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An Investigation of Stress Dependent Atomic Oxygen Erosion of Black Kapton Observed on MISSE 6

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Abstract

Black Kapton XC polyimide was flown as part of the Polymer Film Tensile Experiment (PFTE) on Materials International Space Station Experiment 6 (MISSE 6). The purpose of the experiment was to expose a variety of polymer films, typical of those used for thermal control blankets or supporting membranes on Earth orbiting spacecraft, to the low Earth orbital (LEO) environment under both relaxed and tension conditions. Black Kapton XC under tensile stress experienced a higher erosion rate during exposure in LEO than the same material that was flown in a relaxed condition. Testing conducted to determine the magnitude of the stress and erosion dependence using a ground-based thermal energy atomic oxygen plasma showed a slight dependence of erosion yield on stress for Kapton HN and Black Kapton XC, but not to the extent observed on MISSE 6. More testing is needed to isolate the factors present in LEO that cause stress dependent erosion.

Introduction

Thin film polymers are used in many spacecraft applications for thermal control (multi-layer insulation and sunshields), as lightweight structural members (solar array blankets, inflatable/deployable structures) and have been proposed for propulsion (solar sails). Polymers in these applications are exposed to the space environment and are vulnerable to degradation by solar ultraviolet radiation, solar flare x-rays, solar wind electrons and protons trapped in Earth's magnetic field, temperature and orbital thermal cycling, and low Earth orbit (LEO) atomic oxygen (Ref. 1). In applications where the polymer film is under tension while exposed to these environmental factors, it is important to understand the effect of stress in combination with the environment on the durability of thin polymer films. Polymer films were flown previously in the Polymer Film Thermal Control Experiment and the Gossamer Materials Experiment as part of Materials International Space Station Experiment (MISSE) 1 as well as on MISSE 3, MISSE 4, and MISSE 5 (Refs. 2 and 3). The MISSE 6 exposure is different from prior such experiments in that a number of the samples were designed to be exposed while under tension to better simulate their use in space and determine if the stress level affects the durability. The dog-bone shaped tensile samples of polymers were flown on both the ram and wake facing sides of the MISSE 6 Passive Experiment Containers (PECs). A description of all of the samples flown as part of PFTE is contained in Reference 4. This paper focuses on the results observed for Black Kapton XC flown on MISSE 6 and the results of ground testing conducted in an attempt to gain a better understanding of the erosion observed in LEO.

Flight and Ground Based Experiment Description and Procedure

MISSE 6 Environment and Ground Based Atomic Oxygen Exposure

MISSE 6 was composed of two Passive Experiment Containers (PECS), 6A and 6B. Both PECS had one side of the suitcase style containers facing ram and the other side facing wake. They were both installed on the European Columbus module of the International Space Station (ISS) on March 22, 2008, during the flight of STS-123. They were retrieved on September 1, 2009, by the crew of STS-128 after slightly over 17 months in LEO. Environment exposure condition estimates that have been determined to date are the atomic oxygen exposure level on each side and the UV radiation level (Refs. 5 to 7). There were also two Kapton HN/VDA tensile dogbones that were flown as part of this experiment on the wake side of MISSE 6A from which scanning electron microscope images of protected locations on the surface were used to determine the erosion depth and ultimately the atomic oxygen fluence (Ref. 4). All of the data seemed in good agreement with an estimate of the atomic oxygen arrival fluence for the ram side of 6A and 6B of approximately 2×10^{21} atoms/cm^2, and for the wake side approximately 1.2 to 1.4×10^{20} atoms/cm^2. This indicates that the wake side of MISSE 6, which was to have received very low atomic oxygen exposure was oriented in the ram direction long enough to have received an atomic oxygen dose about 6.5 percent that of the ram oriented side. Estimates of the UV radiation exposure in equivalent sun hours (ESH) were 2600 ESH for the ram sides of 6A and 6B and 1950 ESH for the wake sides of 6A and 6B (Ref. 7). Temperature, thermal cycling, and ionizing radiation estimates were not available at this time.

Exposure to atomic oxygen for the ground based tests was conducted in an AXIC LF-5 plasma system pumped with a Varian SH110 Scroll Pump. Air was used as the feed gas and a radio frequency power of about 36 W was applied to the internal electrodes to form the thermal energy (~0.04 eV) atomic oxygen plasma that was used to expose samples placed inside the chamber. The effective atomic oxygen fluence for these tests was about a factor of 6 higher than for the flight samples.

Experiment Design for Application of Tensile Stress

The flight experiment was designed to allow some of the polymer dog-bone type samples to be exposed under a tensile load typical of expected conditions for the James Webb Space Telescope sunshield. The tensile load of approximately ~2.22 N (0.5 lb) was applied by mounting the sample in a holder similar to that shown on the left side of the photo in Figure 1 and then compressing a spring with a spring constant of ~385 N/m (2.2 lb/in.) by approximately ~0.0058 m (0.227 in.) to put the sample under an approximately constant tensile load. The drawing in Figure 1 shows a double sample holder where the sample on the left did not have an applied tensile stress and the one on the right did. For the samples exposed under stress, the resulting stress was dependent on the polymer film thickness per Equation (1) with an average gage width of approximately 0.0032 m (0.126 in.). For the Black Kapton XC samples, the applied stress was ~2.76×10^7 N/m^2 (~4000 psi) as the films were 2.54×10^{-5} m (0.001 in.) in thickness.

$$\text{Stress} = (\text{Force/Area}) = (\text{Force})/(\text{Gage width}) * (\text{Thickness}) \quad (1)$$

For the ground based testing, the same sample holders were used but slightly modified to allow other spring compression lengths to achieve a wider variety of tensile stress on the samples. This was accomplished by modifying the rod underneath to allow greater travel of the spring and through the use of copper u-shaped shims to compress the spring and place the sample under a fixed tensile load.

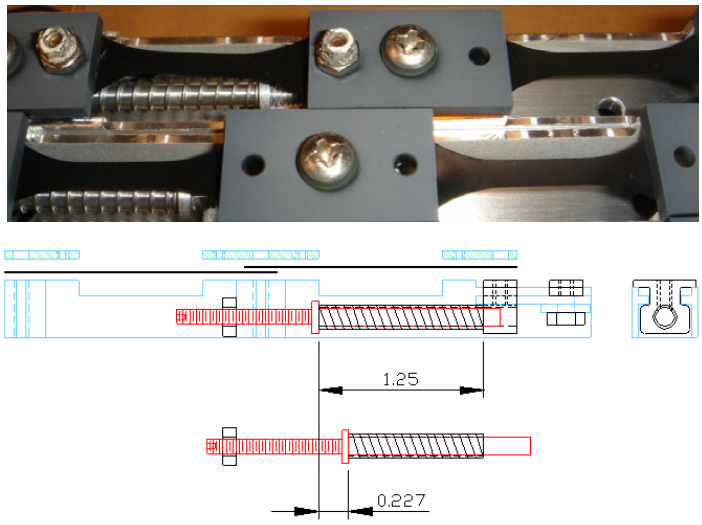

Figure 1.—Photo of stressed (left) and unstressed (right) sample holders from above, and a side view drawing of a holder showing the unstressed sample position on the left and the stressed on the right. (Dimensions are in inches.) The stressed sample is fixed on the left side and allowed to move to the right by having the mount hole on the right slotted. Tension is supplied by compression of the spring.

Sample Description

All of the samples, both for flight and for ground testing were punched from polymer sheets using a die manufactured according to specimen "Type V" under the American Society for Testing and Materials (ASTM) Standard D-638 (Ref. 8). The dog-bone shaped die had a gage length of 7.62 mm (0.3 in.) and an average gage width of 3.21 ± 0.02 mm (0.126 in.). The Black Kapton XC (100XC10E7), a carbon pigmented polyimide, was manufactured by DuPont. It had a vapor deposited aluminum coating on the back that was originally intended to provide electrical contact for an active sample break indication but due to some wiring issues prior to fight, the flight samples were not wired for active monitoring. The unflown extra control samples were used for the ground experiment testing. There were also Kapton HN samples flown on MISSE 6 but the ones on the ram side had a SiO_x protective coating on the space exposed side to prevent erosion by atomic oxygen, and the ones on the wake side were not under stress. For ground tests, Kapton HN manufactured by DuPont of 5.08×10^{-5} m (0.002 in.) in thickness was used to obtain atomic oxygen flux maps and for stress erosion comparison for the ground based experiments due to the limited supply of Black Kapton XC samples. The Kapton HN samples were punched from the same die, but a thin layer of magnetron sputter deposited gold was applied to the back of the sample to prevent atomic oxygen erosion of the back side. Thin pieces of aluminum foil were cut to fit and wrapped over the grip and transition area on the front side of the Kapton HN and Black Kapton XC samples, so that only the gauge length would be exposed to atomic oxygen for more accurate measurement of the erosion at a fixed stress level, before they were secured in the holder.

Analysis

The mass of the samples before and after exposure was measured using a Sartorius ME5 microbalance. Samples were dehydrated at a vacuum of approximately 8.67 Pa (65 mTorr) for 48 hr prior to weighing to minimize errors due to absorption of moisture from the air. The change in mass of the Kapton HN was used to determine the atomic oxygen fluence at the surface according to ASTM E-2089 (Ref. 9). Change in mass was also used to determine the erosion during flight or in ground tests. Overall

and close up photos were taken of the samples post flight, and before and after ground testing, using a Sony Cybershot DSC T-9 camera. Initial observations were recorded and a few selected samples were gold coated and mounted for scanning electron microscopy with a JEOL JSM-6390 LV scanning electron microscope (10 keV) with energy dispersive analysis by X-rays (EDAX).

Results and Discussion

Kapton XC Samples on MISSE 6

Two unstressed samples of black Kapton (XC) were exposed on the ram side of MISSE 6. Both samples appeared to show evidence of texturing of the surface with a darker appearance near each end of the dog-bone sample. One of these samples designated AO-S-1 was inadvertently put under stress when one end of the sample holder was moved, which changed the overall sample length by ~0.0017 m (~0.068 in.). This sample was installed in a sample holder initially designed for putting the sample under a tensile load so the holder on the top side of the sample had a slotted mount hole. It appears that the sample was inadvertently bumped and the one end of the holder moved putting the sample under a high tensile stress even though it was not initially intended to be stressed. There was a cable that was passed up between this sample tray (G3) and the neighboring tray very close to AO-S-1 which may have provided the opportunity for inadvertent bumping of the one end of the sample holder during experiment installation. The movement put a strain on the sample (for an undetermined length of time) of ~0.07. This represents approximately 26 percent of the maximum strain for Kapton XC. The resulting stress on the sample was 2.32×10^8 N/m^2 (33,600 psi) which is greater than the yield strength of Kapton XC. The sample had a silvered appearance on the end that was stretched and distorted which is evident from the photograph in Figure 2.

Figure 2.—Kapton XC flown on ram side of MISSE 6 (AO-S-1) showing stretching of the sample at the silver area at the top of the sample in the photograph

The appearance of the sample raises two questions. The first is why the stretched end appears silver and the second is why the black Kapton appears to be darker in the region of higher stress? In order to try to answer these questions, SEM and EDAX analysis was performed on the sample at selected locations shown in Figure 3, SEM images in Figure 3 show a significant change in surface morphology from the center to the edge. The center portion has a smooth lumpy appearance typical of black Kapton which progresses to an area which looks as if it had a thin film gossamer coating on it with many cracks perpendicular to the pull direction. There are fine cone-like peaks in areas where there is cracking which progresses to almost all peaks with thin wisps of film on the surface nearer to the silver area. At the edge where the sample separated, there are only a few short peaks remaining. EDAX scans indicate mostly carbon and oxygen signals in the central region progressing to a high concentration of aluminum near the stretched end. This sample was originally intended to be put under stress and wired so there was a vapor deposited aluminum coating on the back side. It appears that as the stress on the sample is increased, the erosion rate of the black Kapton increases which results in first development of surface texture cones and a thin film of ash from oxidation of the black Kapton. This progresses to loss of ash and erosion of the mostly carbon cones to the point at which the vapor deposited aluminum is predominantly what is left looking like a blanket of snow at the base of the remaining carbon peaks. If erosion of the Kapton XC is dependent on the level of stress, then there should be an observable difference between the stressed and unstressed samples of Kapton XC that were flown on the wake side of MISSE 6.

The stressed and unstressed samples of Kapton XC flown on the wake side did appear very different from each other. The two unstressed samples appeared slightly textured while the two stressed samples were very dark matte black in appearance. A photo of the two sample pairs is shown in Figure 1. The stress level during exposure was ~2.76×10^7 N/m^2 (~4000 psi) and the strain was ~0.008 which represents about 3 percent of the maximum strain. The stress on the sample was about 24 percent of the tensile strength. This does not appear to be a significant amount of strain on the material but is enough to cause a difference in the appearance of the erosion of the material due to oxidation by atomic oxygen. Figure 4 shows side-by-side SEM images at 45° tilt of the stressed (UV-S-2) sample of Kapton XC on the left and the unstressed (UV-U-2) sample of Kapton XC on the right. There is more surface texturing occurring on the stressed sample than the unstressed sample as can be seen in the top 2/3rds of the image. The bottom 1/3 was under the sample mount and protected from erosion by atomic oxygen. The unstressed sample is only slightly different in appearance to the unexposed surface, while the surface of the stressed sample has undergone very noticeable erosion.

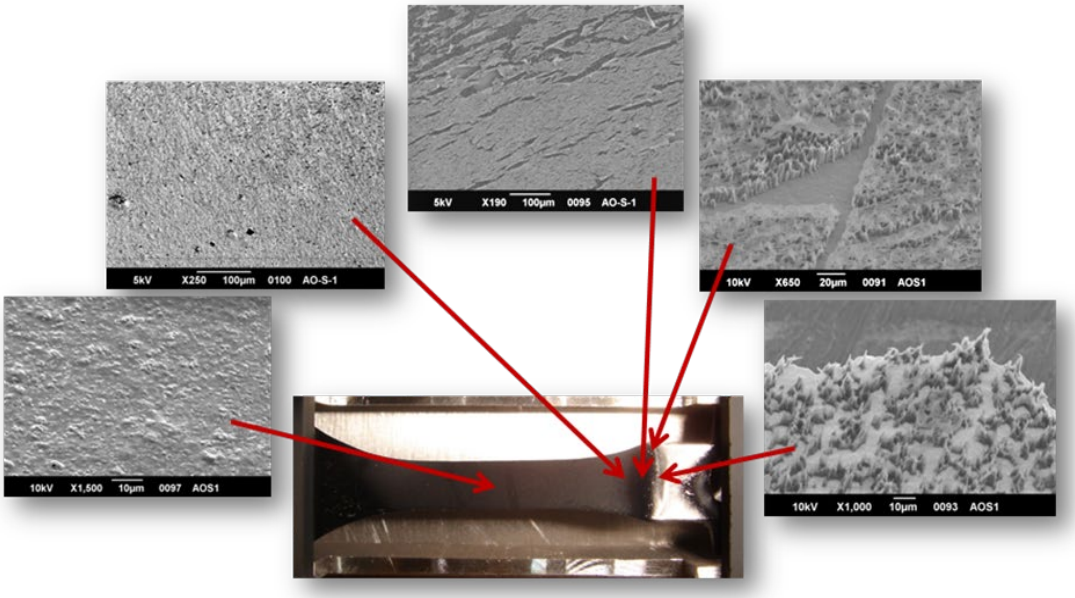

Figure 3.—SEM images of selected positions on sample AO-S-1 exposed on the ram side of MISSE 6.

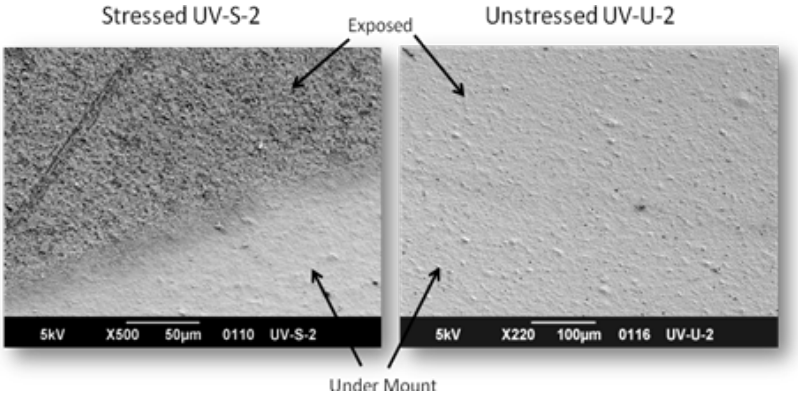

Figure 4.—Scanning electron microscope images of stressed (left) and unstressed (right) Kapton XC at 45° tilt. Top approximately 2/3 was exposed to the space environment on the wake side of MISSE 6 while the bottom ~1/3 was protected by the clamp holding the sample in place.

To better quantify the erosion, both the stressed and unstressed flight samples (UV-S-1 and UV-U-1) were dehydrated and weighed. The samples had not been weighed prior to flight, so a pre-flight mass estimate was made by taking four measurements on each of six control specimens and using the average mass as the pre-flight mass for both samples. The resulting erosion yield for each sample (cm^3/atom) was $7.01\times10^{-25} \pm 9.94\times10^{-25}$ for the unstressed sample and $2.93\times10^{-24} \pm 1.84\times10^{-24}$ for the stressed sample. The error is large due to the very low change in mass and the error in the pre-flight mass estimate. In spite of this, the erosion yield for the stressed sample was still greater than the error. A rough estimate from this data is that the erosion yield for Black Kapton XC at a tensile stress of ~2.76×10^7 N/m^2 (~4000 psi) is about a factor of 4 higher than for the same material not under stress. In order to obtain a better measurement and determine if there is a stress level dependence on erosion of Kapton and Black Kapton, ground tests were conducted with the samples at different stress levels.

Ground Testing Using Thermal Energy Atomic Oxygen

Dehydrated and weighed unstressed Kapton HN tensile samples were mounted in each of eight sample positions of the four modified flight sample holders sitting on an aluminum plate. The plate was placed in the vacuum chamber and the samples exposed to the RF atomic oxygen plasma. The mass change of each sample was used to determine the atomic oxygen flux at each position and the data from the flux map was used to correct the erosion data from the exposure tests with Kapton HN and Black Kapton XC under stress to account for spatial variation in the atomic oxygen arrival.

Kapton HN samples were installed in the same sample holders and four of the samples were loaded to varying stress levels with two unstressed samples included for fluence witnesses. The atomic oxygen arrival ratio for the two unstressed samples was within error of that observed for the flux map so the fluence at the locations of the stressed samples was estimated using the flux map and fluence measured at the control locations. Figure 5 contains a graph of the resulting erosion yield (cm^3 removed per incoming atom) as a function of tensile stress for Kapton HN. As can be seen from the graph, at stress levels above 2×10^7 Pa, the erosion yield exhibited a slight increase with increasing stress.

The erosion yield for two stress levels of Kapton HN normalized with respect to the unstressed erosion yield for that material was compared with that obtained for two Black Kapton XC samples exposed in the ground based atomic oxygen plasma chamber in the same positions and with the same spring compression level as that of the Kapton HN. The resulting stressed to unstressed erosion ratios shown in Figure 6 were within error of each other but much lower than the erosion ratio for the stressed to unstressed Black Kapton XC exposed on MISSE 6. The error in the flight data is not shown but is expected to be large in comparison to that for the ground based atomic oxygen exposure data. However, there was very little difference in the surface appearance of the ground test data compared with that observed on MISSE 6.

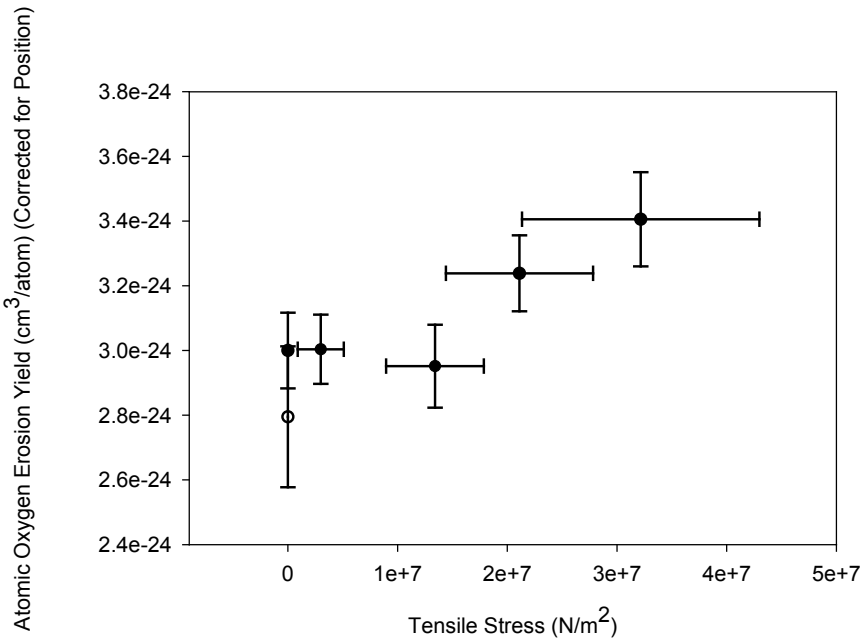

Figure 5.—Atomic oxygen erosion ratio as a function of stress for Kapton HN exposed to ground based thermal energy atomic oxygen.

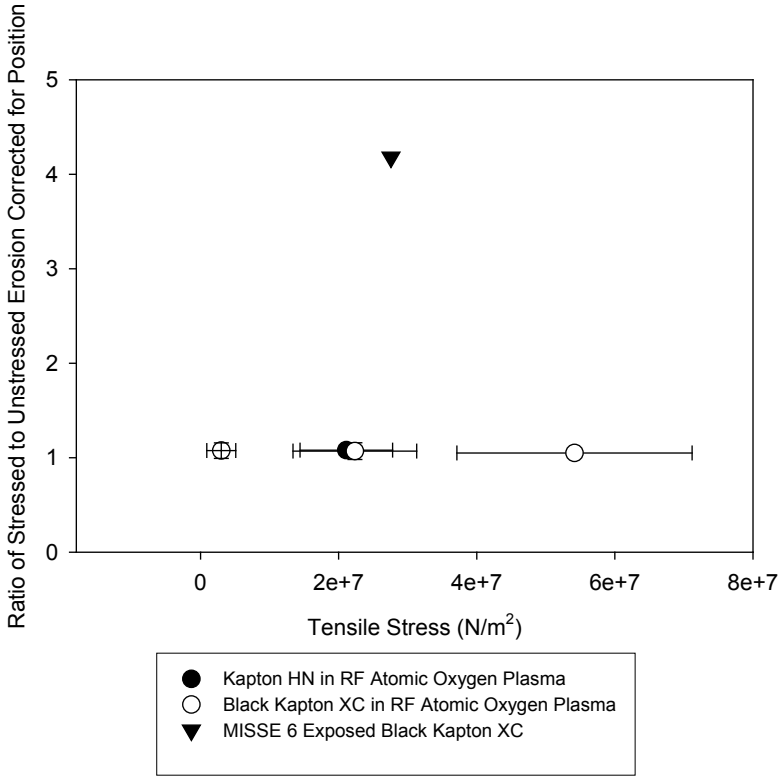

Figure 6.—Ratio of stressed to unstressed erosion yields for Kapton HN, and Black Kapton XC exposed to a ground based thermal energy atomic oxygen plasma as a function of tensile stress compared to that for Black Kapton XC exposed on MISSE 6.

Conclusions

Black Kapton XC exposed on MISSE 6 exhibited statistically a significantly higher erosion rate when placed under tensile stress as observed for the highly stressed sample on the ram side and two stress loaded samples on the wake side. Although a slight stress dependence was also observed in the ground based thermal energy atomic oxygen chamber for Kapton HN, and Black Kapton HN, the magnitude of the difference between the stressed and unstressed samples both in appearance and in the erosion yield was not the same as that observed in LEO. There are, however, differences between the ground based and LEO exposure environments which could cause this difference. There is an energy difference for the atomic oxygen (4.5 eV in LEO compared to 0.04 eV in the ground chamber) and different levels of UV radiation, temperature, and charged particles. The ground system also lacks energetic protons, electrons, and x-rays which are present in LEO. It is possible that two or more environment factors must be present to greatly affect the erosion rate of a stressed polymer such as Kapton HN and Black Kapton XC. Further testing is needed to isolate the factors which result in increased erosion under stress.

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13. SUPPLEMENTARY NOTES

14. ABSTRACT
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