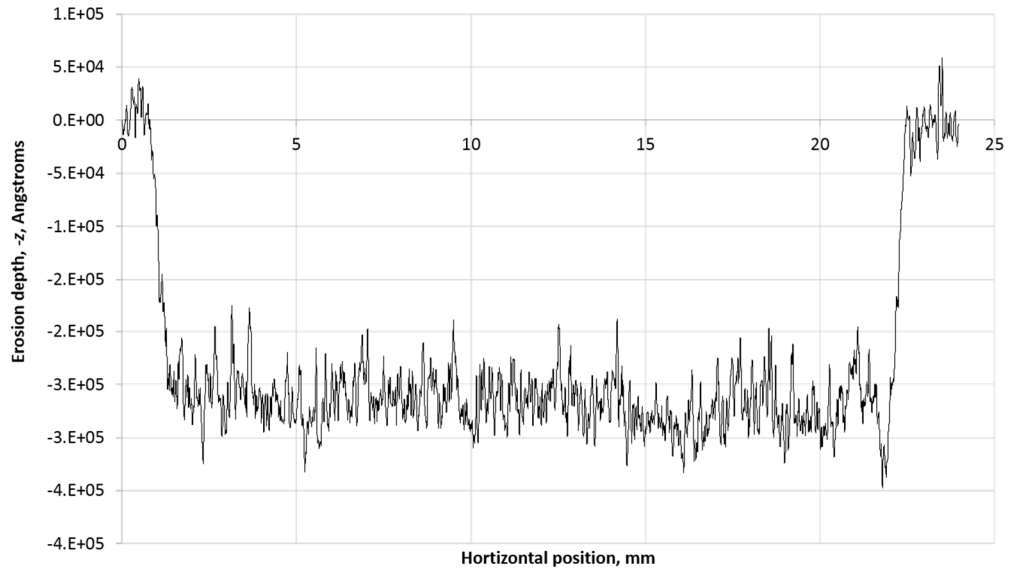


Figure 5.—Average of depth profiles across the pyrolytic graphite sample from MISSE 2 after exposure to a ram atomic oxygen fluence of  $8.43 \pm 0.10 \times 10^{21}$  atoms/cm<sup>2</sup>.

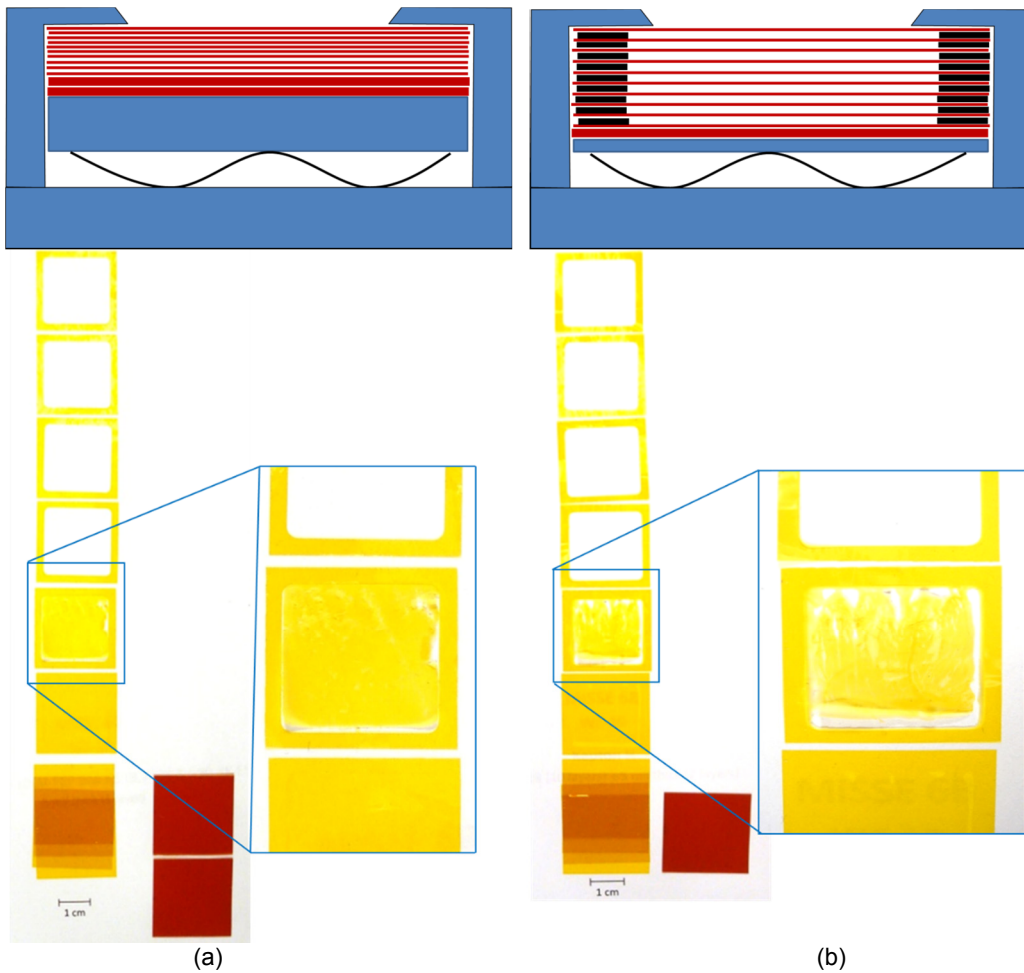


Figure 6.—Schematics and post-flight photos of thin stacked Kapton H samples, showing individual sample layers: (a) Kapton H (10 - 0.5 mil layers plus two 5 mil layers at bottom) with no spacers and (b) Kapton H (10 - 0.5 mil layers plus one 5 mil layer at bottom with 0.014 in. thick Al spacers in between each layer).

Based on the profilometry data, the erosion yield was found to be

$$E_{yP} = 3.03 \times 10^{-25} \text{ cm}^3/\text{atom}$$

However the erosion yield measured by mass loss (Ref. 3) was found to be

$$E_{yM} = 4.15 \times 10^{-25} \text{ cm}^3/\text{atom}$$

Thus, the correction factor to take into account the erosion in the valleys below the tops of the cones is

$$K = \frac{E_{yM}}{E_{yP}} = 1.37$$

The radius of the central erosion plateau,  $r_c$ , was found to be approximately 0.700 cm thus resulting in the erosion yield for large area samples to be

$$E_{yL} = 4.37 \times 10^{-25} \text{ cm}^3/\text{atom}$$

The surprising result is that the erosion yield for large area samples is projected to be 5.2 percent greater than that for the small MISSE 2 sample size. This result is thought to be a consequence of the rather narrow trench near the edge of the chamfer, probably caused by predominantly specularly-scattered AO, which enables a cut thru but does not compensate for the lack of oxidation of the sample material that is closer to the chamfer edge. Without knowing the impact energy of the scattered atoms and the erosion yield at that energy it is difficult to compute the actual fluence of scattered atoms. This slight increase in etching causing the trench is evident in the Figure 6 photographs where the enhanced perimeter erosion is visible only after four layers of Kapton H are fully eroded. However, the maximum depth of the erosion trench for the pyrolytic graphite sample was only 30 percent deeper than the average depth of erosion across the central plateau of the sample which is consistent with the tendency for samples that are almost fully eroded to tear near their perimeter. Enhanced AO fluence arriving near the perimeter of samples would be the same for all materials with inclined incidence that were held down by anodized aluminum 45° chamfers that are 0.030 in. (0.762 cm) thick with an exposed diameter of 0.838 in. (2.13 cm). However, the erosion enhancement from off normal incidence of the scattered AO fluence and differences in cone height and erosion depth may be material chemistry dependent. If most polymers behave similar to pyrolytic graphite then the durability of large area samples (based on average erosion depth) should be ~5.2 percent lower than predicted based on mass loss erosion of small area materials held by 1.0 in. diameter chamfered sample holders. In addition, enhanced perimeter erosion of the large area samples would still occur.

## Summary

Several LEO flight experiments have shown tearing of polymer samples close to their perimeters after they were significantly atomic oxygen eroded. Scattering of atomic oxygen off the chamfers appears to be dominated by specularly scattered AO causing enhanced atomic oxygen flux at the perimeters of the samples. A pyrolytic graphite sample was used to measure the erosion across the face of the sample in six different azimuthal directions. Averaging the profile results indicate that there is enhanced AO erosion caused by the chamfer near the sample perimeters. In spite of the enhanced erosion near the edges, the lower erosion immediately adjacent to the edge of the chamfer causes less measured erosion for small samples while for large samples, the central erosion area dominates over the edge effects. The results indicate that central area AO erosion is 5.2 percent greater than the average erosion for samples 2.13 cm in exposed diameter. The consequences indicate that the durability of large area samples should be ~5.2 percent lower than predicted based on mass loss of small samples with exposed diameters of 0.838 in. (2.13 cm) in chamfered sample holders.

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