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Comparison of the Results of MISSE 6 Atomic Oxygen Erosion Yields of Layered Kapton H Films With Monte Carlo Computational Predictions

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Abstract

A space experiment flown as part of the Materials International Space Station Experiment 6B (MISSE 6B) was designed to compare the atomic oxygen erosion yield (Ey) of layers of Kapton H polyimide with no spacers between layers with that of layers of Kapton H with spacers between layers. The results were compared to a solid Kapton H (DuPont, Wilmington, DE) sample. Monte Carlo computational modeling was performed to optimize atomic oxygen interaction parameter values to match the results of both the MISSE 6B multilayer experiment and the undercut erosion profile from a crack defect in an aluminized Kapton H sample flown on the Long Duration Exposure Facility (LDEF). The Monte Carlo modeling produced credible agreement with space results of increased Ey for all samples with spacers as well as predicting the space-observed enhancement in erosion near the edges of samples due to scattering from the beveled edges of the sample holders.

Introduction

MISSE 6 Stacked Layer Experiment

The Materials International Space Station Experiment (MISSE) series consisted of flight experiment trays that were mounted to the exterior of the International Space Station (ISS) (Ref. 1). As part of the Stressed Polymers Experiment, a set of multilayer samples of Kapton H polyimide were located on the ram side of MISSE 6 Passive Experiment Carrier B (PEC 6B, see Figs. 1 and 2) (Ref. 2). It was deployed on the exterior of the ISS during the STS-123 shuttle mission on March 22, 2008. The experiment was oriented to receive directed AO exposure during the majority of the mission. The experiment remained in low Earth orbit (LEO) for approximately 1.45 years, until it was retrieved during the STS-128 shuttle mission on September 1, 2009. Figure 1 shows MISSE 6A and 6B on the exterior of the Columbus Laboratory, as imaged in March 2008 during the STS-123 mission.

The set of multilayered samples flown on MISSE 6B was designed to compare the atomic oxygen erosion yield, Ey, (volume removed by oxidation per incident atom) of layers of Kapton H polyimide with no spacers between layers with that of layers of Kapton H with spacers between layers. It was theorized and confirmed by the tests in this paper that as atomic oxygen begins to erode through the layers of Kapton H, the erosion yield increases for layers that are spaced compared to that of a solid, thick, single layer or for a stacked sample with no or minimal spacing between the layers, because of trapping of atomic oxygen after it begins to penetrate through the layers of Kapton (Ref. 1).

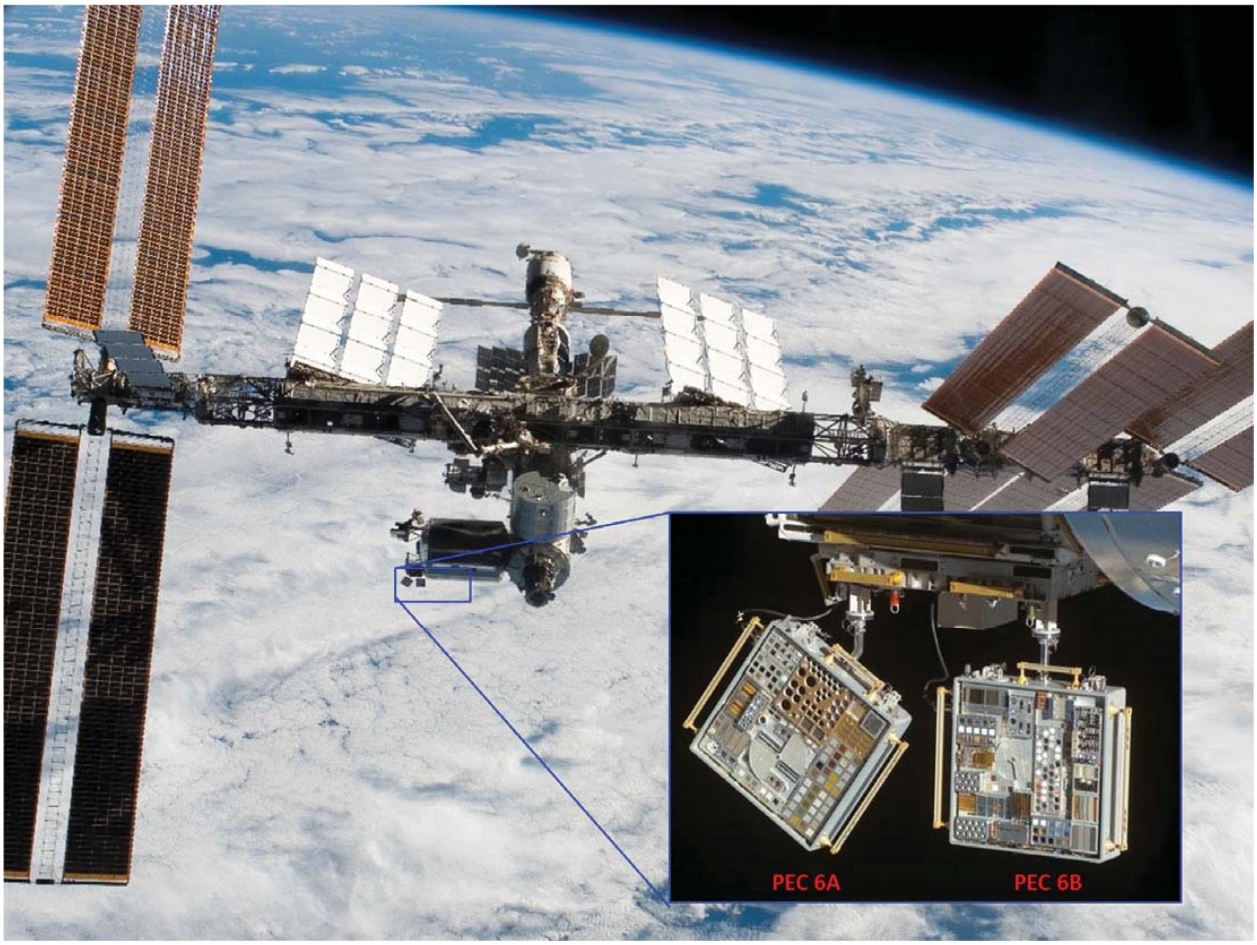


Figure 1.—The International Space Station (photographed in March 2008) with a close-up photo of MISSE 6A and 6B on the Columbus Laboratory (Ref. 3).

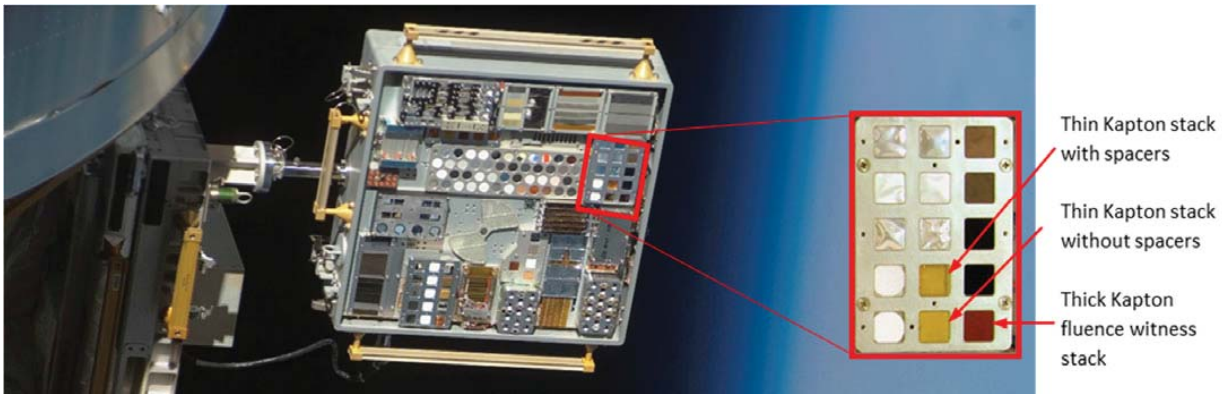


Figure 2.—MISSE 6B on the Columbus Laboratory showing the location of the W6 tray and the multilayer samples (Ref. 2).

The space experiment consisted of a 1 in. (2.54 cm) square Kapton H fluence witness sample (W6-5) consisting of three 0.005 in. (0.0127 cm) thick layers and two sets of 1 in. (2.54 cm) square samples each consisting of 10 layers of 0.0005 in. (0.00127 cm thick) Kapton H with one (W6-10) or two (W6-9) 5 mil thick Kapton H samples at the bottom. The purpose of the thick Kapton H layers on the bottom of the thin layered samples was simply to make sure that some Kapton H remained in case of a high fluence exposure due to delayed retrieval of the MISSE-6 carrier. One sample (W6-10) was flown with no intentional spacing between each layer even though the sample probably contained some random small gaps due to the wavy nature of the very thin sample layers. The other sample was flown with 0.014 in. (0.0356 cm) thick aluminum spacers between each of the 0.0005 in. (0.00127 cm thick) layers of Kapton H. Both samples were exposed in the ram direction on MISSE 6B for 1.45 years and were exposed to an atomic oxygen fluence of $1.90\pm0.05\times10^{21}$ atoms/cm^2 as determined by the Kapton H fluence witness sample (Ref. 2). Because of the mission fluence, only the top layer of the 0.005 in. Kapton H fluence witness sample experienced erosion. Table 1 compares the atomic oxygen erosion yields of a 0.005 in. (0.00127 cm) single sheet of Kapton H (i.e., the Kapton witness fluence sample W6-5), along with the thin layered samples W6-9 and W6-10 based on post-flight weight measurements (Refs. 2 and 4).

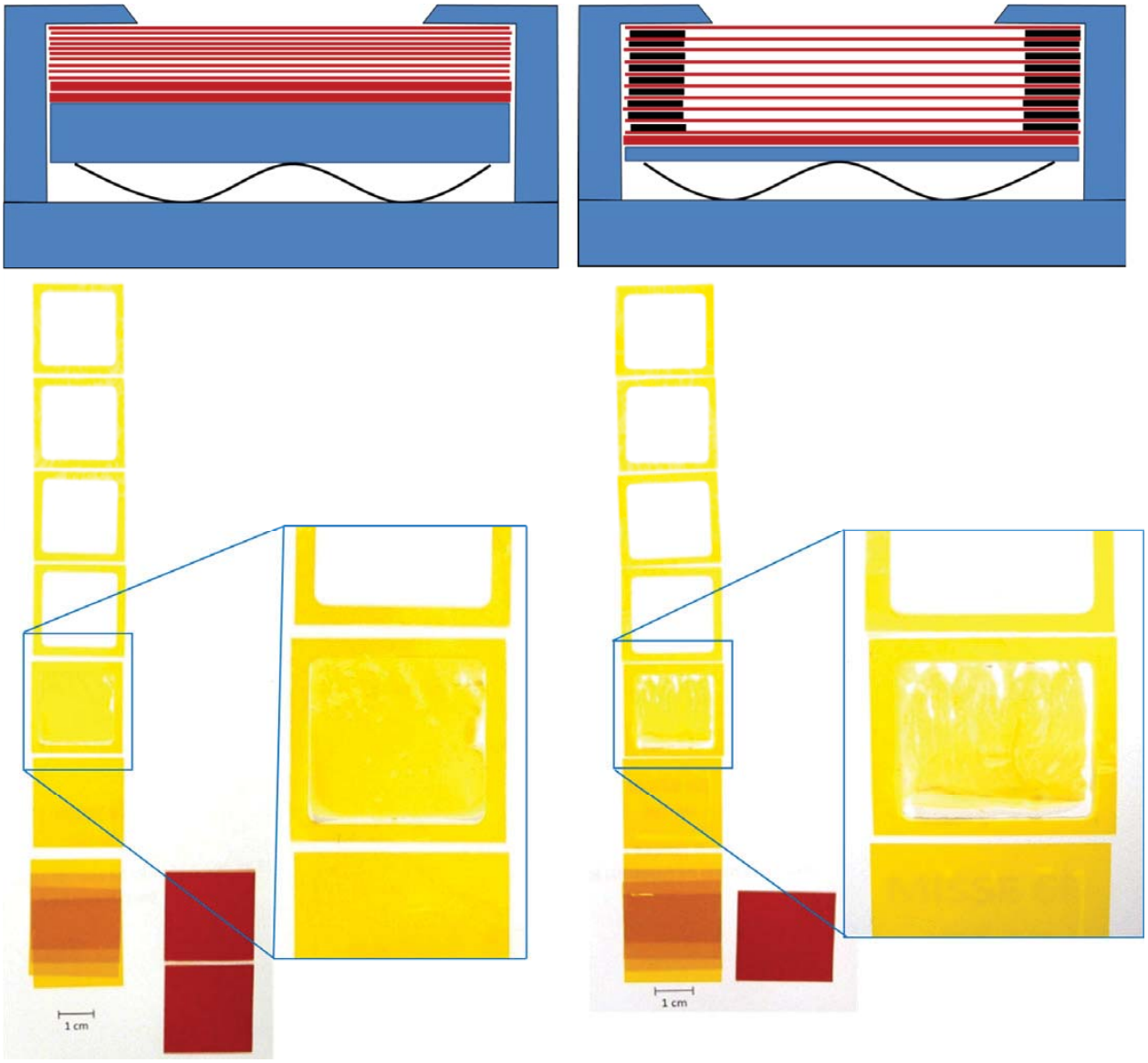
The slightly higher erosion yield (2.67 percent) of the layered sample with no spacers (3.08×10^{-24} cm^3/atom) compared to the thick single layer (3.00×10^{-24} cm^3/atom) is thought to be due to random small gaps due to the wavy shape of the samples. Figure 3 compares post- flight photographs which show the differences between the tightly-spaced and widely-spaced layered MISSE 6B Kapton H samples (Ref. 2).

As can be seen from Figure 3, the atomic oxygen eroded completely through the top four layers of Kapton H, and the fifth layer down shows indications that there was a locally higher flux at the perimeter of the samples. Such enhanced edge erosion has also been observed on many samples on this and other MISSE missions (Ref. 5). This phenomenon is thought to be due to scattered atomic oxygen from the 45° chamfered edges of the sample holder which added to the direct ram atomic oxygen flux. A MISSE Atomic Oxygen Scattering Chamber Experiment with atomic oxygen scattering off an aluminum base has been found to not scatter in a cosine distribution but predominantly at 45° from the arriving direction which may contribute to some enhancement of oxidation in the vicinity of the chamfered edges (Ref. 6).

The intent of this paper is to improve the interaction parameter values used for Monte Carlo atomic oxygen interaction modeling to determine if the erosion yield results of the space exposure can be predicted.

TABLE 1.—COMPARISON OF ATOMIC OXYGEN EROSION YIELDS OF MISSE 6 LAYERED KAPTON H SAMPLES

Sample configuration	Measured atomic oxygen erosion yields, cm^3/atom
1 to 0.005 in. thick layer (MISSE 6 Sample W6-5)	$3.00\pm0.07\times10^{-24}$
10 to 0.0005 in. thick layers with no spacers (MISSE 6 Sample W6-10)	3.08×10^{-24}
10 to 0.0005 in. thick layers with 0.014 in. spacers (MISSE 6 Sample W6-9)	3.25×10^{-24}



(a) (b)

Figure 3.—Schematics and post-flight photos of thin stacked Kapton H samples, showing individual sample layers: (a) Kapton H (10 to 0.5 mil layers plus two 5 mil layers at bottom) with no spacers and (b) Kapton H (10 to 0.5 mil layers plus one 5 mil layer at bottom with 0.014 in. thick Al spacers in between each layer).

Monte Carlo Computational Model

A Monte Carlo computational model has been developed to mathematically simulate the erosion of polymers by modeling atomic oxygen interaction on and oxidation of Kapton H (Refs. 7 to 9) using a finite array of cells to represent the polymer. The model is a two dimensional array of simulated polymer square cells (1000×1000 cells) that are removed when atomic oxygen oxidizes them. The model allows great flexibility in assumptions so that the interaction parameters can be tuned to replicate observed erosion in a variety of atomic oxygen environments including ram or sweeping hyperthermal low Earth orbital atomic oxygen, or isotropic thermal energy oxygen representative of radio frequency plasma ashers. Atoms are mathematically impinged upon a test surface in random locations upon a polymer surface or a surface that adjoins a polymer.

Upon impact with a non-reactive surface such as aluminum or silicon dioxide, the atomic oxygen can recombine to become chemically inactive or remain atomic and scatter with reduced energy in either a specular, random cosine (Lambertian) direction, or a prescribed direction relative to the local surface normal. After scattering, the atoms then have another chance of reacting, recombining, or scattering.

If the atomic oxygen impacts a polymer with volatile oxides it can react causing the removal of a computational cell or it can be ejected without reaction in a similar manner as scattering off nonreactive materials. An energy dependent reaction probability, P_E, was assumed given by

$$P_E = ce^{-E_A/E}$$

Where c = 3.178×10^{-24}

E_A = activation energy = 0.26 eV based on optimization (Ref. 10)

E = atomic oxygen impact energy, eV = 4.5 eV

In addition to energy dependence of reaction there appears to be a reaction probability dependence on the arriving direction relative to the local surface normal direction. This reaction probability dependence, P_C is given by

$$P_C = (\cos\ \theta)^n$$

Where θ = the angle between the arriving atomic oxygen direction and the local surface normal

n = cosine exponent = 0.5 based on optimization (Ref. 10).

Because the Monte Carlo model is two dimensional, the arriving atomic oxygen can be assumed to be in the horizontal or vertical plane relative to the Earth's surface. There is a slightly different angular distribution in arriving atoms for each plane due to the combined effects of co-rotation of the Earth's atmosphere, orbital inclination, and the hot Maxwell Boltzman distribution of the thermospheric atomic oxygen (Ref. 10).

An analysis of the results of the LEO reaction of atomic oxygen with protected Kapton H at coating defect sites, allowed the Monte Carlo interaction parameters to be optimized to produce erosion prediction results that replicated LEO results (Ref. 8). These interaction parameters are listed in Table 2. It is the differences in erosion yield of atomic oxygen interacting with multilayer films that may help optimize the Monte Carlo interaction parameter values to improve the model's ability to replicate space results.

TABLE 2.—MONTE CARLO INTERACTION PARAMETERS (REF. 10)

Interaction parameter	Value
Atomic oxygen initial impact reaction probability	0.09
Activation energy, E_A, in eV for energy dependent reaction probability	0.26
Atomic oxygen reaction probability dependence exponent upon angle of impact, n, where the reaction probability = $P_E \cdot (\cos \theta)^n$ where θ is the angle between the arrival direction and the local surface normal and P_E is the energy dependent reaction probability at normal incidence	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, f, 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	0.045
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	0.5
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	0.9
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

Results and Discussion

Due to the finite size of the Monte Carlo model simulation, atomic oxygen erosion of five layers of Kapton H was not possible for the current model. However the simulation of four layers was possible. Simulation of multiple layers of Kapton with no spacers between the layers would appear to a computational model as identical to a single sheet of Kapton. Thus a minimum spacing of one Monte Carlo cell (equivalent to 0.056 mills) was used to represent a gap with no spacers between layers. For modeling consistency with the space experiment, the ratio of the polymer thickness to the spacer thickness was made to be the same for both the space experiment and the Monte Carlo model. The number of Monte Carlo atoms entered was the same (400,000) for each configuration simulated. Use of the Table 2 Monte Carlo interaction parameters resulted in slightly higher erosion yields for the single thick polymer layer than either of the two thin multilayer configurations. As a result computational tests were conducted with various modifications to the values of the Table 2 interaction parameters in an effort to more closely match the relative erosion yields of Table 1 as well as the undercut cavity data from Reference 10. Figure 4 compares the erosion from a crack defect in an aluminized Kapton H sample retrieved from the Long Duration Exposure Facility (Ref. 10). The modified Monte Carlo interaction parameter values are listed in Table 3.

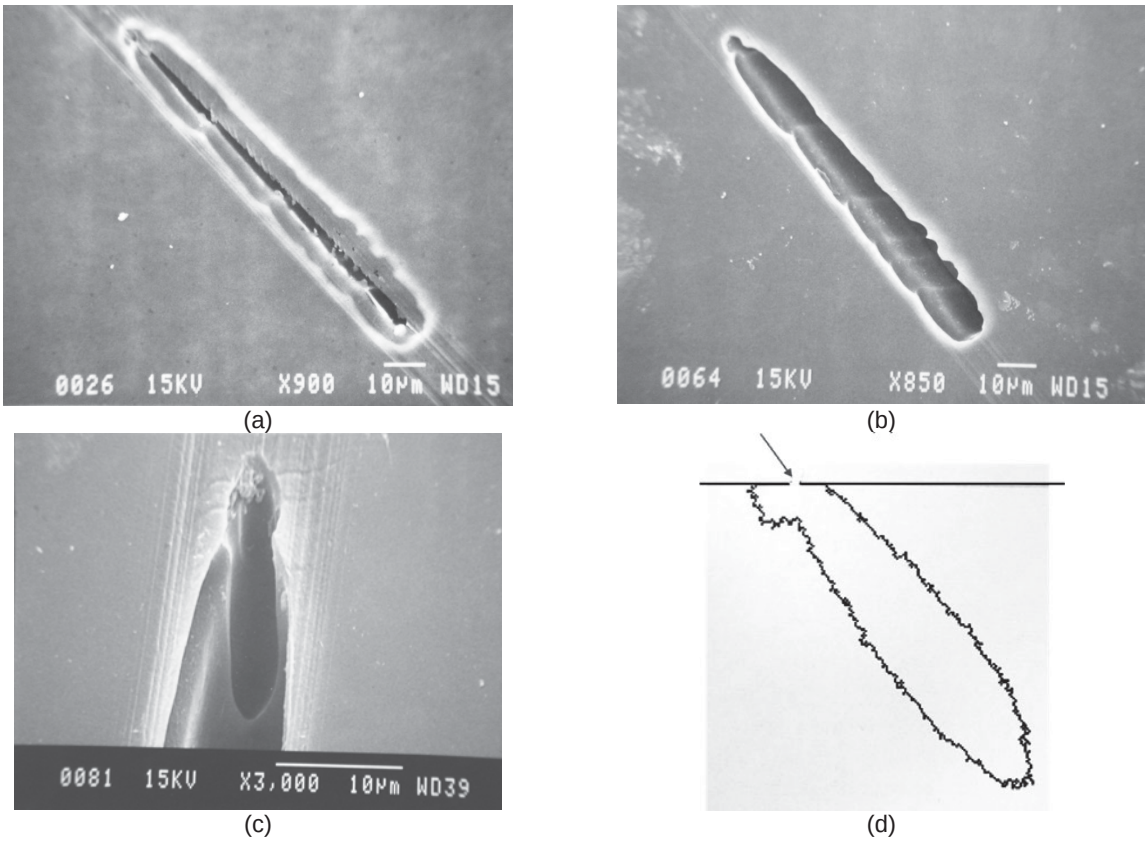


Figure 4.—Comparison of the erosion profiles from a crack defect in an aluminized Kapton H sample retrieved from the LDEF (Ref. 10) and Monte Carlo results from using the interaction parameters of Table 3: (a) Scanning electron microscope image showing crack defect in the aluminum coating, (b) Scanning electron microscope normal incident image showing undercut cavity below crack with aluminum coating chemically removed, (c) Scanning electron microscope tilted image showing undercut cavity below crack with aluminum coating chemically removed, and (d) Monte Carlo simulation using Table 3 interaction parameters.

TABLE 3.—MODIFIED MONTE CARLO INTERACTION PARAMETERS

Interaction parameter	Value
Atomic oxygen initial impact reaction probability	0.062
Activation energy, E_A, in eV for energy dependent reaction probability	0.26
Atomic oxygen reaction probability dependence exponent upon angle of impact, n, where the reaction probability = $P_E \bullet (\cos \theta)^n$ where θ is the angle between the arrival direction and the local surface normal and P_E is the energy dependent reaction probability at normal incidence	0.5
Probability of atomic oxygen recombination upon impact with protective coating	0.33
Probability of atomic oxygen recombination upon impact with polymer	0.3
Fractional energy loss, f, upon impact with polymer	0.4
Fractional energy loss upon impact with protective coating	0.05
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	0.2
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	0.5
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	0.9
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

TABLE 4.—COMPARISON OF RELATIVE ATOMIC OXYGEN EROSION YIELDS

Space sample configuration	Measured atomic oxygen erosion yields, cm^3/atom	Model sample configuration	Monte Carlo model predicted erosion yield, cm^3/atom
One thick layer	$3.00\pm0.07\times10^{-24}$	One thick layer	$3.00\pm0.07\times10^{-24}$
10 thin layers with no spacers	$3.08\pm0.07\times10^{-24}$	4 thin layers with 0.056 mil spacers	$3.205\pm0.08\times10^{-24}$
10 thin with 14 mil spacers	$3.25\pm0.07\times10^{-24}$	4 thin layers with 14 mil spacers	$3.253\pm0.08\times10^{-24}$

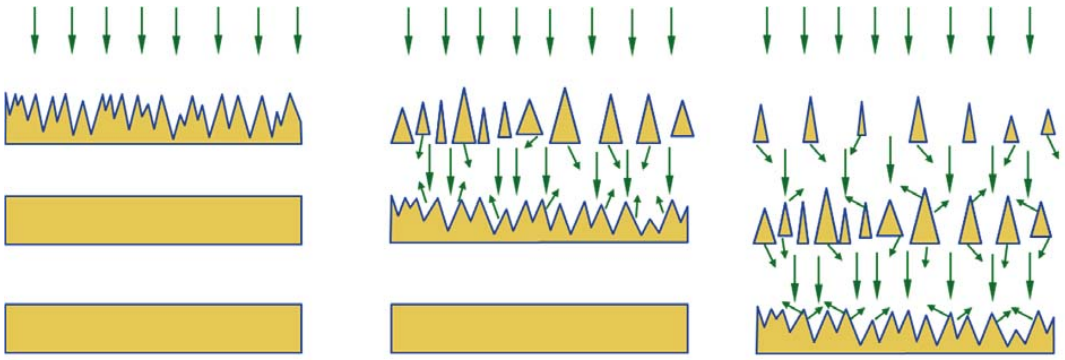
These interaction parameters produce the relative erosion yields shown in Table 4 which are compared to those resulting from the in-space experiment. As one can see, the modified Monte Carlo model predicted erosion yield for the sample configuration with 14 mil spacers is in agreement with that measured from the space exposure. However, the modified Monte Carlo model prediction for the sample configuration with no spacers is only slightly less than that for the widely spaced sample. The Monte Carlo modeling indicated that the sample with 14 mil spacers would have an 8.4 percent increase over solid Kapton H (compared to the space data showing 8.3 percent increase). However the sample with no spacers (simulated by 0.056 mil spacers) was predicted to have 6.8 percent increase over solid Kapton H (compared to space data showing 2.7 percent increase). A possible explanation for this result may be that the multilayer sample without spacers may have been closer together than the 0.056 mil gap assumed by the Monte Carlo model or that further refinement of the model may better predict the results for a small gap.

Figure 5 compares a schematic of a three layer polymer showing the erosion at three different fluence levels indicating how the diffuse scattering of the atomic oxygen off the polymer tends to trap the atoms contributing to increased opportunity to react either at a layer above or below. This results in greater erosion yields for polymers with space between layers.

When cones become completely eroded such that they could be free standing, they may loosely attach themselves by means of van derWaals forces to adjoining polymer until they are completely oxidized (Ref. 1). As the fluence is increased, there is trapping of unreacted and scattered atoms for both single and multiple layer films as shown in Figure 6.

As can be seen from Figure 6, there is a gradual increase in erosion yield due to trapping of scattered atoms between the left-standing surface cones as directed erosion gradually increases. This occurs as the number of Monte Carlo atoms is increased to the extent that valleys of cones on the textured surface form holes in the second film layer, then additional trapping of unreacted atoms between the layers of films begins to occur (after ~60,000 atoms have been entered) and continues as another layer is gradually penetrated. Figure 7 shows holes at cone valleys of a thin film layered Kapton H sample (3 to 5 mil (127 μm) thick layers) exposed to LEO ram AO for 4 years on MISSE 2 (Ref. 1). The enhancement in atomic oxygen erosion yield is dominated by only two layers of polymer films and becomes a constant erosion yield with fluence after that. It is not clear to what degree the enhancement in Ey would occur for different polymers or different thicknesses of a polymer.

The indication of locally higher flux at the perimeter of the samples shown in Figure 3 was found to be consistent with Monte Carlo simulation using the optimized interaction values listed in Table 3. The average depth of perimeter erosion was found to be 6 percent greater than the erosion at the center of the samples based on averaging 10 Monte Carlo simulations. Based on the enhanced erosion observed near the edge and predicted Monte Carlo results it is recommended that samples used for erosion yield evaluation under normal incidence atomic oxygen use a reverse bevel (largest opening facing space) on sample holders and with the smallest opening used for defining the effective exposure area.



(a) (b) (c)

Figure 5.—Comparison of Monte Carlo predicted erosion patterns for a three layer polymer sample exposed to atomic oxygen in LEO at three different fluence levels: (a) Low fluence, (b) Medium fluence, (c) High fluence.

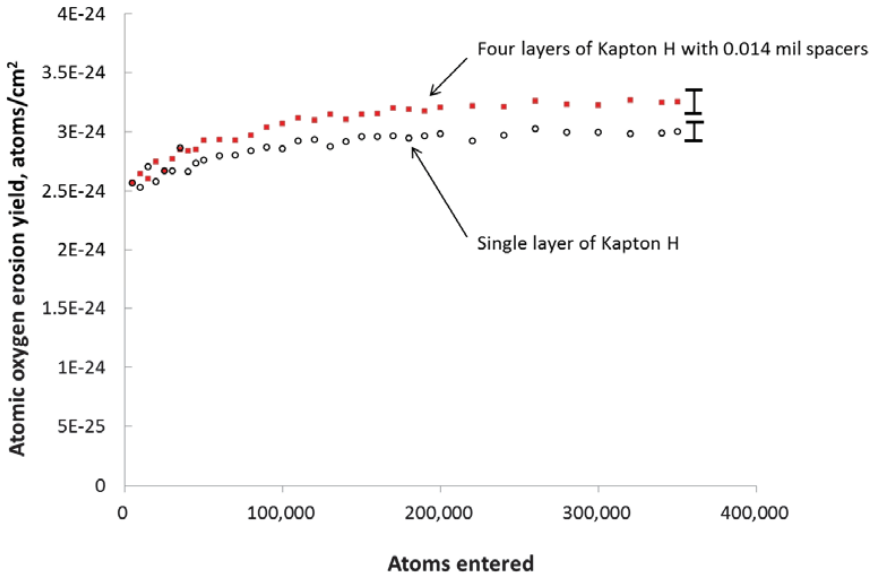
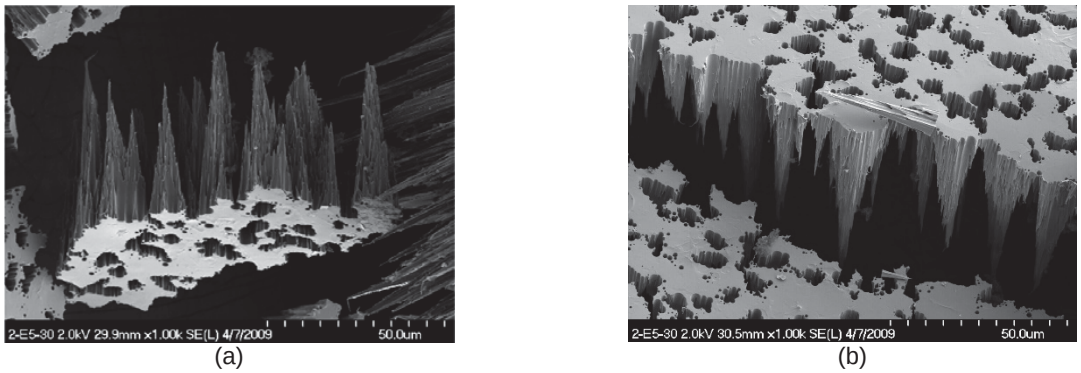


Figure 6.—Erosion yield dependent fluence for single and multilayer space films. The error bars on the right of each plot represents the span of ± one standard deviation for the respective data points which also includes the uncertainty of the erosion yield of Kapton H ($\pm 7.41 \times 10^{-26}$ atoms/cm^2 from Ref. 12).



(a) (b)

Figure 7.—Images of cone morphologies and holes at cone valleys observed from the back surface of the second layer of a multi-layered sample flown on MISSE 2: (a) An isolated cone cluster (1 kX, 45° tilt), and (b) A crack in the film where the cone structure is visible (1 kX, 45° tilt).

Summary

A space experiment flown as part of the Materials International Space Station Experiment 6B (MISSE 6B) was designed to compare the atomic oxygen erosion yield (Ey) of tightly spaced thin layers of Kapton H polyimide (no spacers between layers) with slightly spaced layers of Kapton H (thin spacers between layers). The flight results indicated that there was a 2.7 and 8.3 percent increase in erosion yield respectively for tightly stacked (no spacers) and loosely stacked (with spacers) Kapton H films compared to a solid Kapton H sample. Monte Carlo computational modeling was performed to optimize atomic oxygen interaction parameter values to match the results of both the MISSE 6B multilayer experiment and the erosion profile from a crack defect in an aluminized Kapton H sample retrieved from the LDEF. This resulted in the Monte Carlo modeling producing close agreement with space results for the layered sample with spacers compared to solid Kapton (8.4 percent increase for Monte Carlo predictions compared to 8.3 percent increase for space results) but a higher than expected (6.8 percent increase for Monte Carlo predictions compared to 2.7 percent increase for space results) for the stacked sample without spacers. The Monte Carlo results also predicted the observed space results that scattering from the beveled edges of the sample holders caused a slight (6 percent) enhancement in erosion of samples near their perimeters.

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