



Bend-Test Results of the MISSE 7 Flexural Stress Effects Experiment After 1.5 Years of Space Exposure

Kate E. Snow

Hathaway Brown School, Shaker Heights, Ohio

Kim K. de Groh

Glenn Research Center, Cleveland, Ohio

Bruce A. Banks

Science Application International Corporation, Cleveland, Ohio

Edward A. Seckar

ZIN Technologies, Inc., Middleburg Heights, Ohio

NASA STI Program . . . in Profile

Since its founding, NASA has been dedicated to the advancement of aeronautics and space science. The NASA Scientific and Technical Information (STI) Program plays a key part in helping NASA maintain this important role.

The NASA STI Program operates under the auspices of the Agency Chief Information Officer. It collects, organizes, provides for archiving, and disseminates NASA's STI. The NASA STI Program provides access to the NASA Technical Report Server—Registered (NTRS Reg) and NASA Technical Report Server—Public (NTRS) thus providing one of the largest collections of aeronautical and space science STI in the world. Results are published in both non-NASA channels and by NASA in the NASA STI Report Series, which includes the following report types:

- **TECHNICAL PUBLICATION.** Reports of completed research or a major significant phase of research that present the results of NASA programs and include extensive data or theoretical analysis. Includes compilations of significant scientific and technical data and information deemed to be of continuing reference value. NASA counter-part of peer-reviewed formal professional papers, but has less stringent limitations on manuscript length and extent of graphic presentations.
- **TECHNICAL MEMORANDUM.** Scientific and technical findings that are preliminary or of specialized interest, e.g., "quick-release" reports, working papers, and bibliographies that contain minimal annotation. Does not contain extensive analysis.
- **CONTRACTOR REPORT.** Scientific and technical findings by NASA-sponsored contractors and grantees.
- **CONFERENCE PUBLICATION.** Collected papers from scientific and technical conferences, symposia, seminars, or other meetings sponsored or co-sponsored by NASA.
- **SPECIAL PUBLICATION.** Scientific, technical, or historical information from NASA programs, projects, and missions, often concerned with subjects having substantial public interest.
- **TECHNICAL TRANSLATION.** English-language translations of foreign scientific and technical material pertinent to NASA's mission.

For more information about the NASA STI program, see the following:

- Access the NASA STI program home page at <http://www.sti.nasa.gov>
- E-mail your question to help@sti.nasa.gov
- Fax your question to the NASA STI Information Desk at 757-864-6500
- Telephone the NASA STI Information Desk at 757-864-9658
- Write to:
NASA STI Program
Mail Stop 148
NASA Langley Research Center
Hampton, VA 23681-2199



Bend-Test Results of the MISSE 7 Flexural Stress Effects Experiment After 1.5 Years of Space Exposure

Kate E. Snow

Hathaway Brown School, Shaker Heights, Ohio

Kim K. de Groh

Glenn Research Center, Cleveland, Ohio

Bruce A. Banks

Science Application International Corporation, Cleveland, Ohio

Edward A. Seckar

ZIN Technologies, Inc., Middleburg Heights, Ohio

Prepared for the

2017 International Space Station Research and Development Conference

cosponsored by NASA, American Astronautical Society, and CASIS

Washington, D.C., July 17–20, 2017

National Aeronautics and
Space Administration

Glenn Research Center
Cleveland, Ohio 44135

Acknowledgments

We would like to thank Patty Hunt and Dr. Crystal Miller of Hathaway Brown School (HB) for their support of the NASA Glenn Research Center and HB collaboration, which made this research possible. We acknowledge and thank Joyce Dever of NASA Glenn for the initial MISSE 7 FSEE sample planning and pre-flight preparations. We would express our sincere appreciation to Gary Pippin (retired) of Boeing for providing the opportunity to fly the FSEE as part of the MISSE 7 mission. And, finally, we would like to thank Don Jaworske (retired) of NASA Glenn for coordinating Glenn's MISSE 7 experiments, and also for experiment delivery, integration and deintegration.

Trade names and trademarks are used in this report for identification only. Their usage does not constitute an official endorsement, either expressed or implied, by the National Aeronautics and Space Administration.

Level of Review: This material has been technically reviewed by technical management.

Available from

NASA STI Program
Mail Stop 148
NASA Langley Research Center
Hampton, VA 23681-2199

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
703-605-6000

This report is available in electronic form at <http://www.sti.nasa.gov/> and <http://ntrs.nasa.gov/>

Bend-Test Results of the MISSE 7 Flexural Stress Effects Experiment After 1.5 Years of Space Exposure

Kate E. Snow

Hathaway Brown School
Shaker Heights, Ohio 44122

Kim K. de Groh

National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Bruce A. Banks

Science Application International Corporation
Cleveland, Ohio 44135

Edward A. Seckar

ZIN Technologies, Inc.
Middleburg Heights, Ohio 44130

Abstract

Low Earth orbit (LEO) environmental exposure can cause degradation of exterior spacecraft materials. Radiation and thermal exposure often result in bond-breaking and embrittlement of polymers, reducing mechanical strength and structural integrity. The Flexural Stress Effects Experiment (FSEE) was flown with the objective of determining the role of space exposure on the degradation of polymers under flexural stress. The FSEE samples were flown in a wake orientation on the exterior of International Space Station for 1.5 years. Twenty-three polyimide and fluorinated polymers with various coatings were flown: 11 bent over a 0.375-inch diameter holder and 12 over a 0.25-inch diameter holder. A non-standard bend-test procedure was used to determine the surface strain at which embrittled polymers crack. None of the control samples cracked, even under surface strains up to 19.7%, although one coating cracked. Of the 10 flight samples tested, seven indicated increased embrittlement through bend-test cracking at surface strains from 0.65% to 8.11%. Therefore, most of the tested polymers were embrittled due to space exposure, when compared to their control samples. The samples flown over the 0.375-inch holder were more embrittled than those on the 0.25-inch holder. Determination of the extent of space induced embrittlement of polymers is important for designing durable spacecraft.

Introduction

Materials on the exterior of spacecraft are exposed to many environmental threats that can be harmful to spacecraft and their operations. These threats include solar radiation (ultraviolet (UV), x-rays), charged particle radiation (electrons, protons), thermal cycling (hot and cold cycles), micrometeoroid impacts, debris impacts (space particles), and atomic oxygen (AO). Many of these environmental exposures can weaken polymers and affect their mechanical and structural properties (Ref. 1). An example of space induced materials degradation can be seen on the Hubble Space Telescope (HST) (Ref. 1). When sent up into space, the HST was covered with multilayer insulation blankets comprised of a normally stretchy outer layer of aluminized Teflon® fluorinated ethylene propylene (FEP). However after being exposed to solar radiation, charged particle radiation, and thermal cycling the FEP on the HST became brittle causing it to crack after being exposed to the space environment for only 5.8 years as shown in Figure 1. To prevent this



Figure 1. Example of cracking of the aluminized-Teflon FEP insulation on the HST after 5.8 years of space exposure (Ref. 2).

type of damage from occurring in the future, the space community has been sending flight experiments with polymer samples into space to gain information on how space exposure affects the materials prior to using them for spacecraft applications. Many of the flight experiments have been flown as part of the Materials International Space Station Experiment (MISSE) missions. An experiment called the Flexural Stress Effects Experiment (FSEE) was flown on the MISSE 7 mission with the objective of determining the role of space exposure on the degradation of polymers under flexural stress.

Materials International Space Station Experiment

MISSE is a series of materials flight experiments consisting of suitcase like trays called Passive Experiment Containers (PECs) which were exposed to the space environment on the exterior of the International Space Station (ISS). A total of 10 MISSE PECs, as part of six MISSE missions have been flown between 2001 and 2013. Figure 2 shows the MISSE 7A and 7B PECs on the Express Logistics Carrier-2 (ELC-2). Future MISSE missions will be flown on the new ISS external MISSE-Flight Facility (MISSE-FF) starting in 2018. The FSEE was flown as part of the MISSE 7 mission to determine the role of space exposure on the degradation of polymers under flexural stress.

Flight Orientations

A diagram showing ram, wake, zenith, and nadir directions on the ISS is shown in Figure 3. The flight orientation highly affects the environmental exposure. Ram facing experiments receive a high flux of directed AO and sweeping (moderate) solar exposure. Zenith facing experiments receive a low flux of grazing arrival AO and the highest solar exposure. Wake experiments receive very low AO flux and moderate solar radiation levels similar to ram experiments. Nadir experiments receive a low flux of grazing arrival AO and minimal solar radiation (albedo sunlight). All surfaces receive charged particle and cosmic radiation, which are omni-directional. It should be noted that the actual orientation of the ISS varies due to operational requirements with the majority of time spent within ± 15 degrees of the +XVV Z nadir flight attitude (X Axis Near Velocity Vector, Z Axis Nadir/Down). Deviations from this attitude to accommodate visiting spacecraft, and other ISS operational needs, can cause variations in the orientation directions, and hence variations in environmental exposures.

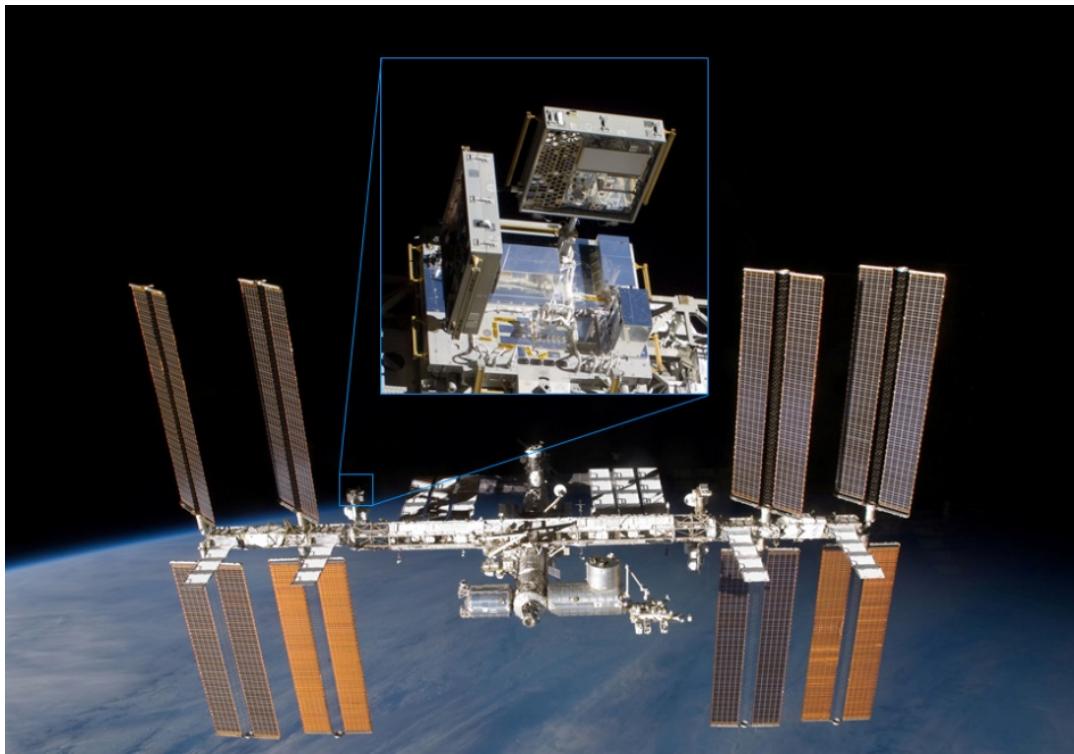


Figure 2. The MISSE 7 PECs on the ISS are visible inside the highlighted box. In the close-up image, which is rotated with respect to the ISS flight orientation, the MISSE 7A zenith/nadir PEC is on the right with the nadir surface visible and the MISSE 7B ram/wake PEC is on the left with the ram surface facing to the left.

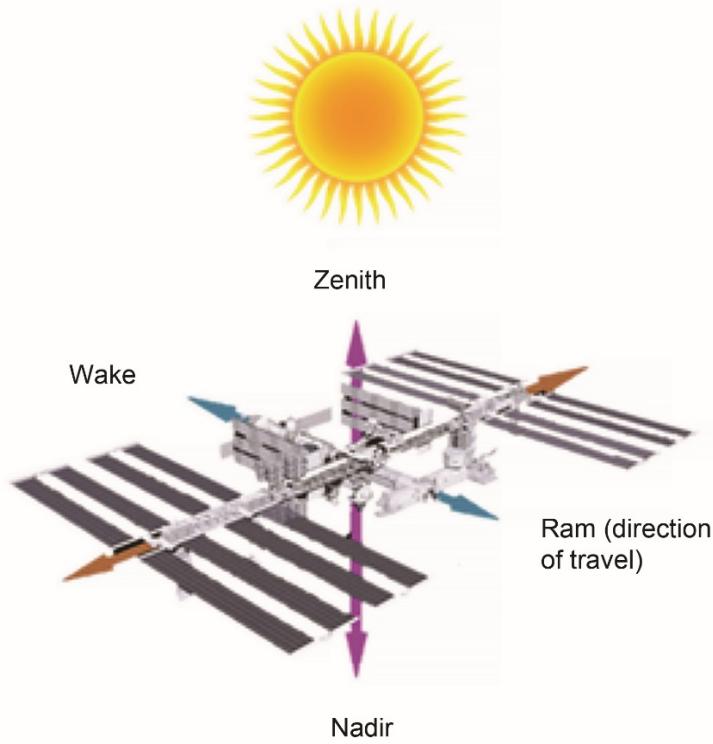


Figure 3. Diagram depicting the different orientations relative to the ISS.

Flexural Stress Effects Experiment (FSEE)

The FSEE was flown in a wake orientation on the ISS from November 23, 2009 to May 20, 2011 for 1.49 years as a part of the MISSE 7 mission in order to study the role of surface flexural stress on space environment induced polymer degradation. The flight samples were flown in a wake orientation because the experiment was intended to test the effects of UV radiation with as little AO exposure as possible. Twenty-three rectangular samples were flown: 11 were flown bent over a 0.375-inch sample holder and 12 were bent over a 0.25-inch sample holder. The samples were bent over the sample holders without applying any tensile stress. They consisted of nine assorted polyimide and fluorinated polymers with assorted coatings. A set of drawings of one of the FSEE holders showing the rectangular samples and how they are attached is shown in Figure 4. Figure 5 is a pre-flight photograph of the two FSEE holders with the flight samples loaded and ready for flight. Figure 6 provides a pre-flight photo of the MISSE 7B wake tray, with the location of the FSEE samples. Half the samples were designated for post-flight bend-testing and the other half were designated for post-flight tensile testing. This paper reports the bend-testing results.

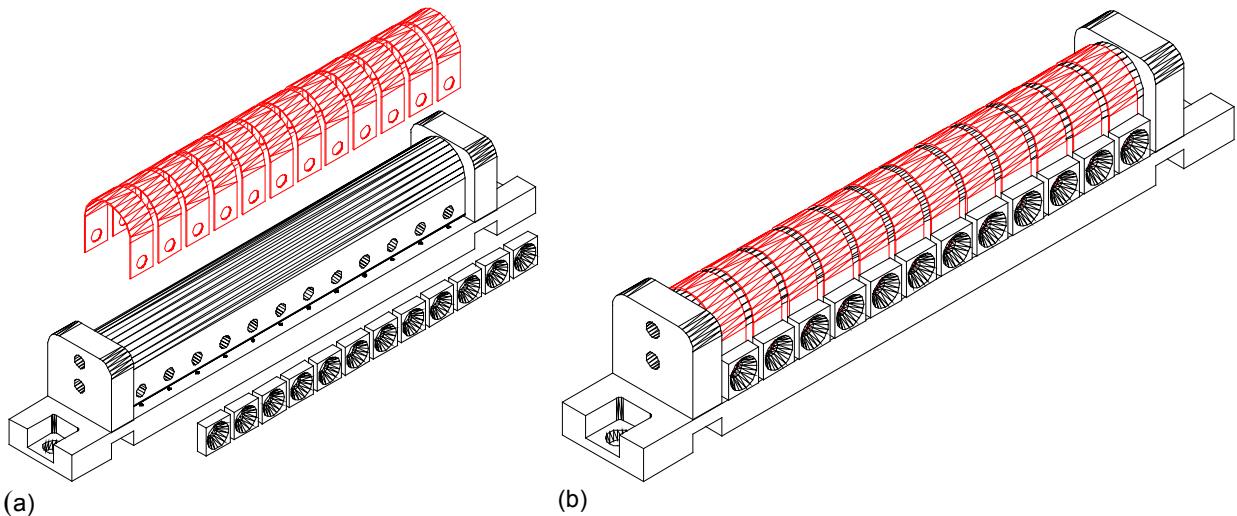
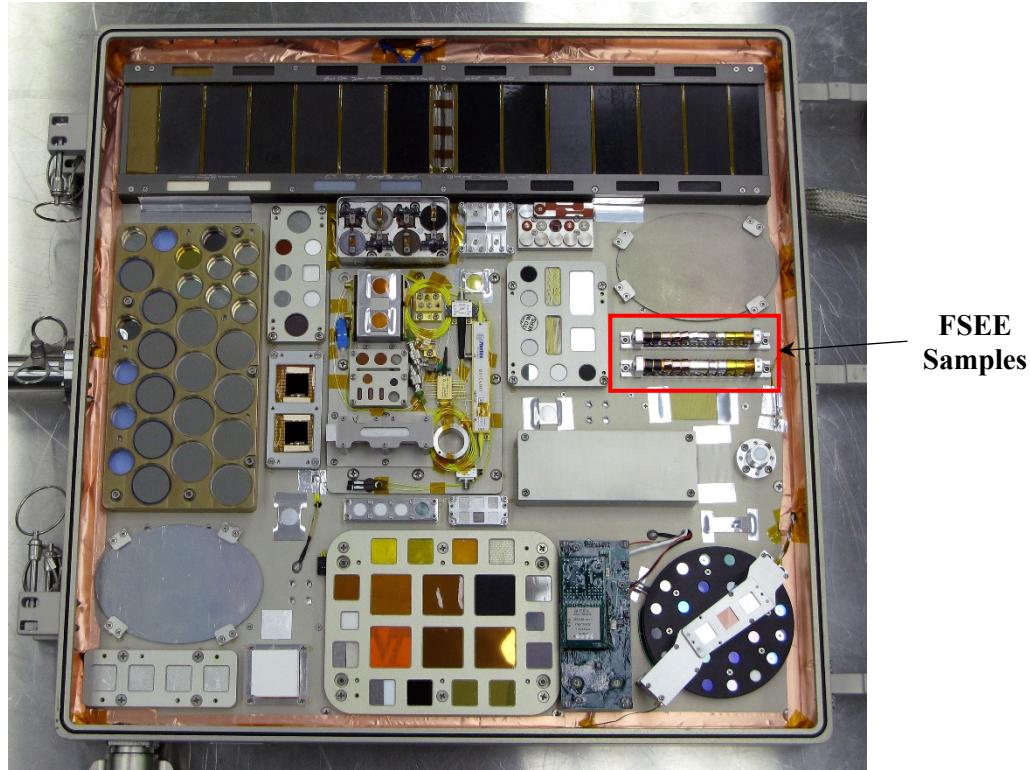


Figure 4. Graphics of a FSEE holder with 12 samples: (a) Exploded view, and (b) View with samples mounted on the holder.



Figure 5. Pre-flight photograph of the two FSEE holders with the flight samples.



*Figure 6. Pre-flight photo showing the location of the FSEE sample holders on the MISSE 7B wake tray.
(Photo credit: Naval Research Laboratory)*

Materials and Methods

The materials for this flight experiment are listed in Table 1. The materials were chosen for one of two reasons: the materials were either commonly used spacecraft materials, or there was a need to test a specific material for a flight project. For example, the Kapton XC and Si/Kapton E/Al/Inconel/Al were being considered for the James Webb Space Telescope. Materials such as FEP/Al and Kapton HN/Al are spacecraft thermal control materials, which have been flown on many spacecraft in the past, including the HST. Coated DC-93-500 silicone had been flown previously as well, but never with the intent of studying the effects of radiation embrittlement. It should be noted that although two stretched lens array (SLA) multi-layer oxide coated DC-93-500 samples were planned for flight (A-12 and B-12), only the 0.25-in diameter holder sample (A-12) was flown. B-12 was not flown. Finally, CP1/Al was flown for assessment as a potential solar sail material. Samples A-1, A-3, A-7, A-9, A-10, B-1, B-3, B-7, B-9 and B-10 were designated for bend-testing. For every set of flight samples (i.e., A-1 and B-1) there is a corresponding control sample of the same materials and thickness (i.e., C-1). The control samples are not listed in Table 1. The remaining samples will be tensile tested at a later time. It should be noted that the Al coatings are also designated as vapor deposited aluminum (VDA) coatings.

Photo Documentation

Photographs were taken of the samples upon their return to Earth using a Sony DSC T-7 camera. First, photos were taken while the samples were still on their flight holders. Then, the samples were removed from their holders and laid flat using alligator clips and photographed next to their respective control samples.

Table 1. Flexural Stress Effects Experiment Flight Samples.

A-Holder (0.25-inch dia), Sample Length: 1.412 in.				B-Holder (0.375-inch dia), Sample Length: 1.483 in.			
ID	MISSE ID	Material	Thickness (mils)	ID	MISSE ID	Material	Thickness (mils)
A-1	N7-W-A1	Kapton XC/Al	1	B-1	N7-W-B1	Kapton XC/Al	1
A-2	N7-W-A2	Kapton XC	1	B-2	N7-W-B2	Kapton XC	1
A-3	N7-W-A3	Si/Kapton E/ Al/Inconel/Al	2	B-3	N7-W-B3	Si/Kapton E/ Al/Inconel/Al	2
A-4	N7-W-A4	Si/Kapton E/ Al/Inconel/Al	2	B-4	N7-W-B4	Si/Kapton E/ Al/Inconel/Al	2
A-5	N7-W-A5	Si/Kapton E/ Al/Inconel/Al	2	B-5	N7-W-B5	Si/Kapton E/ Al/Inconel/Al	2
A-6	N7-W-A6	Al/CP1/Al	1	B-6	N7-W-B6	Al/CP1/Al	1
A-7	N7-W-A7	CP1/Al	1	B-7	N7-W-B7	CP1/Al	1
A-8	N7-W-A8	FEP/Al	2	B-8	N7-W-B8	FEP/Al	2
A-9	N7-W-A9	FEP/Al	5	B-9	N7-W-B9	FEP/Al	5
A-10	N7-W-A10	Kapton HN/Al	1	B-10	N7-W-B10	Kapton HN/Al	1
A-11	N7-W-A11	SiOx/Kapton HN/ SiOx/Al	1	B-11	N7-W-B11	SiOx/Kapton HN/ SiOx/Al	1
A-12	N7-W-A12	SLA/DC 93-500	8	B-12	N7-W-B12	SLA/DC 93-500 (not flown)	8

CP1: Clear polyimide

FEP: Fluorinated ethylene propylene

SLA: Stretched lens array (SLA) multi-layer oxide coating

Note: Samples in the shaded rows were bend-tested, those in the white rows will be tensile tested.

Bend Testing

Typically, ASTM D790 Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials is used for bend-test procedures of polymers. However, the ASTM technique is used to measure the stress and strain of a polymer at a particular bend radius (Ref. 3), which was not the objective of the FSEE experiment. Therefore, a non-standard test was designed that allows the strain at which an embrittled polymer's surface cracks while under tensile loading to be determined (Ref. 4). This procedure was used to test the embrittlement of MISSE 5 polymers flown in a nadir flight orientation for 13 months (Ref. 5).

Testing was conducted on a semi-suspended pliable surface held up with two supports, as shown in Figure 7. Successively smaller mandrels (cylindrical steel pieces) were used to apply surface strain to samples. The materials were rested, with the space-exposed surface facing down, upon the semi-suspended surface and the mandrels were used to press the samples down, causing the bend-test pliable surface and the flight sample to wrap around the mandrel, as shown in Figure 8 and as illustrated in Figure 9. This put the space exposed surface of the polymer in tension and induced cracking in the embrittled samples without applying a net tensile load to the sample. A total of 23 mandrels were used and ranged in size from 1.253 cm (0.493 in) to 0.052 cm (0.020 in). A decrease in diameter resulted in an increase in strain as indicated in Equation (1). The strain values ranged from 0.202% to 4.678% in 1 mil samples, 0.404% to 8.939% in 2 mil samples and 1.003% to 19.704% in 5 mil samples.

$$E = \left(\frac{t}{d+t} \right) \times 100 \quad (1)$$

Where:

E = % surface strain

t = film thickness (cm)

d = mandrel diameter (cm)



Figure 7. The bend-test mechanism in its idle state; the polymer being tested rests atop the Kapton stretched across the center, with the space exposed surface face down.



Figure 8. The bend-test mechanism in its testing state; the polymer being tested wraps around the circumference of the mandrel with the space exposed surface in tension.

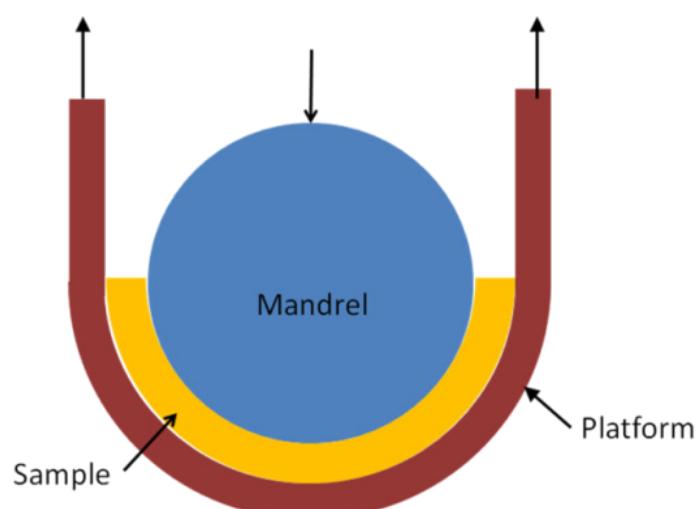


Figure 9. Illustration of the bend-test configuration showing a cradle platform used to bend the sample around the mandrel (Ref. 5).

After each individual bend procedure, the polymers were examined under a microscope to determine whether any cracking had occurred. Cracking was expected to occur perpendicularly to the sample given the way they were being tested. Crack-like markings running in other directions typically were later determined to just be scratches. Either an Olympus stereo zoom SMZ optical microscope equipped with a Cannon EOS D30 digital camera or an Olympus SZH OM equipped with a Leica DFC29S camera was used to photograph the samples. In addition to testing space-exposed polymers, five non-space exposed control polymers were tested as a point of comparison.

Results and Discussion

MISSE 7B Environmental Exposure

As mentioned previously, the FSEE was exposed to the LEO wake space environment on the exterior of ISS for 1.49 years. Estimates of solar exposure in equivalent sun hours (ESH) have been determined for the MISSE 7 surfaces (Ref. 5). The MISSE 7B wake surface received an estimated 2,000 ESH based on UV diode measurements on readings from the wake tray, monitored by the Lead-Free Technology Experiment in Space Environment (Ref. 6). The AO fluence for the MISSE 7B wake tray was determined to be $2.9 \pm 0.3 \times 10^{20}$ atoms/cm² as reported by Finckenor (Ref. 6). This fluence is many orders of magnitude higher than would be expected for “true” wake exposure. This is because the samples received ram AO exposure for short durations during ISS flight attitude changes.

Post-flight Observations

Figure 10 is a post-flight photo of the MISSE 7B wake tray. Figure 11 shows a post-flight photo of the FSEE sample holders. Figure 12 shows a top view of the FSEE samples on their flight holders before (left) and after (right) space exposure along with a material legend. In Figure 12, the right side of the samples was zenith facing, while the left side was nadir facing. As mentioned previously, the samples were separated into two groups for post-flight testing, one which would be bend-tested and another which would be tensile tested. This way as much data as possible could be collected. Additionally, the SLA/DC 93-500 sample, shown in Figure 13, will be undergoing a different kind of testing, as the sample appeared cracked post-flight, and cannot be bend-tested as it is already cracked. Figure 14 shows the cracks in the SLA/DC-93-500 sample. In addition, DC-93-500, the substrate, is a silicone which when exposed to the space environment converts to a silicate and shrinks and cracks. Mud tile-like cracking of AO exposed DC-93-500 silicone has been previously reported (Ref. 7).

Atomic Oxygen Exposure

The samples were flown in a wake orientation with the intent of exposing them to minimal AO. Upon observation, however, it was noticed that some of the samples were discolored in some areas, as shown in Figures 15-25 (the samples are held flat with alligator clips in Figures 15-25). It was concluded that this discoloring was a result of AO erosion. The ISS flight orientation was rotated during a part of the mission causing the samples to be flown temporarily in a ram orientation, exposing them to significantly more AO than anticipated. As mentioned in the MISSE 7B Environment Exposure section, the MISSE 7B wake surface was exposed to an AO fluence of 2.9×10^{20} atoms/cm² (Ref. 6).



Figure 10. Post-flight photo of the MISSE 7B wake tray.

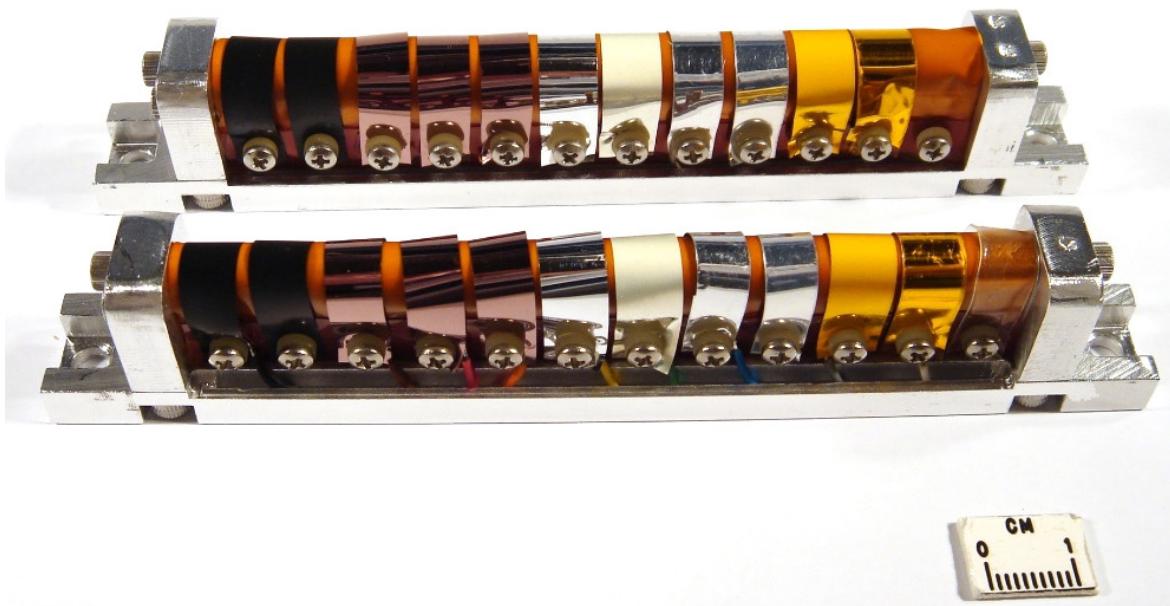
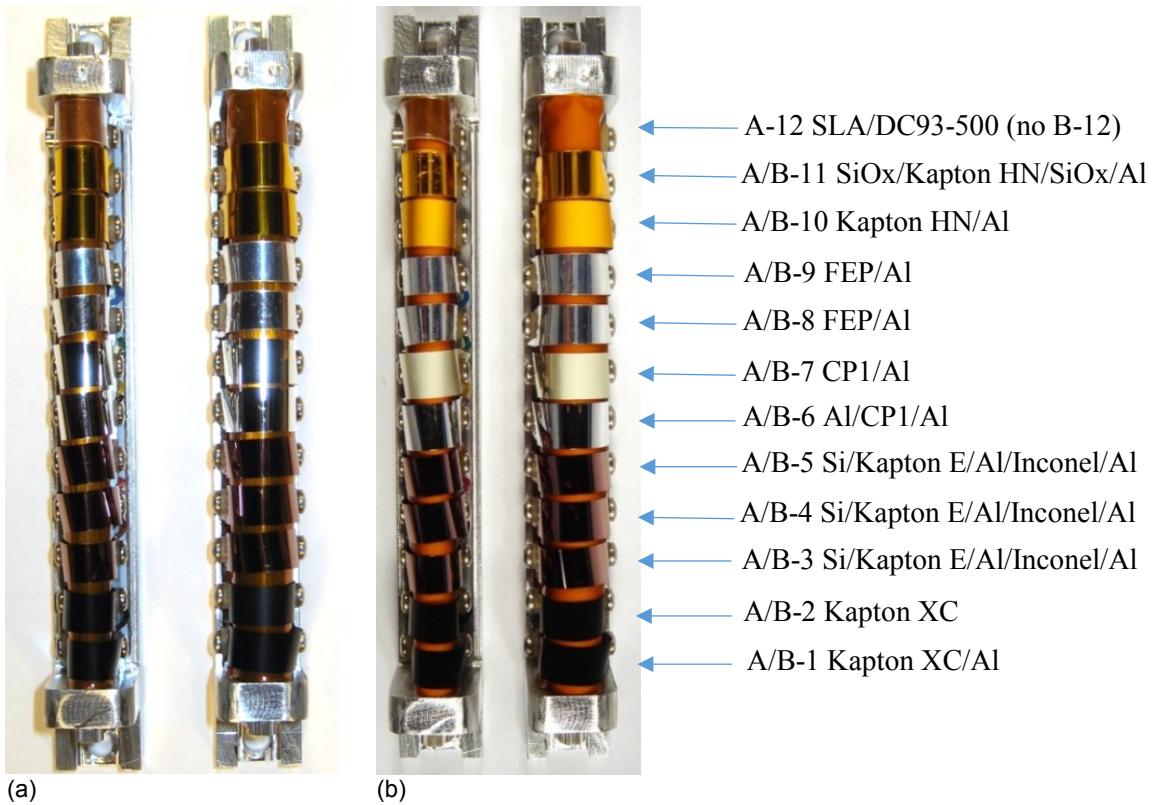


Figure 11. Post-flight photo of the sample holders.



*Figure 12. Top view of the sample holders before, and after, being flown on the ISS for 1.49 years:
 (a). Pre-flight, and (b). Post-flight. The holder with one dot on top is 0.25-inch mandrel (left),
 holder with two dots is 0.375-inch mandrel (right).*

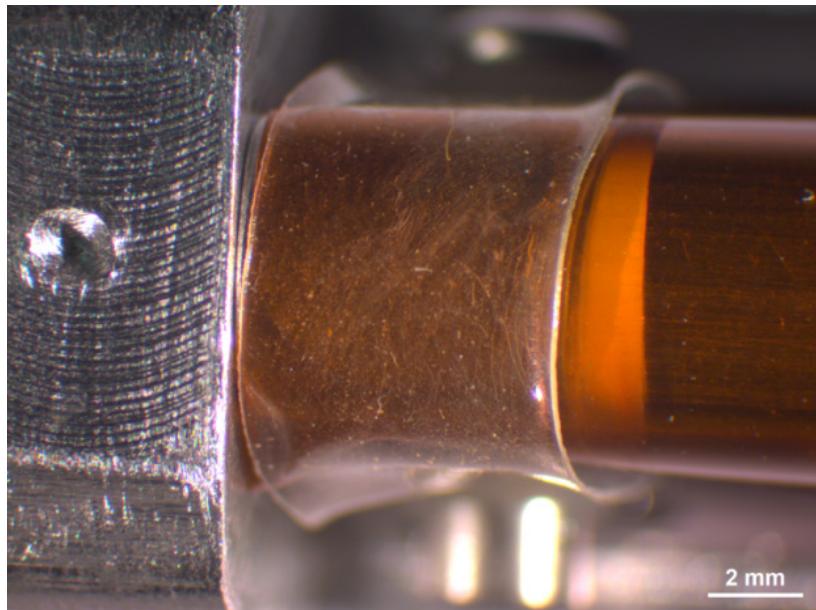
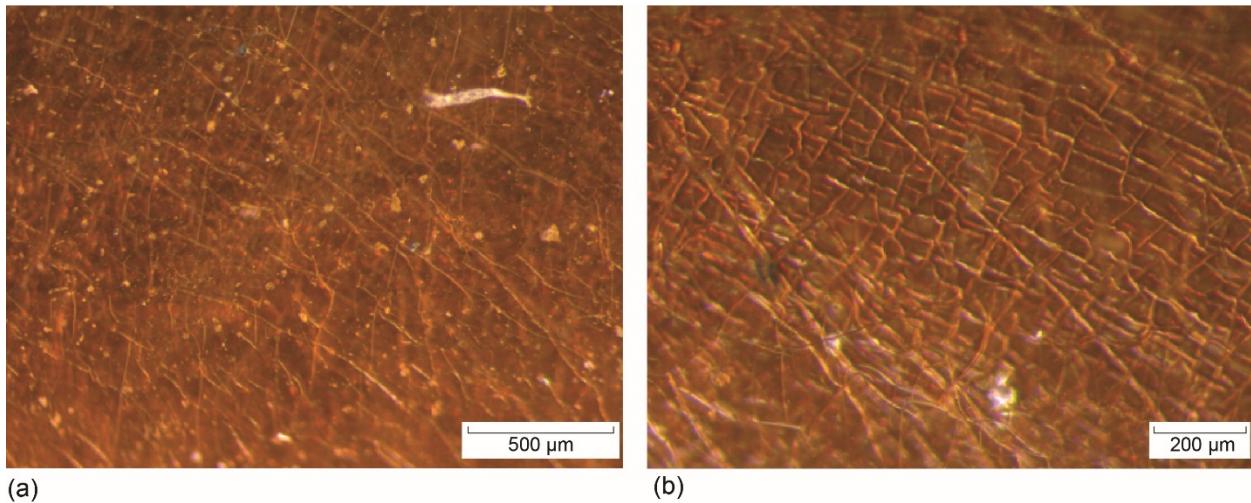


Figure 13. SLA/DC93-500 on its holder after being exposed to the space environment.



(a)

(b)

Figure 14. SLA/DC93-500 on its holder after being exposed to the space environment (a). Cracking that looks similar to mud-tiles is visible (b).

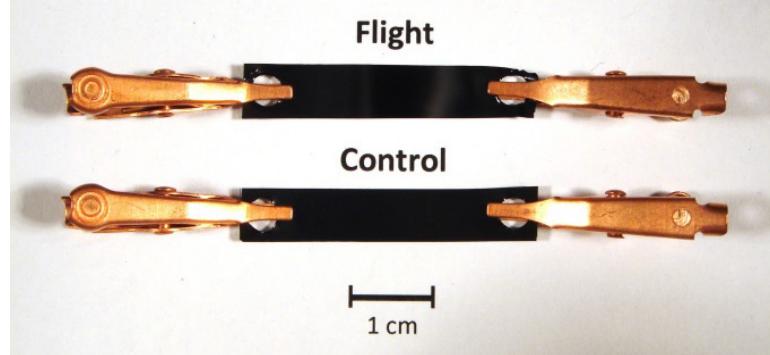


Figure 15. Post-flight photograph of Kapton XC/Al (A-1) flight and control samples. Atomic oxygen erosion lightening can faintly be seen in the flight sample.

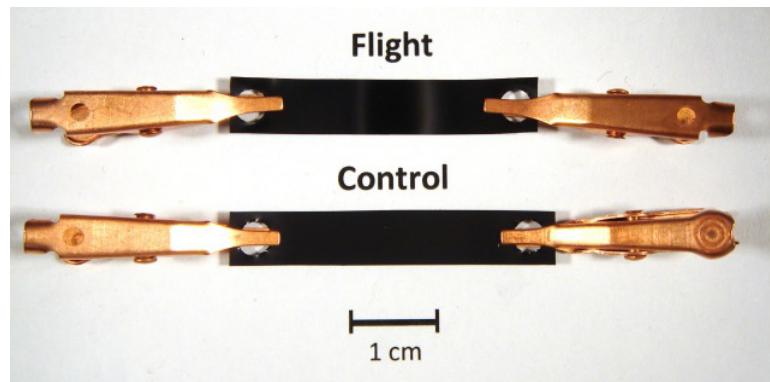


Figure 16. Post-flight photograph of Kapton XC (A-2) flight and control samples. Atomic oxygen erosion lightening can faintly be seen in the flight sample.

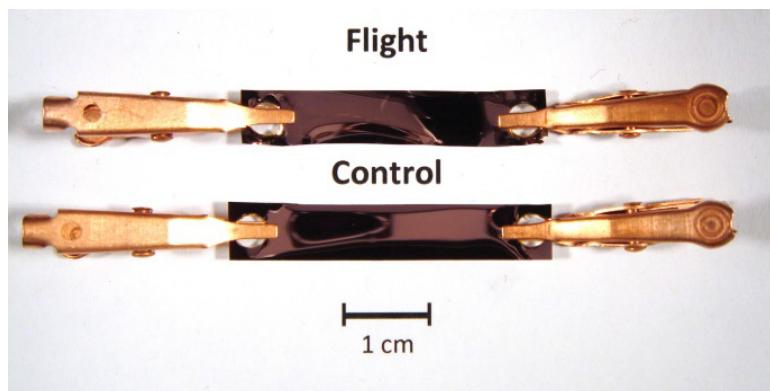


Figure 17. Post-flight photograph of Si/Kapton E/Al/Inconel/Al (A-3) flight and control samples. Atomic oxygen erosion lightening cannot be seen in the flight sample.

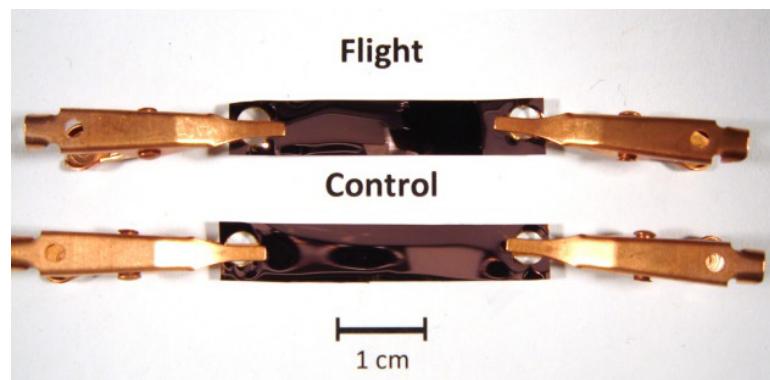


Figure 18. Post-flight photograph of Si/Kapton E/Al/Inconel/Al (A-4) flight and control samples. Atomic oxygen erosion lightening cannot be seen in the flight sample.

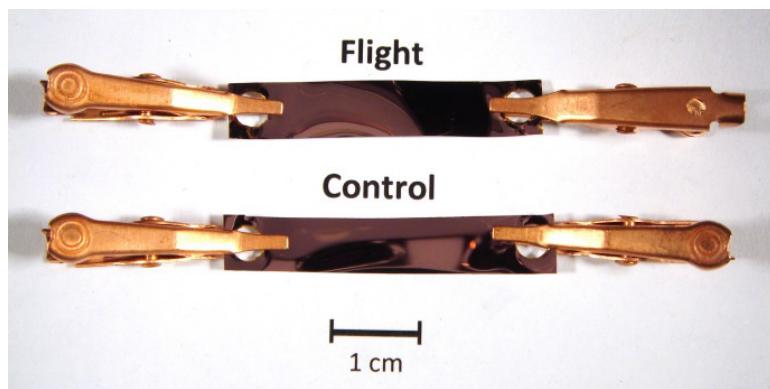


Figure 19. Post-flight photograph of Si/Kapton E/Al/Inconel/Al (A-5) flight and control samples. Atomic oxygen erosion lightening cannot be seen in the flight sample.

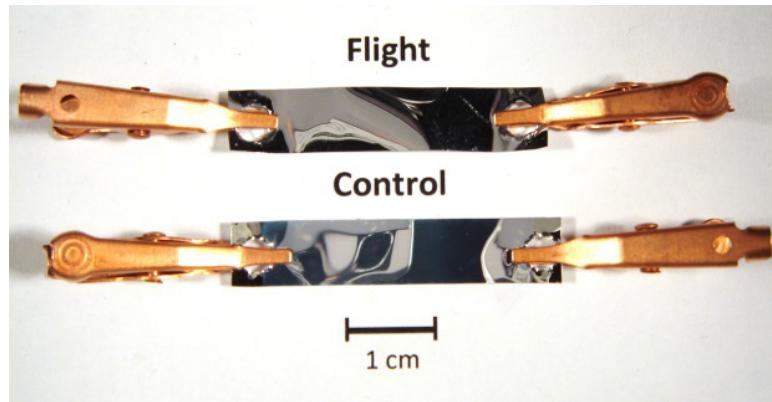


Figure 20. Post-flight photograph of Al/CPI/Al (A-6) flight and control samples. Atomic oxygen erosion lightening cannot be seen in the flight sample.

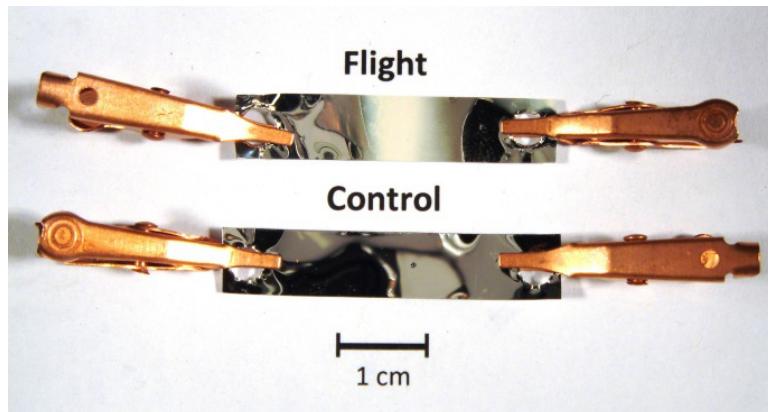


Figure 21. Post-flight photograph of CP1/Al (A-7) flight and control samples. Atomic oxygen erosion lightening can be seen in the flight sample.

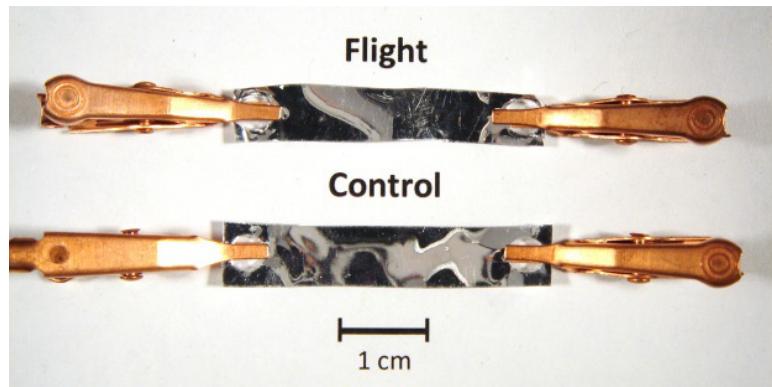


Figure 22. Post-flight photograph of FEP/Al (A-8) flight and control samples. Atomic oxygen erosion lightening cannot be seen in the flight sample.

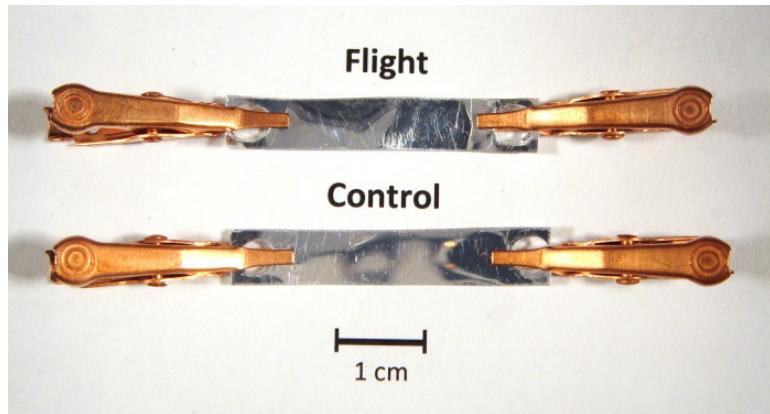


Figure 23. Post-flight photograph of FEP/Al (A-9) flight and control samples. Atomic oxygen erosion lightening cannot be seen in the flight sample.

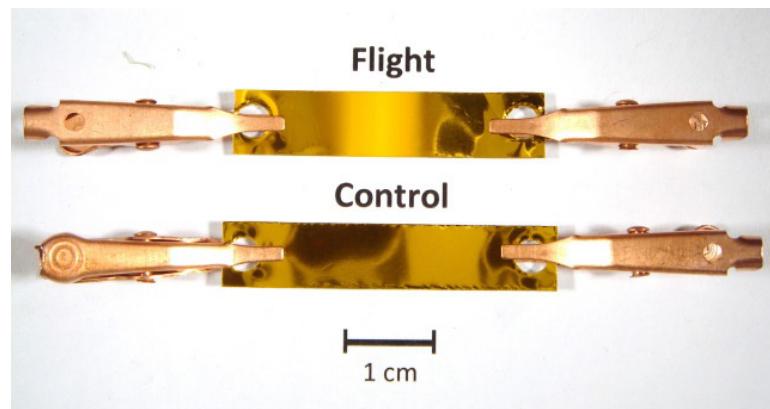


Figure 24. Post-flight photograph of Kapton HN/Al (A-10) flight and control samples. Atomic oxygen erosion lightening can be seen in the flight sample.

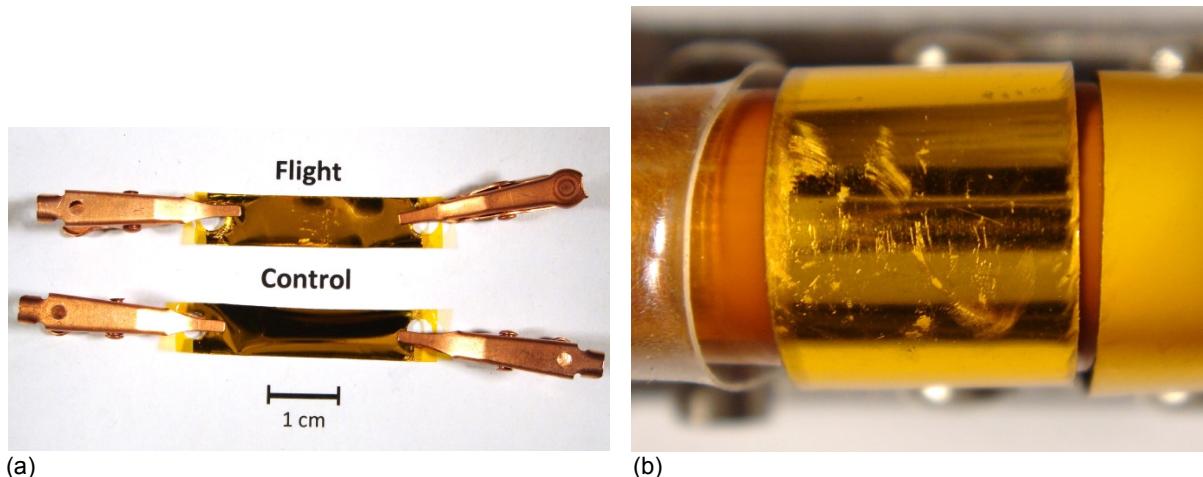


Figure 25. SiO_x/Kapton HN/SiO_x/Al (A-11): (a) Post-flight photograph of flight and control samples, and (b) Flight sample on holder showing cracks and coating damage.

Bend-Test Results

The bend-test results for the FSEE flight and control samples are provided in Table 2. The materials cracked at a variety of surface strains ranging from 0.65% to 8.11% whereas their control counterparts didn't crack at strains of up to the maximum strain of 19.7%. The one exception was the control sample C-3 (Si/Kapton E/Al/Inconel/Al), which displayed cracking in its Si coating but not in the polyimide Kapton layer. Microscopy images of what the samples looked like before and after undergoing bend-testing to a variety of strains can be seen in Figures 26-44. The microscope magnification is provided in terms of both zoom and optical lens magnification. For example, 7.5z 2x means the 2x optical lens was used and then zoomed to a magnification of 7.5.

When testing the polymers, it is fairly common that the mandrels will cause some minor surface markings on the samples, especially those that are softer materials. These scratches and markings can be mistaken for cracking and lead to error. Additionally, since the samples were flown while bent over a mandrel, when they were taken off their holders, many of them had a tendency to maintain their curved position. This meant that the samples had to be flattened in order to effectively photograph and observe them with the microscope. To do this, microscope slides were used which may have led to additional scratches on the samples. It is important to make sure scratches are not mistaken for cracks, as they can often be difficult to tell apart. Figure 33 provides an example of this as there are many visible surface scratches amidst the polymer cracks. After the bend-testing concluded, the samples were all tested for a second round of bend-testing for added accuracy. This is why a percent strain range is provided for each sample.

Table 2. Bend-Test Results for the FSEE Flight and Control Samples.

Sample ID	Flight Holder Diameter (in)	Material	Thickness (mils)	Mandrel Diameter* (in)	% Strain Range
A-1	0.25	Kapton XC/Al	1	Did Not Crack (DNC)	
A-3		Si/Kapton E/Al/Inconel/Al	2	0.071-0.209	0.95-2.75%
A-7		CP1/Al	1	0.107-0.153	0.65-0.93%
A-9		FEP/Al	5	0.057-0.113	4.24-8.11%
A-10		Kapton HN/Al	1	DNC	
B-1	0.375	Kapton XC/Al	1	DNC	
B-3		Si/Kapton E/Al/Inconel/Al	2	0.107-0.132	1.49-1.84%
B-7		CP1/Al	1	0.038-0.143	0.70-2.59%
B-9		FEP/Al	5	0.209-0.355	1.39-2.34%
B-10		Kapton HN/Al	1	0.021 & DNC	4.49% & DNC

*Mandrel diameter where sample cracking was first observed

Kapton XC/Al

Flight samples A-1, B-1 and control sample C-1 were composed of 1 mil thick Kapton XC, a black Kapton material, with an aluminized backside coating. Due to the samples being black, it was difficult at times to determine whether the markings on the sample were due to scratching or cracking. The sample appeared to have AO texturing. Bend testing the sample seemed to damage the matte surface as indicated by increased texture visible in Figure 28(b). It was determined that neither of the flight samples nor the control sample exhibited any signs of cracking. Ultimately, this was the only material to not experience cracking on any mandrel during both rounds of testing as seen in Figures 26-30. Thus, the back-surface aluminized black Kapton XC was the least embrittled after exposure to the space environment.

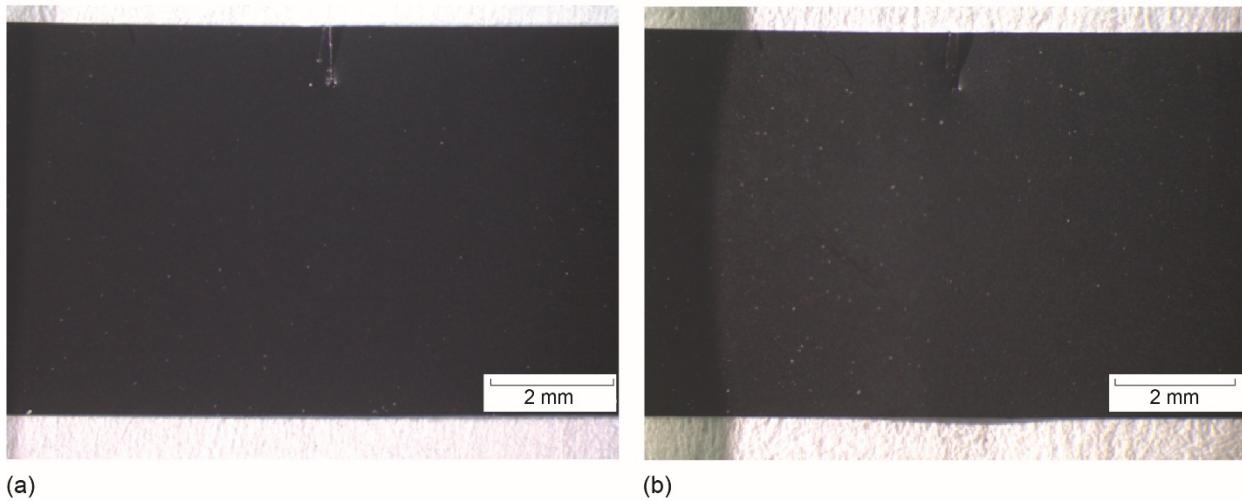


Figure 26. Control sample C-1 (Kapton XC/Al) at 7.5z 2x: (a) before undergoing testing and (b) after undergoing 4.68% surface strain, the sample did not crack.

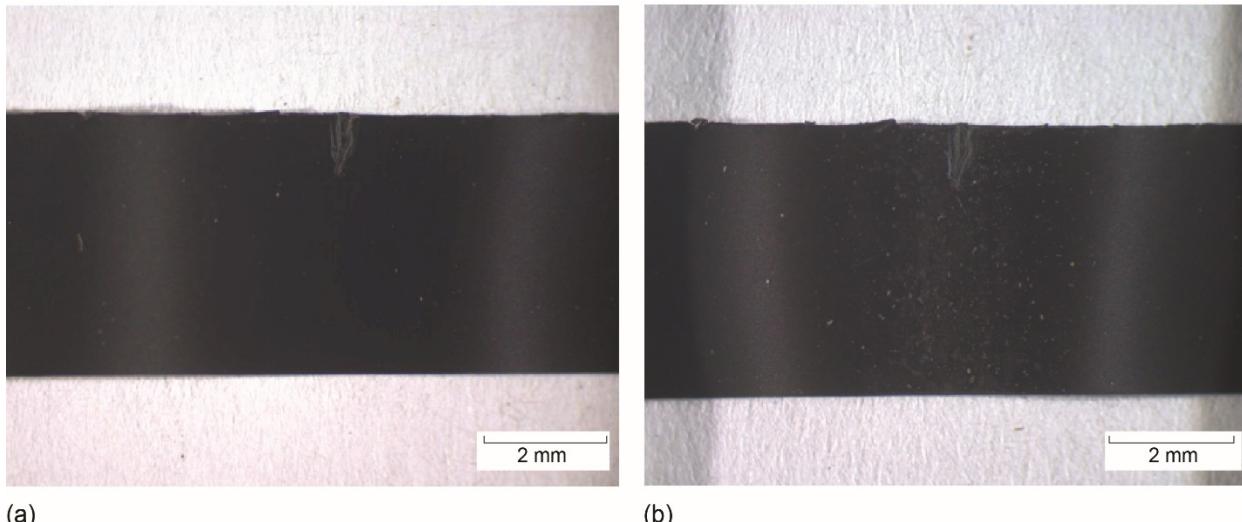


Figure 27. Flight sample A-1 (Kapton XC/Al) at 7.5z 2x: (a) before undergoing testing, and (b) after undergoing 3.69% surface strain, the sample did not crack; initial round of testing.

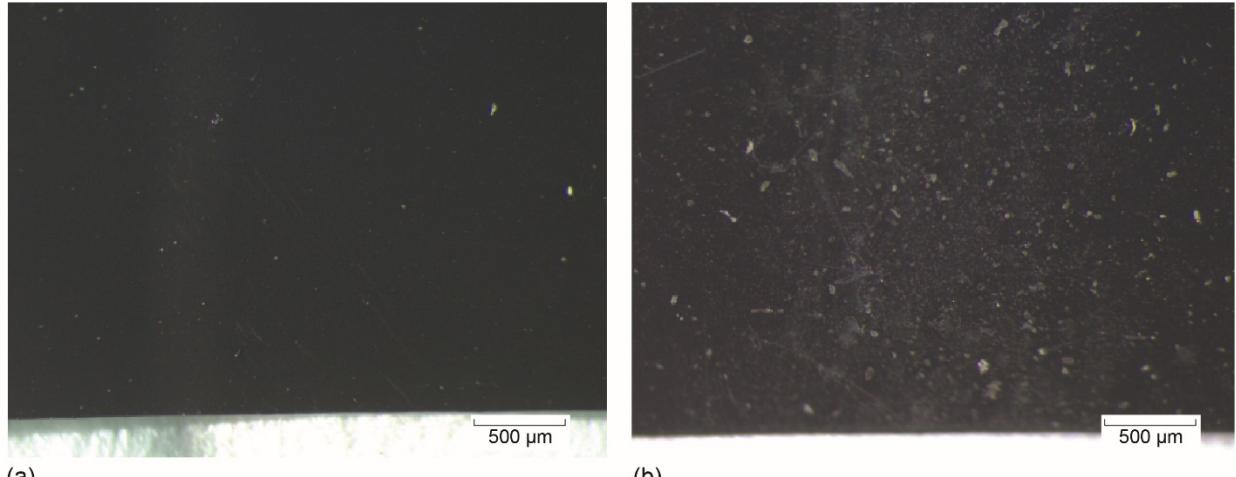


Figure 28. Flight sample A-1 (Kapton XC/Al) at 25z 2x: (a) before undergoing testing and (b) after undergoing 3.69% surface strain, the sample did not crack; initial round of testing.

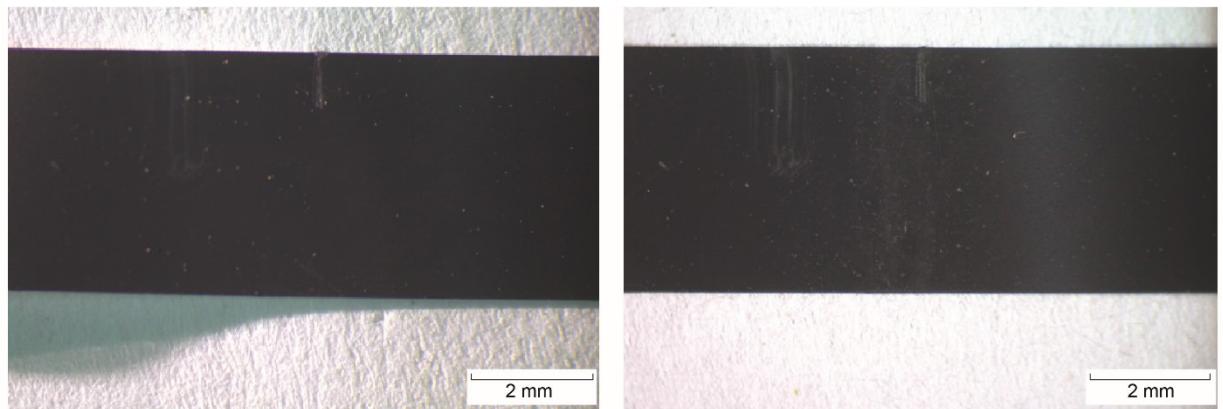


Figure 29. Flight sample B-1 (Kapton XC/Al) 7.5z 2x: (a) before undergoing testing and (b) after undergoing 3.69% surface strain, the sample did not crack; initial round of testing.

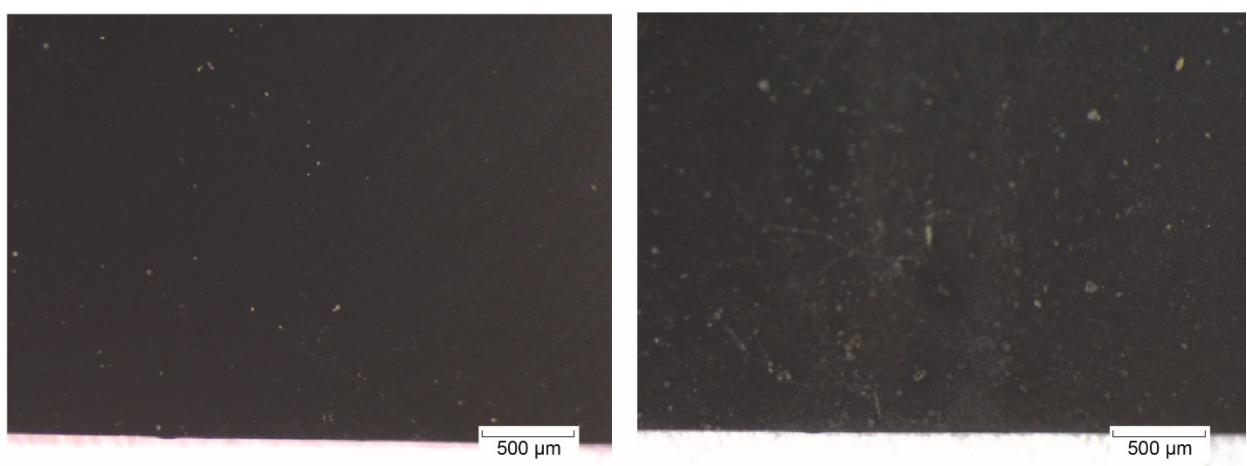


Figure 30. Flight sample B-1 (Kapton XC/Al) 25z 2x: (a) before undergoing testing and (b) after undergoing 3.69% surface strain, the sample did not crack; initial round of testing.

Si/Kapton E/Al/Inconel/Al

Flight samples A-3, B-3 and control sample C-3 were composed of 2 mil thick Kapton E with an Si front surface coating and double aluminized Inconel back surface coatings. At times it was difficult to determine whether the markings on the sample were cracks in the Kapton E or cracks in the coating. Extra caution was taken to ensure that any noticeable cracking was in the Kapton E itself, not just the Si coating. Ultimately, it was determined that this was the second most embrittled material of all those tested, cracking at low amounts of strain in both the 0.25-inch holder as well as the 0.375-inch holder as seen in Figures 31-33. In Figures 16 and 20, there are visible lines running perpendicular to the control sample. These lines are a result of cracking in the Al/Inconel/Al layer, making the Si/Kapton E/Al/Inconel/Al samples to be the only material to experience cracking of any kind in the control samples.

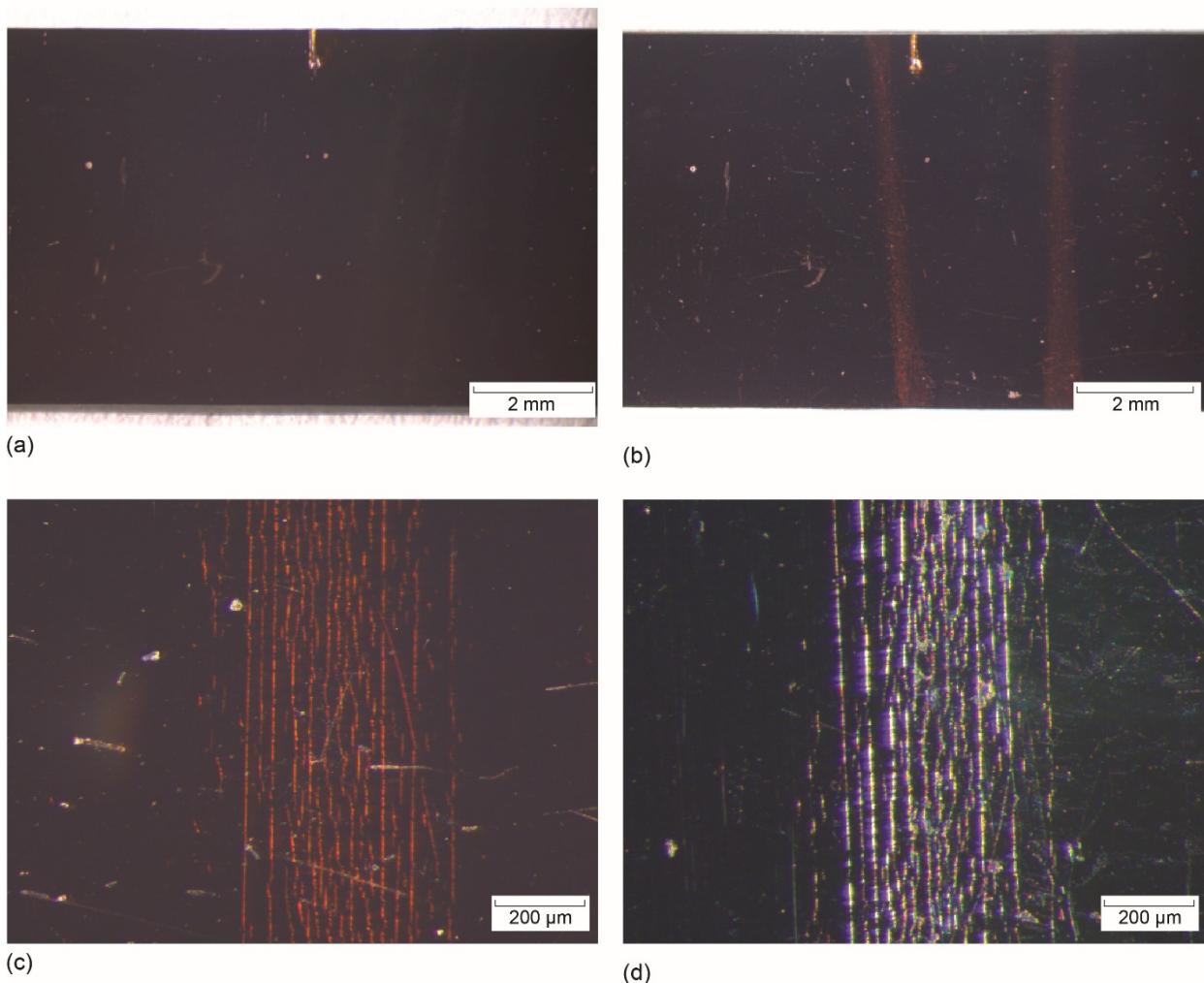
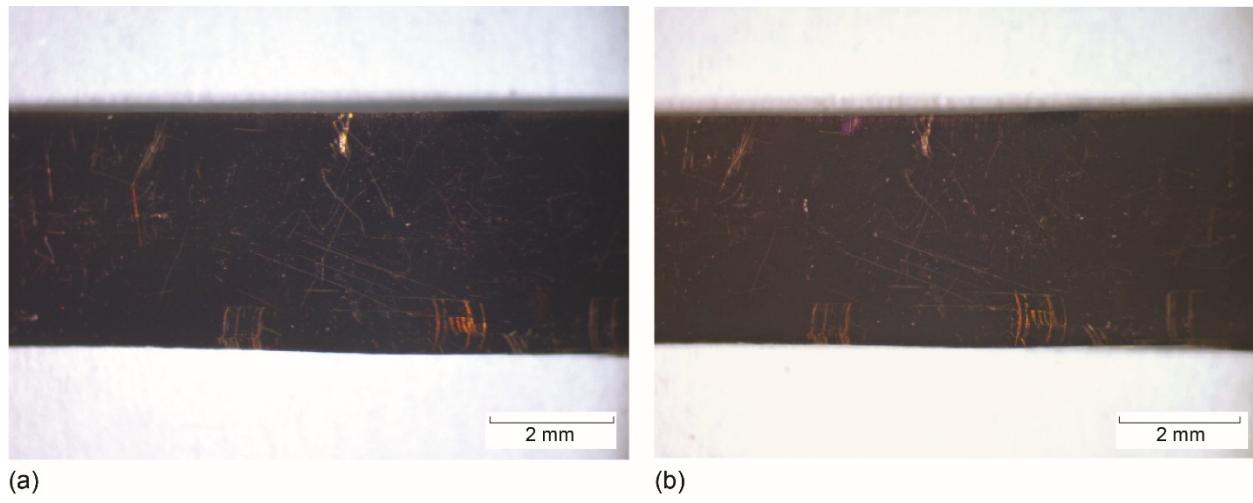


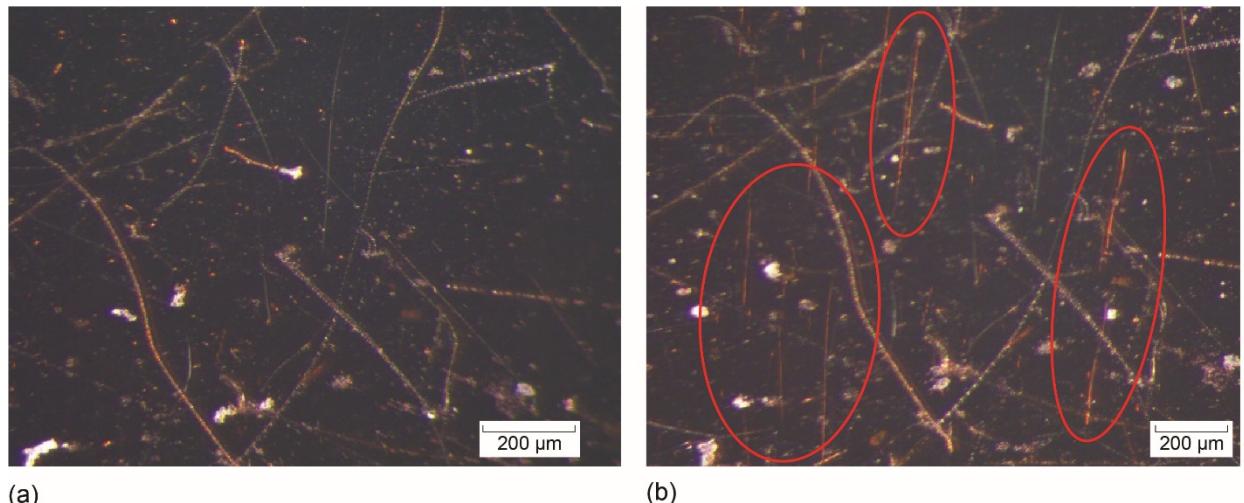
Figure 31. Control sample C-3 (Si/Kapton E/Al/Inconel/Al) 7.5z 2x: (a) before undergoing testing (7.5z 2x), (b) after 8.94% surface strain (7.5z 2x), (c) after 8.94% surface strain (64z 2X) showing cracks, and (d) after 8.94% surface strain (64z 2X) showing cracks are present in the back surface metallized layer; initial round of testing.



(a)

(b)

Figure 32. Flight sample A-3 (Si/Kapton E/Al/Inconel/Al) 7.5z 2x: (a) after undergoing surface strains of 2.54%, and (b) after 2.75% surface strain; initial round of testing.



(a)

(b)

Figure 33. Flight sample A-3 (Si/Kapton E/Al/Inconel/Al) 64z 2x: (a) after undergoing surface strains of 2.54% (with scratches, but no cracks), and (b) after 2.75% surface strain; initial round of testing. Bend-test induced cracks shown within circles.

CPI/Al

Flight samples A-7, B-7 and control sample C-7 were composed of 1 mil thick clear polyimide CP1 with an Al (VDA) coating on the backside of the material. Pre- and post-bend-test images are provided in Figures 34-36. As seen before in Figure 21, due to AO erosion there is a drastic difference in coloring between the flight and control samples. In the control sample, the reflective Al layer appears black in Figure 34. In Figures 35 and 36 the sample appears more white and matte due to AO erosion of the CP1 polyimide. This material had very visible cracking, however it did not run perpendicularly to the sample as was expected, as seen in Figures 35 and 36. This irregular cracking pattern raised suspicion that the sample may be scratched and not cracked, however it was confirmed that the markings were indeed cracks after close analysis. The cracks first appeared with 0.65% strain, but became more pronounced after 0.70% strain. This material was the most embrittled after exposure to the space environment as it cracked under the lowest strain of any of the samples tested.

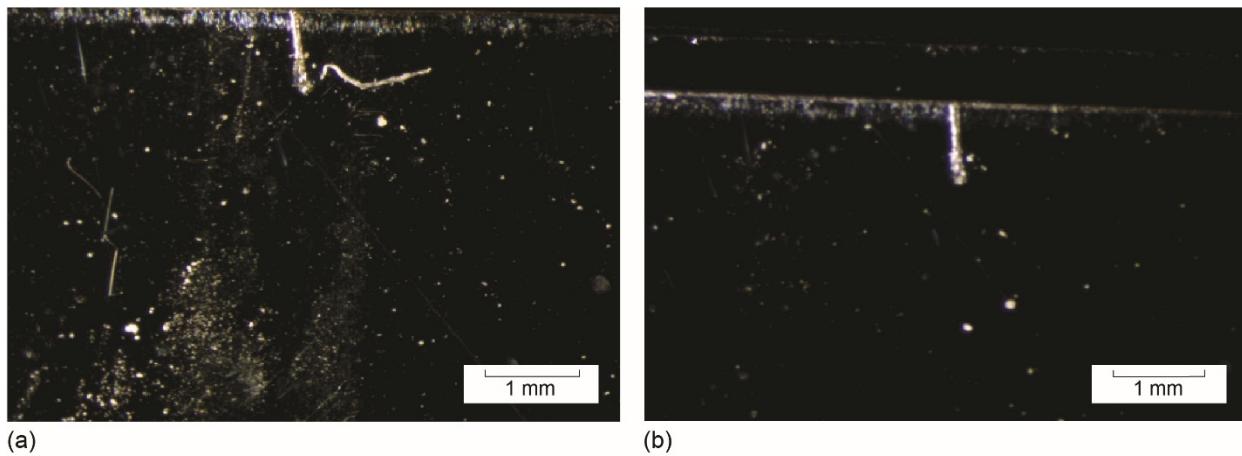


Figure 34. Control sample C-7 (CP1/Al) 7.5z: (a) before undergoing testing and (b) after 4.68% surface strain (sample area shifted in images).

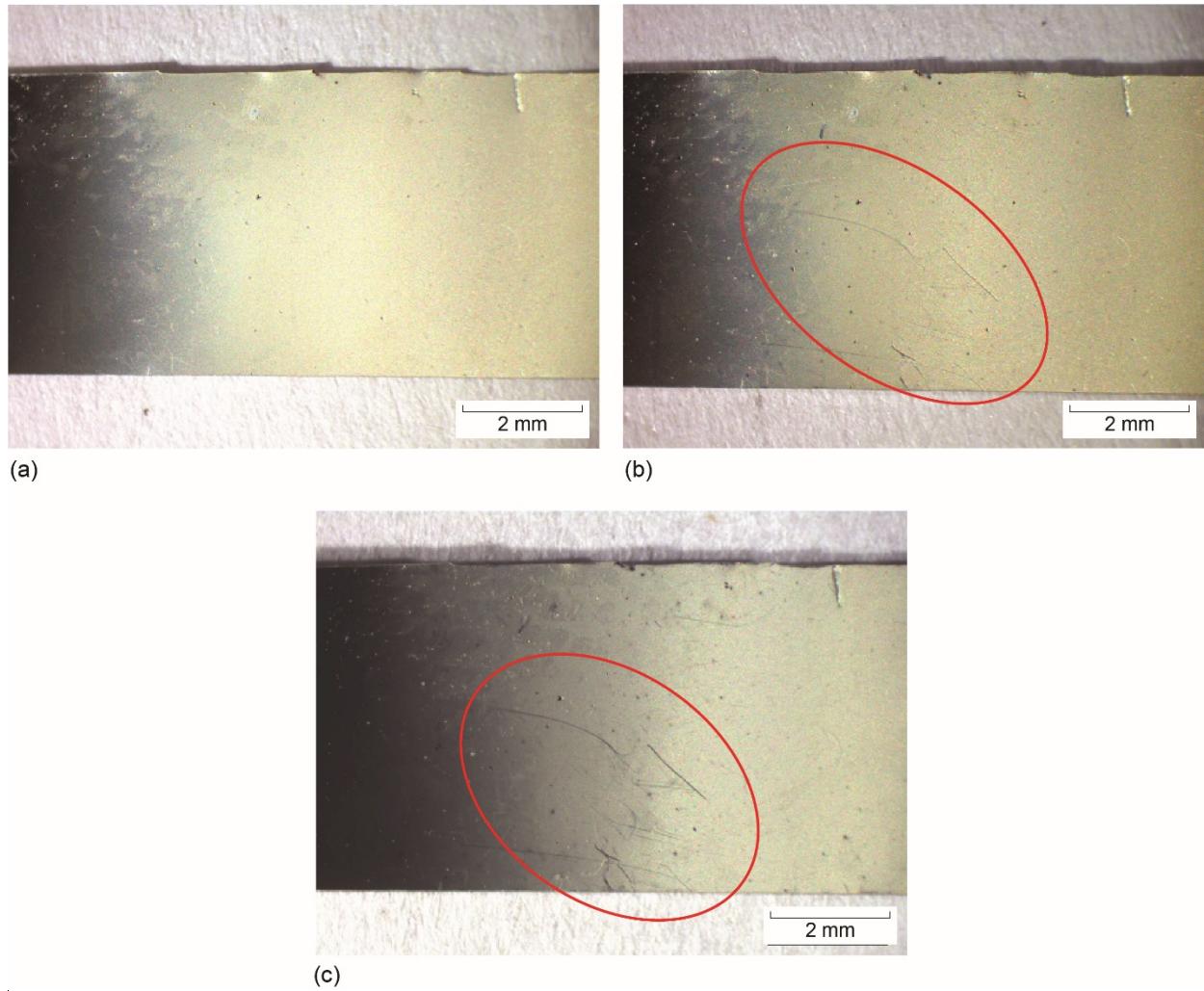


Figure 35. Flight sample A-7 (CP1/Al) 7.5z 2x: (a) before undergoing testing, (b) after 0.65% surface strain with initial cracks circled; second round of testing, and (c) after 70% surface strain with cracks circled.

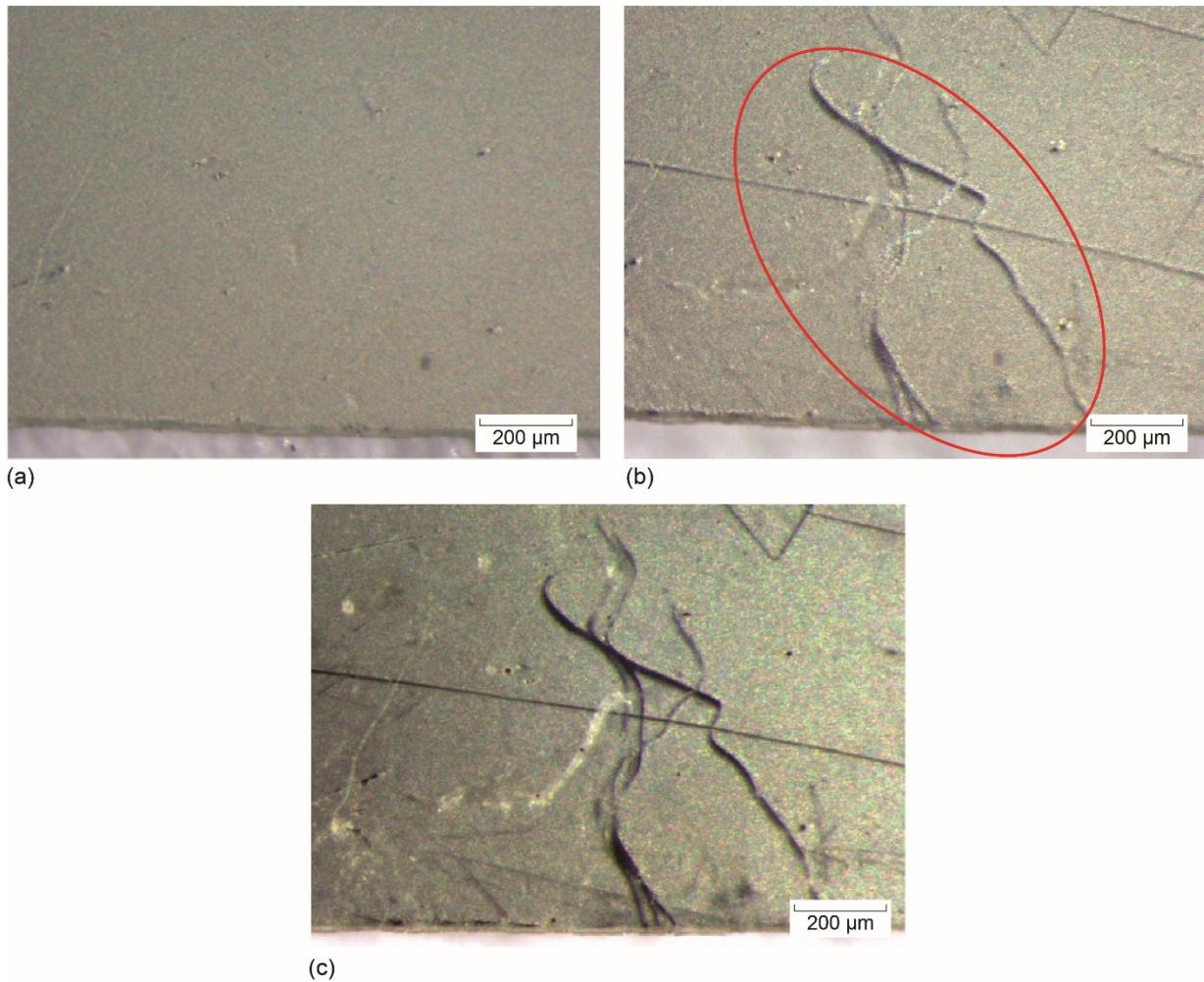


Figure 36. Flight sample A-7 (CP1/Al) 64z 2x: (a) after undergoing surface strains of 0.41%, (b) after 0.65% surface strain; second round of testing (cracks shown within circle), and (c). after 0.70% strain with cracks enhanced.

FEP/Al

Flight samples A-9, B-9 and control sample C-9 were composed of 5 mil thick Teflon FEP with an Al (VDA) coating on the backside of the material. Pre- and post-bend-test images are provided in Figures 37-39. This material had very visible surface cracking in the FEP. In the A samples, cracking occurred after fairly high amounts of strain, making the samples appear to have held up well in the space environment. On the other hand, the B samples cracked at fairly low strain, showing that the sample was actually quite embrittled. This shows that there was a visible difference between the embrittlement of the samples flown over larger diameters and smaller diameters. This sample also scratched easily, as seen in the pre- and post-bend test images.

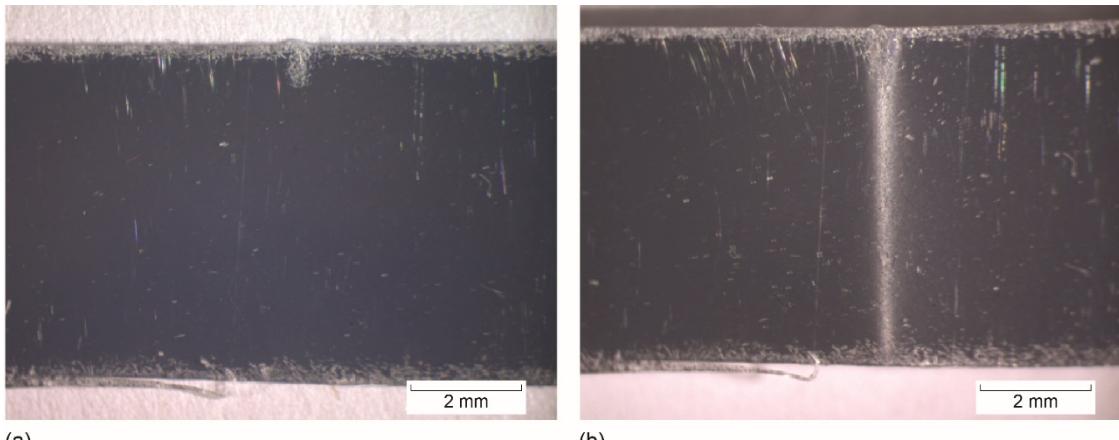


Figure 37. Control sample C-9 (FEP/Al) 7.5z 2x: (a) before undergoing testing and (b) after undergoing 19.70% surface strain. The markings in the post-bend image are a result of the aluminum cracking, not the Teflon itself.

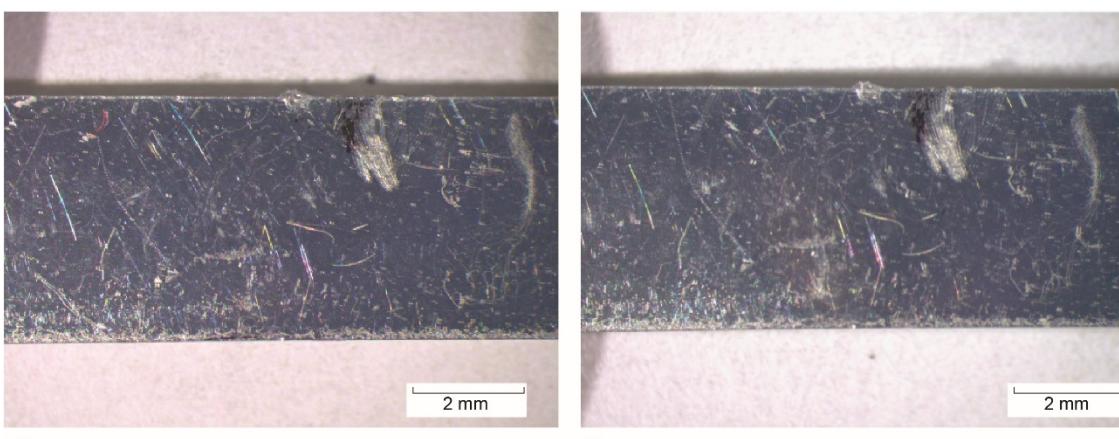


Figure 38. Flight sample B-9 (FEP/Al) 7.5z 2x: (a) before undergoing testing and (b) after undergoing 2.34% surface strain; second round of testing.

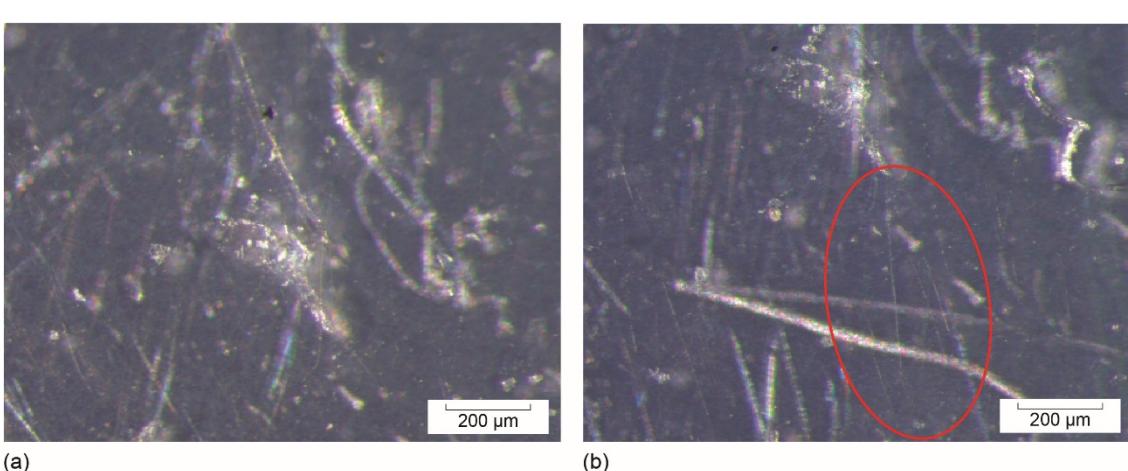


Figure 39. Flight sample B-9 (FEP/Al) 64z 2x: (a) before undergoing testing and (b) after undergoing 2.34% surface strain; second round of testing. Cracks shown within circle, image area is shifted slightly up as compared to the area in a.

Kapton HN/Al

Flight samples A-10, B-10 and control sample C-10 were composed of 1 mil thick Kapton HN with an Al (VDA) coating on the backside of the material. Pre- and post-bend-test images are provided in Figures 40-44. The cracking in these samples was not very visible, making it rather difficult to determine whether or not the samples have cracked. In the A sample, Figures 41-42, there was no cracking. In the B sample, Figures 43-44, however, there was cracking after the first test but not after the second test. This shows once again that there was a difference in embrittlement as a result of being flown under different levels of stress.

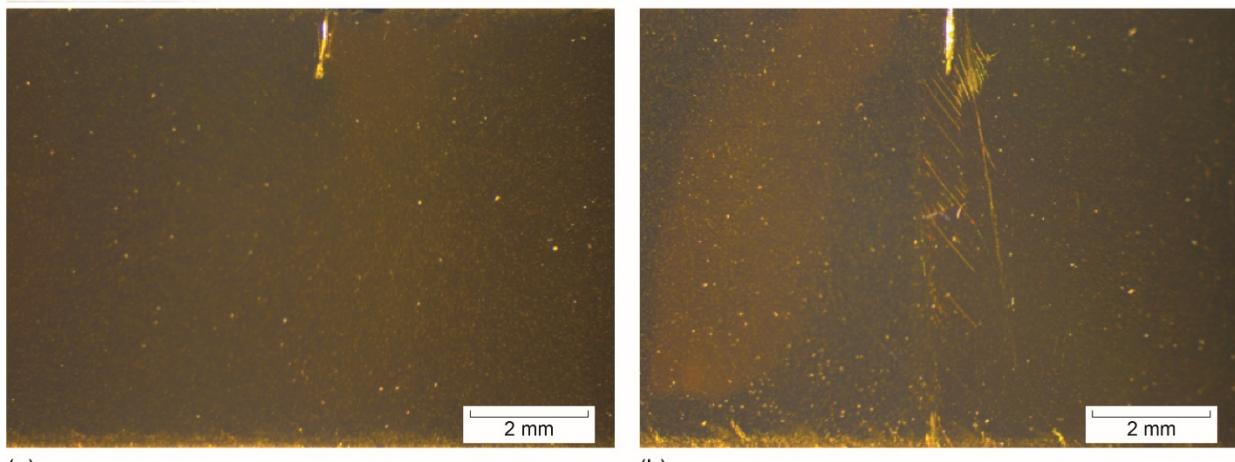


Figure 40. Control sample C-10 (Kapton HN/Al) 7.5z 2x: (a) before undergoing testing and (b) after 4.68% surface strain, the sample did not crack – visible lines are scratches in the bottom Al layer.

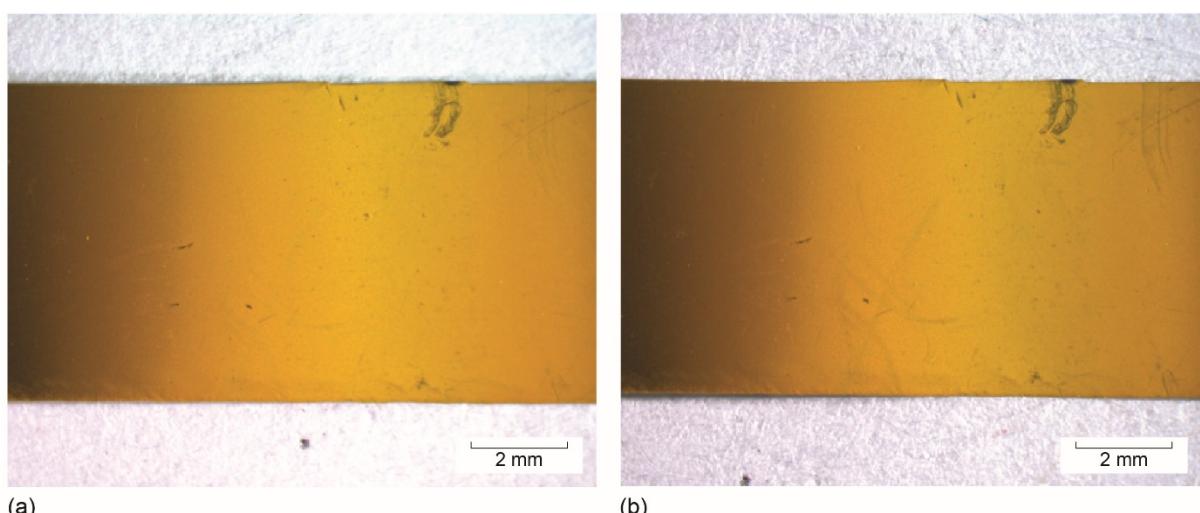


Figure 41. Flight sample A-10 (Kapton HN/Al) 7.5z 2x: (a) before undergoing testing and (b) after 4.49% surface strain, the sample did not crack; second round of testing.

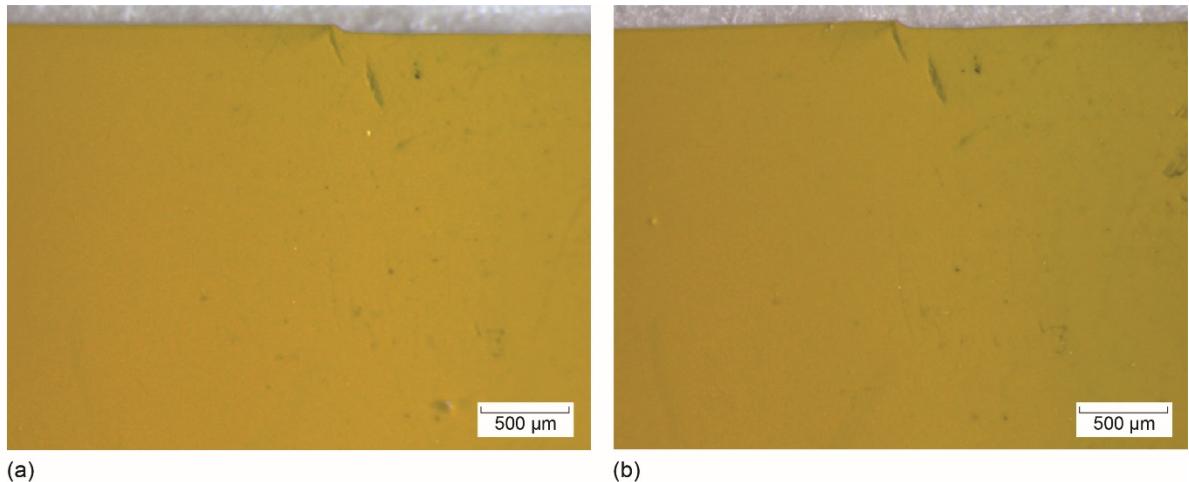


Figure 42. Flight sample A-10 (Kapton HN/Al) 25z 2x: (a) before undergoing testing and (b) after 4.49% surface strain, the sample did not crack; second round of testing.

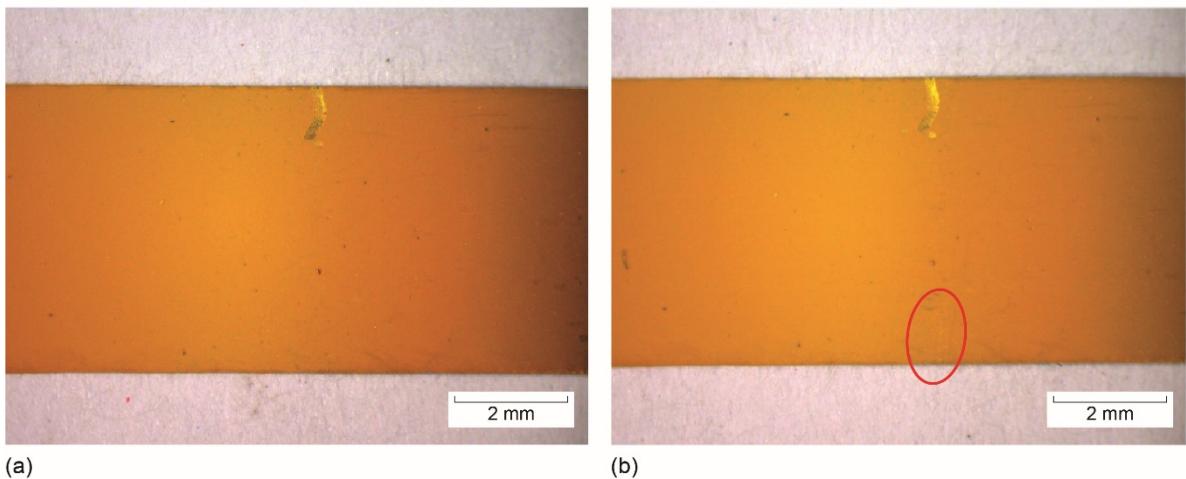


Figure 43. Flight sample B-10 (Kapton HN/Al) 7.5z 2x: (a) after 3.69% surface strain (no cracking), and (b) after 4.49% strain, with sample cracking; initial testing.

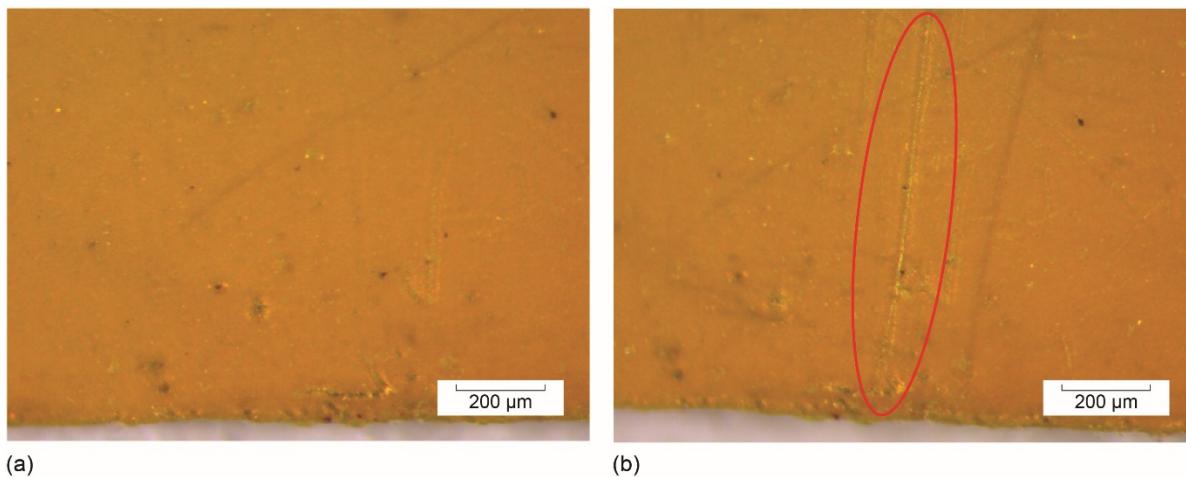


Figure 44. Flight sample B-10 (Kapton HN/Al) 64z 2x: (a) after 3.69% surface strain (no cracking), and (b) after 4.49% surface strain, with sample cracking; first round of testing.

Unexpected Atomic Oxygen

As mentioned previously, AO erosion is visible in six of the samples that underwent bend-testing: A-1, A-7, A-10, B-1, B-7, and B-10. Because the samples were flown while bent over a rounded sample holder, the top of the sample faced the wake orientation while the sides faced zenith and nadir orientations. As seen in Figure 24, the AO erosion was shifted slightly to the left of center. While it may be difficult to tell from the previous figures, due to the samples not laying completely flat, it was determined that this shift was consistently to the left on all samples that exhibited a significant amount of AO exposure. The left side of the samples was the zenith facing side. From this, it can be concluded that the AO eroded the samples at a slight angle, as seen in Figure 45. The AO erosion can remove radiation embrittled material. Therefore, the flight samples that experienced AO erosion are likely less embrittled than they would have been if there had been no AO exposure.

MISSE 6 Polymer Film Tensile Experiment

Many similar polymers were flown as part of the MISSE 6 Polymer Film Tensile Experiment (PFTE), which was exposed to ram and wake LEO exposure for 1.45 years on the exterior of the ISS Columbus Laboratory (Ref. 8). The PFTE flight samples included FEP/Al (2 and 5 mil), Si/Kapton E/Al/Inconel/Al (2 mil), Kapton XC (1 mil), Kapton HN/Al (2 mil), SiO_x/Kapton HN (2 mil) and Al/CP1 (2 mil). The PFTE samples were flown as both stressed and non-stressed tensile samples. Post-flight tensile data indicates that all of the PFTE polymers became embrittled while in space, both the ram and wake exposed samples (Ref. 8). Although neither the A nor the B FSEE Kapton XC/Al samples cracked during bend-testing, cracks in the textured black samples would be extremely difficult to see. Therefore, it is likely that these samples also became embrittled while on-orbit, like the other flight samples. This is likely the case for the A-10 Kapton HN sample also. Thus, obtaining tensile data of the FSEE samples, which would provide more quantitative data than the subjective bent-test data, is desired.

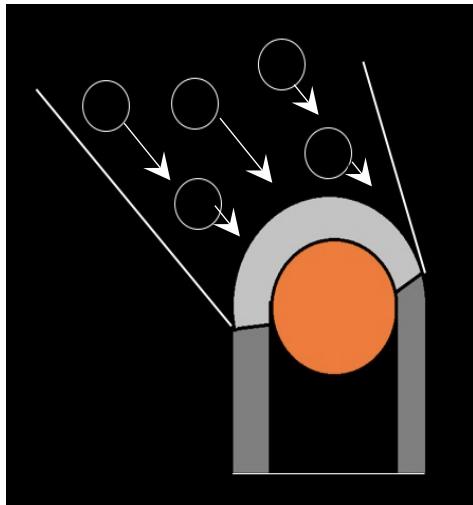


Figure 45. Cross section illustration of AO hitting a sample from an angle. The small circles with arrows represent the AO hitting the sample. The light grey represents the sample and the orange circle is the flight holder the samples are wrapped around.

Conclusions and Future Work

Of the 10 FSEE flight samples tested, seven show increased embrittlement through bend-test induced cracking at surface strains from 0.65% to 8.11%, as compared to the control samples which did not crack. When samples are brittle, less post-flight strain is necessary to induce cracking. The initial set of tests indicated that the samples flown on the 0.375-inch diameter holder cracked at a lower percent strain than the same material flown on the 0.25-inch diameter holder. This would indicate that samples bent over a larger diameter, and under less surface strain on-orbit, became more embrittled while in space. But, the trend was not consistent with the second set of testing. Only the FEP/Al samples consistently required a lower strain for cracking for the sample flown on the 0.375 inch mandrel than for the sample flown on the 0.25 inch mandrel in both sets of tests. It is not statistically clear if more tensile stress during space exposure reduces post flight embrittlement. It may be possible that more surface stress on a sample during flight exposure may result in greater on-orbit creep of the surface material. Therefore, after many thermal cycles and heating, it is possible, for some materials, to gradually have a lower surface tensile stress with time. Thus, samples that experience greater on-orbit creep would require a smaller mandrel, or a higher surface strain, to produce surface cracking post-flight. Because the samples were exposed to high amounts of AO for a short period of time, it is likely that the AO eroded away some of the embrittled surface of the samples, potentially causing the wake samples to be less embrittled than if they were exposed to true wake exposure.

From the surface embrittlement data alone, it was determined that Kapton XC/Al was the least embrittled material, as it did not show any signs of cracking. Kapton HN/Al would be a close second, as its 0.375-inch sample cracked during the first round of testing but both samples underwent the second round of testing without showing any signs of cracking. On the contrary, the CP1/Al sample was the most embrittled as it cracked at an extremely low surface strain in both the 0.25-inch and 0.375-inch samples after both rounds of testing. Thus, the bend-test data indicates that these two Kapton samples were the least embrittled. But, it should be noted that tensile data for the MISSE 6 PFTE indicated that Kapton XC and Kapton HN can become embrittled in a wake flight orientation in space. Thus, obtaining tensile data of the FSEE samples, which would provide more quantitative data than the subjective bent-test data, is desired.

The samples that were not bend-tested are planned to be tensile tested. The samples will have their thicknesses measured with a Heidenhain MT12 digital thickness gauge ($\pm 0.5 \mu\text{m}$) to determine the cross-sectional area for stress calculations. Then, they will be tensile tested to determine bulk embrittlement. The tensile results will be compared to the surface embrittlement bend-test data to help determine which materials are most sensitive to space induced embrittlement and to see if surface embrittlement correlates with bulk embrittlement.

In the future, should a similar experiment be performed, it would be ideal to fly not only samples bent over various diameter holders but also flat samples made of the same materials. This would allow for a more accurate analysis as to whether material blankets that have been bent around corners become more embrittled than flat material blankets.

References

1. Dever, J. A., de Groh, K. K., Messer, R. K., McClendon, M. W., Viens, M., Wang, L. L., and Gummow, J. D., 2001, "Mechanical Properties of Teflon FEP Retrieved from the Hubble Space Telescope," *High Performance Polymers*, 13 (2001), S373-S390.
2. de Groh, K. K., Gaier, J. R., Hall, R. L., Espe, M. P., Cato, D. R., Sutter, J. K., and Scheiman, D. A., "Insights into the Damage Mechanism of Teflon FEP from the Hubble Space Telescope," *High Performance Polymers*, 12 (2000), 83-104.
3. Flexural Test ASTM D790 ISO 178, 2017, *Ptli.com*.
4. Guo, A., Yi, G. T., Ashmead, C. C., Mitchell, G. G., de Groh, K. K., and Banks, B. A., "Embrittlement of MISSE 5 Polymers after 13 Months of Space Exposure," NASA TM-2012-217645, September 2012.

5. de Groh, K. K., Banks, B. A., Yi, G. T., Haloua, A., Imka, E. C., Mitchell, G. G., Asmar, O. C., Leneghan, H. A., and Seckar, E. A., “Erosion Results of the MISSE 7 Polymers Experiment and Zenith Polymers Experiment After 1.5 Years of Space Exposure,” NASA TM-2016-219167, March 2017.
6. Finckenor, M. M., Moore, C., Norwood, J. K., Henrie, B. and de Groh, K. “Estimated Environmental Exposures for MISSE-7B,” Proceedings of the National Space and Missile Materials Symposium, Tampa, FL, June 2012.
7. Hung C-C., de Groh, K. K. and Banks, B. A., “Optical and Scanning Electron Microscopy of the Materials International Space Station Experiment (MISSE) Spacecraft Silicone Experiment,” Proceedings of the 10th International Conference on Protection of Materials and Structures from the Space Environment (ICPMSE-10J), Astrophysics and Space Science Proceedings 32, Springer, 2012, pp 93-103; also NASA TM 2012-217678, August 2012.
8. Miller, S. K. R and Seckar, E. A., “An Examination of Radiation Induced Tensile Failure of Stressed and Unstressed Polymer Films Flown on MISSE 6,” Proceedings of the ‘12th International Symposium on Materials in the Space Environment (ISMSE 12)’, Noordwijk, The Netherlands (ESA SP-705, February 2013).

