Overview of the MISSE-16 Mission and Preliminary Characterization of Novel Space Weathered Materials

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Abstract

The Materials International Space Station Experiment (MISSE) project is a series of experiments that began in 2001, involving the deployment of external experiment platforms on the International Space Station (ISS). Since then, more than 4,000 materials were tested yielding valuable data and insights into the performance of materials and coatings in the harsh space environment, as well as providing a platform for testing and validating new materials and technologies. The results of the MISSE experiments have contributed to the development of more durable and reliable spacecraft and equipment, as well as improving our understanding of the space environment and its effects on human space exploration.

To gain deeper insights into the changes in material properties of both novel and heritage spacecraft materials over the course of a mission, we proposed to enhance the MISSE hardware. This was achieved by integrating a high-resolution camera that can capture both RGB and IR color data, along with an updated illumination scheme. The identical sets of selected materials were mounted on the Zenith, Ram, and Wake faces of the ISS to expose them to predominant vacuum ultraviolet (VUV), atomic oxygen, and high-energy electron conditions. In addition, we exposed flight duplicates of these materials to a simulated space environment, which included sequential exposure to the same irradiation species on the

ground. This allowed us to study the effects of space exposure on the materials in a controlled manner and to compare their behavior in both real and simulated space environments.

The MISSE-FF flight sample collection comprised different classes of polymers, including polyimides from the Kapton® family, manufactured by E.I du Pont de Nemours and Co., Polyethylene terephthalate (PET) materials, liquid crystal polymers, PI/Polyhedral Oligomeric Silsesquioxanes (POSS), and carbon and glass fiber reinforced polymers. The experimental cadence during the MISSE-16 mission (~6 months) included daily images of each sample for the first week, weekly for the next 2 months, and monthly the remaining duration of the mission. This paper will focus on the flight duplicate as well as the postflight optical characterization of novel and heritage materials currently under observation on the MISSE-FF.

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1 Introduction

Spacecraft materials exposed in low Earth orbit (LEO) are subjected to a variety of factors that lead to material changes and/or degradation. These environmental factors come in various forms including, ionizing radiation, electrons, protons, oxygen atoms, micrometeoroids and orbital debris (MMOD), vacuum, and temperature fluctuations [1]. These elements can physically and chemically damage polymer materials used in spacecraft, degrading their functionality, and reducing mission lifetimes. The key aspects that will be addressed in this paper regarding degradation of polymers are atomic oxygen (AO) exposure, vacuum ultraviolet (VUV) exposure, and radiation. [2]. Understanding the effects of these hazards individually and together is essential for selecting suitable materials for specific space projects.

VUV exposure breaks bonds in polymers, degrading their thermo-optical properties but does not cause direct atomic displacement. In contrast, AO exposure is a severe hazard, leading to surface erosion, chemical composition changes, alterations in optical properties, and the formation of particulate and molecular contamination on spacecraft surfaces. The impact of ionizing radiation on materials depends on factors like the type of radiation, its energy, and dose. High-energy electron bombardment can break polymer bonds, leading to degradation in optical, mechanical, and charge transport properties [3] [4]. This report highlights our team's ongoing progress in examining the impact of a simulated LEO environment on the optical, charge transport, and surface morphology properties of specific spacecraft polymers.

In this research, we have utilized a laboratory to simulate space conditions in the LEO environment and separately expose materials to high-energy electrons and AO exposure. Additionally, we plan to establish correlations between the evolving material properties and their behavior over a mission's lifespan. To achieve this, we will analyze the spectral reflectivity changes exhibited by these materials when exposed to different components of the LEO environment throughout the six-month-long 16th Materials International Space Station Experiment Flight Facility (MISSE-FF) mission.

2 Materials and Methods

2.1 Materials

Materials studied in this work represent the flight duplicate and space aged samples that were launched as part of the Materials International Space Station Experiment Flight Facility (MISSE-FF) in July 2022. The MISSE-FF study comprised of 14 different materials that covered different classes of polymers like

polyimide (PI) from the Kapton® family, polyethylene terephthalate (PET), liquid crystal polymer (LCP), Polyhedral Oligomeric Silsesquioxanes (POSS), and carbon fiber and glass fiber reinforced polymers.
The focus of this study is PI material Kapton® CS, PET material Mylar®MO21 and LCP material Zenite®.
It is not feasible to talk about all the materials investigated for the MISSE-16 mission. This paper will focus on one of the Kapton® materials Kapton®CS that is a clear, smooth, and thermoformable film. It shows promise as a potential substitute for traditional PI films in the construction of multi-layer insulation (MLI) blankets. Additionally, Kapton®CS could prove to be an important material in the development of next generation space solar cells.

2.2 Space Simulated Irradiation Procedure

2.2.1 Electron Irradiation and Vacuum Ultraviolet (VUV) Exposure

Experiments at the Spacecraft Charging and Instrument Calibration Laboratory (SCICL) exposed spacecraft materials to 100 keV high-energy electrons and VUV radiation in the Jumbo space irradiation chamber at Kirtland Air Force Research Laboratory (AFRL) as shown in Fig. 1. The maximum electron fluence applied was 8.5 x 10^{13} electrons/cm^2, equivalent to 10 years of LEO orbit exposure, delivered in 5.5 hours. Prior to irradiation, a 24-hour vacuum dehydration bakeout at 60°C removed absorbed water from the samples. Details of the irradiation procedure are reported elsewhere [5].

To simulate VUV radiation effects on spacecraft materials, three Resonance KrLM-LQD12 lamps were used in the SCICL's Jumbo chamber. These lamps emitted photons with peak wavelengths of 116.5 nm and 123.6 nm, with photon fluxes of 5.5 x 10^{20} photons/(s · cm^2 · Å) and 1.06 x 10^{20} photons/(s · cm^2 · Å), respectively.

Each sample received an average of 50.9 equivalent sun hours (ESH) per carousel rotation, calculated based on Lyman-Alpha flux in-vacuum conversion. For a six-month simulation, assuming the ISS completes approximately sixteen 90-minute revolutions per day, the expected solar net flux was 1094 ESH. An in-house developed code determined the sample angles relative to the sun based on orbital beta-angle and ISS position. At an average intensity of 50.9 suns, a 6-month aging experiment was completed in around 23 hours.

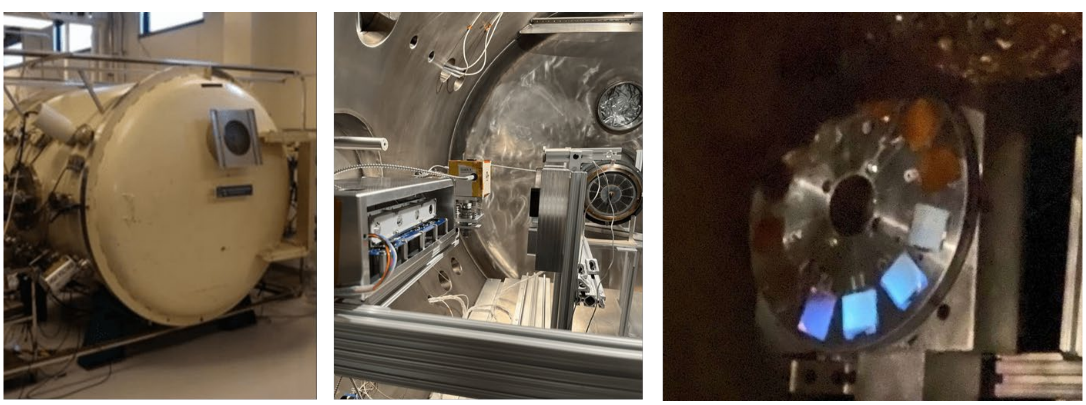
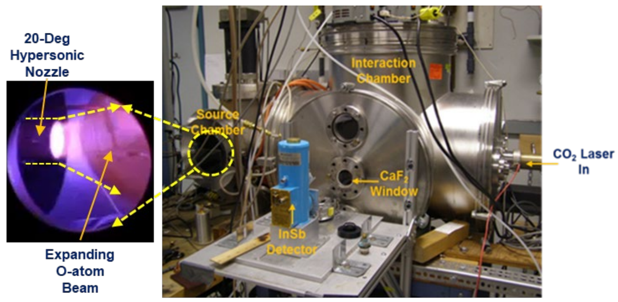


Image credit: AFRL

Fig. 1: JUMBO Weathering Chamber at SCICL

2.2.2 Atomic Oxygen (AO) Exposure

To evaluate the effects of an oxygen atom beam on sample materials, exposure testing was conducted according to ASTM-E2089-15A using the FAST source at Physical Sciences Inc shown in Fig. 2. The effective peak atomic oxygen fluence during the exposure was calculated using the witness sample density (Kapton®HN, 1.427g/cm^2) and LEO erosion yield of the same material (3 x 10^{-24} cm^3/O-atom). The effective peak atomic oxygen fluence during the run was found to be 3.1 × 1020 O-atom/cm^2, which corresponds to 6 weeks of LEO exposure. Prior to exposure, all samples were vacuum sealed for 24 hours to remove any water. They were then weighed over time to monitor water absorption and enable dry mass calculation. Further details on the AO-irradiation procedure can be found in [6].



U.S. Patent 4,894,511, Foreign Patent

Image credit: PSI

Fig. 2: AO exposure chamber at PSI

2.2.3 Characterization

Characterization of materials aged on ground and in space were conducted using multiple techniques. For the flight duplicate samples, the direct hemispherical reflectance (DHR) of control (pristine), electron-irradiated, and VUV-exposed materials were measured in situ during the irradiation procedure according to the optical data acquisition procedure reported elsewhere [7]. Optical properties of electron irradiated, VUV exposed, and AO-exposed materials (space and flight duplicates) were characterized using Perkin-Elmer UV/Vis spectrometer within 200 – 2000 nm spectral range and FTIR spectroscopy. The Bidirectional Reflectance Distribution Function (BRDF) of the pristine and exposed materials were collected using a measurement system designed using a light source and receiver fore-optic that both have a finite angular extent [8]. The measurement is referred to as the hemispherical reflectance factor (HCRF) measurement. Measurements were conducted in the principal plane of illumination using fixed viewing angles while the illumination angles varied within the range of 0◦ to 70◦ degrees (specific values for each measurement are provided separately). Absolute reflectance measurements were conducted at the Optical Measurement Center (OMC) located at NASA JSC Houston. Details of the experimental setup is described in detail elsewhere [9]. Surface morphology and roughness of studied materials were examined using Bruker Dimension ICON atomic force microscopy (AFM) allowing measurement of surface roughness up to 5 μm on areas as large as 200 μm x 200 μm. Mass loss of AO-exposed materials was measured with 10 microgram accuracy using the microgram balance. Prior to weight measurements, all samples were stored in the vacuum chamber for 24 hours to remove water. They were then removed and weighed over time to monitor water absorption and allow for the dry mass to be calculated. Electrical resistivity of the samples was measured on using the standard resistivity test fixture on the Keithley model 8009 meter in accordance with the ASTM D257 standard [10].

3 Results and Discussions

3.1 Ground space simulated aged materials characterization

Figure 3 below represents the DHR curves measured in-situ of the control (pristine) and electron irradiated Kapton CS in 400-2000 nm spectral range taken at different levels of space simulated electron exposure conditions. Transmission characteristics (200 – 800 nm wavelength range) of pristine and exposed to different components of the space-simulated weather is presented on Fig. 4. FTIR transmission curves are shown in Fig. 5. Representative HCRF data which were taken at one selected pair of viewing and illumination angles ($\Theta_I = 0°$ and $\Theta_V = 30°$) is presented in Fig. 6. The absolute reflectance of pristine and electron irradiated sample presented in Fig. 7.

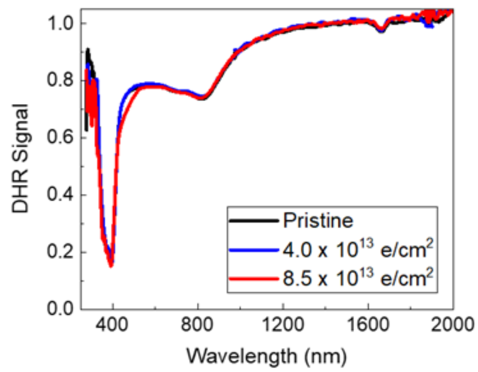


Fig. 3: DHR curves for Kapton®CS material exposed to electron radiation.

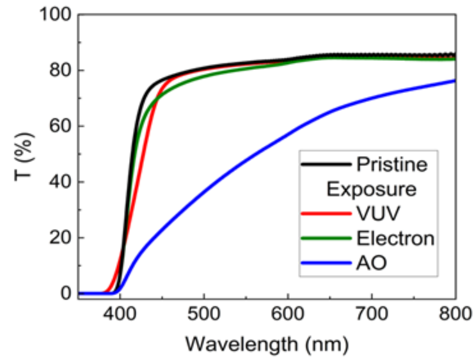


Fig. 4: Transmission curves of Kapton®CS material exposed to different space simulated environments.

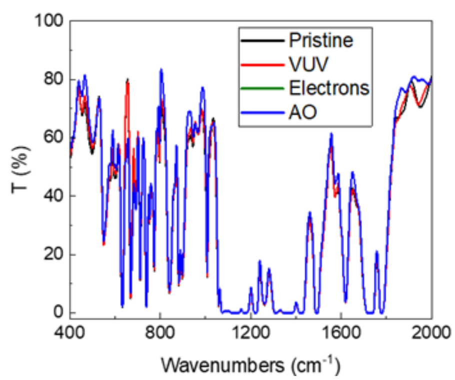


Fig. 5: FTIR curves Kapton®CS exposed to different space simulated environments.

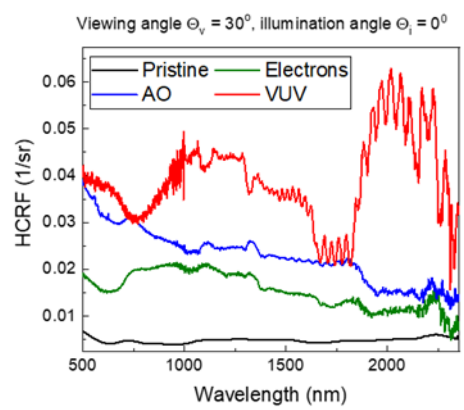


Fig. 6: HCRF curves Kapton®CS exposed to different space simulated environments.

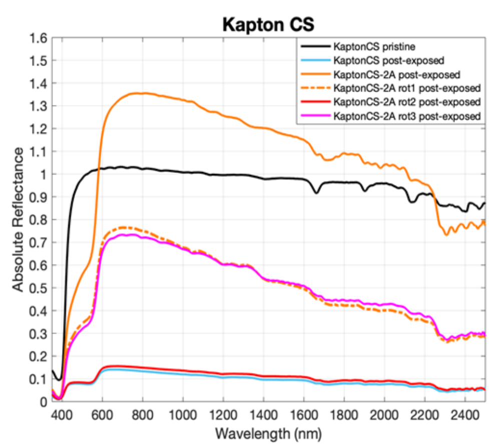


Fig. 7: Absolute reflectance measurement for Kapton® CS for pristine and electron irradiated.

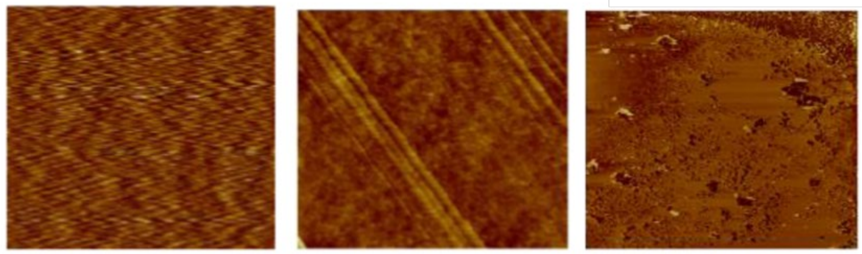


Fig. 8: 5µm x 5µm AFM scans of pristine, electron irradiated and AO-exposes Kapton® CS

Figure 8 depicts the AFM images of pristine, e-irradiated and AO- exposed materials. Table 1 summarizes the surface roughness of the samples. Average surface roughness values were average values of several 5 µm x 5 µm scans taken at different parts of the sample.

Table 1: Surface roughness of Kapton CS exposed to different components of space simulated environment.

Material	Thickness (mil)	Pristine (surface roughness)	E-irradiated (surface roughness)	AO-exposed (surface roughness)
Kapton® CS	2	5.5 nm	12.3 nm	136.3 nm

The electrical conductivity of the pristine, electron irradiated, VUV exposed and AO exposed were evaluated by measuring the volume resistivity and surface potential decay using the ASTM D-257 methodology. Tables 2,3 and 4 summarize the electrical properties of the aged materials.

Table 2: Volume resistivity measurements of pristine, electron irradiated and VUV exposed Kapton® CS

Resistivity (Ω x cm)	**100V**	**500V**	**1000V**
Pristine	1.6×10^{17}	1.43×10^{17}	8.83×10^{16}
E-irradiated	2.9×10^{17}	3.3×10^{17}	1.7×10^{16}
UV Exposed	1.46×10^{17}	9.92×10^{16}	9.4×10^{16}

Table 3: Volume resistivity measurements of pristine, and AO-exposed Kapton® CS

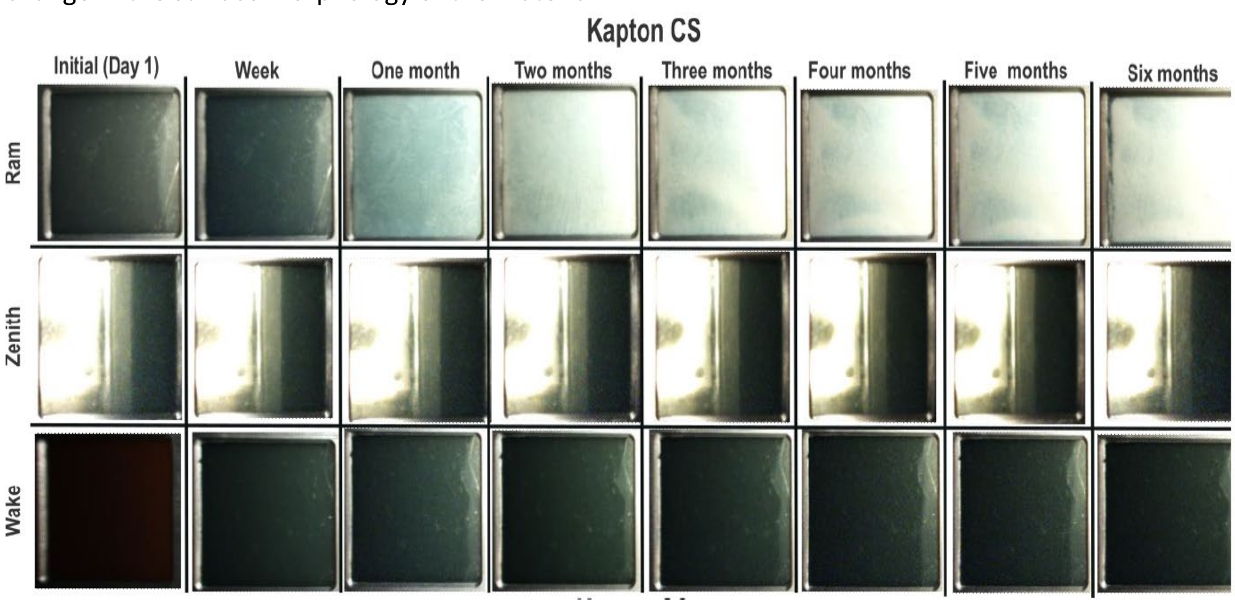
Resistivity (Ω x cm)	**100V**	**500V**	**1000V**
Pristine	5.21×10^{15}	2.53×10^{16}	7.42×10^{16}
AO Exposed	-	2.67×10^{16}	7.58×10^{16}
Δ ($R_{exposed}$-$R_{pristine}$)	-	0.14	0.16

Table 4: Surface resistivity measurements of pristine, and AO-exposed Kapton®CS

Resistivity (Ω)	100V	500V	1000V
Pristine	0.3×10^{16}	0.41×10^{16}	0.8×10^{16}
AO Exposed	0.7×10^{16}	1.7×10^{16}	1.5×10^{16}
Δ ($R_{exposed}$-$R_{pristine}$)	0.4	1.29	0.7

Among three different components of the LEO space environment AO exposure has the most detrimental effect on the surface of Kapton®CS. AO exposure breaks the polymer bonds and induces oxidative decomposition which results in substantial erosion of the polymer surfaces. This is evidenced by the mass loss and increase in roughness on the polymer surface. AO exposure resulted in an increase in surface roughness from 5.5nm (pristine) to 136nm. Mass loss, erosion rate and relative erosion rate with respect to Kapton®HN (used as standard for comparison during AO exposure and erosion of other polymers) of the 2mil Kapton®CS samples after AO exposure was 7.5mg, 4.56 $\times10^{-21}$ g/cm^2/O-atom and 1.60, respectively.

DHR measurements of the electron irradiated sample did not have a significant impact on the material's optical properties. AO exposed samples were significantly degraded and were not measured. Transmission properties of Kapton®CS material underwent a significant degradation following AO exposure and resulted in a loss of transparency of the material apparent when observed visually. Conversely, after high energy electrons and VUV irradiation the transmittance of the studied material did not change significantly. FTIR measurements did not show any new peaks suggesting that no new chemical moieties are formed. HCRF and Absolute reflectance of materials demonstrated a reduction of specularity in the material after space weather aging. There were no significant changes in the volume resistivity of the aged materials. Surface resistivity of AO-exposed materials changed which is most likely caused by the change in the surface morphology of the material.

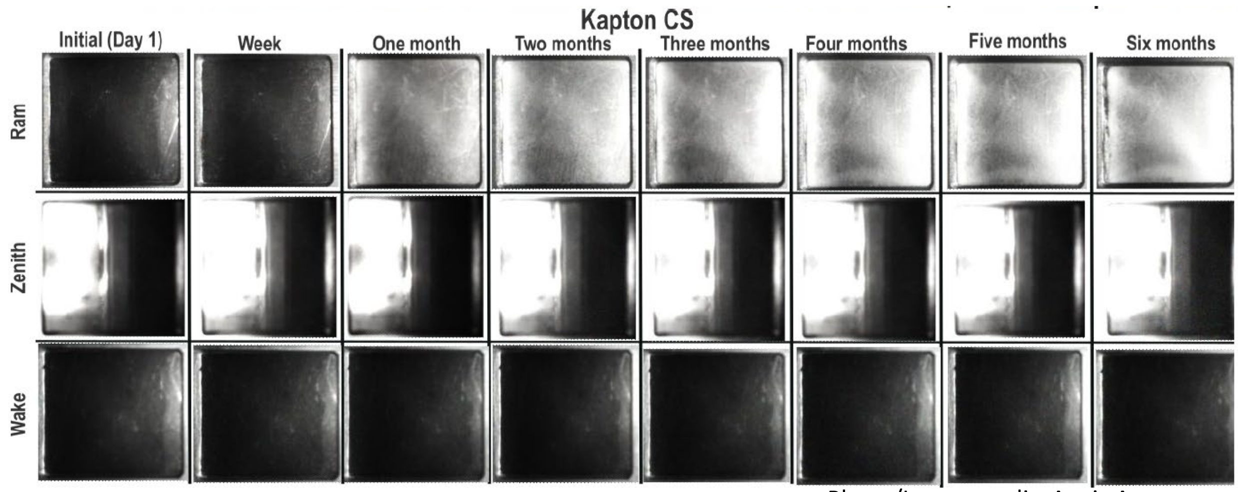


Photo/Image credit: Aegis Aerospace

Fig. 9: On Orbit Visible Camera Images Kapton® CS over six months

3.1.1 On Orbit Aged Samples Characterization

Figures 9 and 10 are RGB and IR images of Kapton CS taken during the MISSE-16 orbital experiment. The samples were mounted on the RAM, Wake and Zenith faces of the ISS.



Photo/Image credit: Aegis Aerospace

Fig. 10: On Orbit IR Camera Images of Kapton® CS over six months

Photo/Image credit: Aegis Aerospace

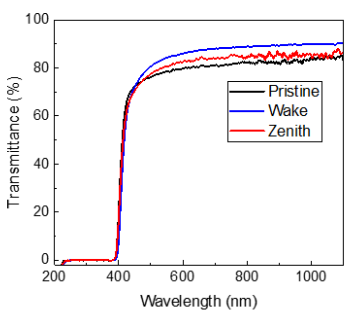


Fig. 11: Transmission curves for space aged Kapton® CS

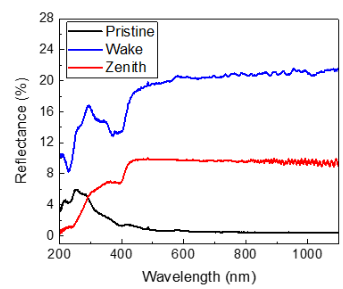


Fig. 12: Reflectance curves for space aged Kapton® CS

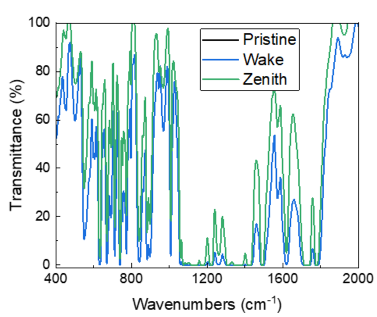


Fig. 13: FTIR curves for space aged Kapton® CS

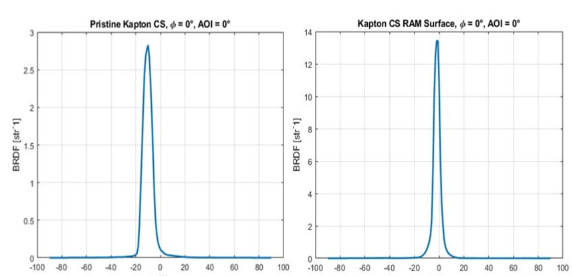


Fig. 14: Mini-Diff BRDF for pristine and AO exposed space aged Kapton® CS

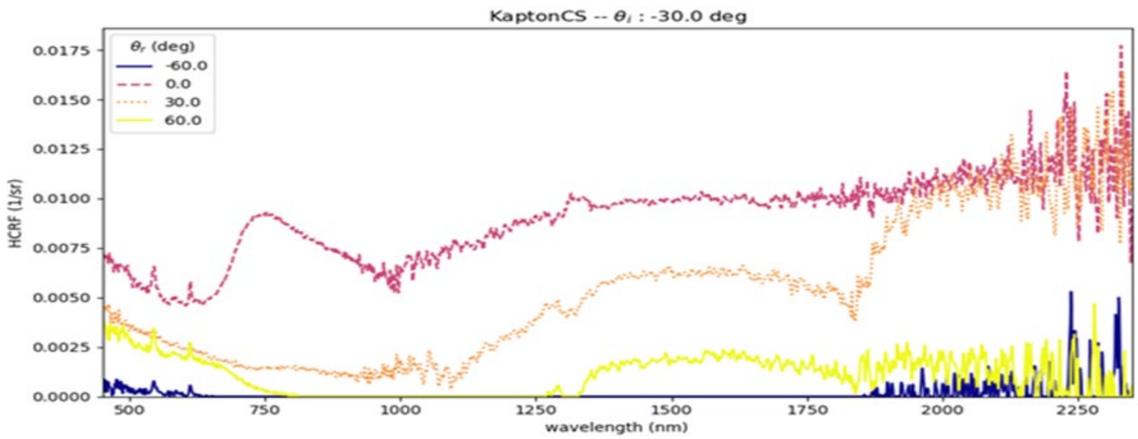


Fig. 15: HCRF curves for space aged Kapton® CS Θ_I = -30° and range of viewing angles

Once the on-orbit samples were returned they were carefully catalogued, and their visual appearances documented. The outermost layer of the wake exposed Kapton CS showed reduced transparency compared to the pristine sample and had a brownish hue. The space facing zenith sample appear darkened and a had a slight brown tint. The ram sample had significant peeling (delamination) and formation of fine particle residue which prevented the optical characterization of that sample. There was no mechanical degradation such as distortion, cracks or delamination on the wake and zenith samples.

DHR measurements for the on-orbit samples were not conducted as optical properties change significantly once samples are exposed to atmosphere. Figure 11 depicts the transmission measurements on the wake and zenith samples appear unchanged from pristine materials. Conversely the reflectance measurement in Fig. 12 show changes between 200-400nm range from the pristine sample. The FTIR spectra for the wake and zenith sample shown in Fig. 13 has overall the same trends as the pristine except the transmission from the zenith sample is slightly increased. This increase at 1331 cm^{-1} and 1400cm^{-1} corresponds to the stretching vibrations of the C-N bonds. In Fig. 14 and Fig 15 are the BRDF measurements on ram exposed Kapton®CS has increased intensity when compared to the pristine sample. This measurement needs further investigation as most of the ram exposed sample was delaminated and hard to place in the instrument to measure.

4 Summary

In-depth characterization of the material properties of both new and historical spacecraft materials during simulated space weather experiments holds significant importance. This is crucial for establishing correlation factors between the actual space exposure and accelerated space weather experiments conducted on the ground. Furthermore, it enables us to make precise predictions about how materials will perform in orbit based on laboratory testing.

The data presented in this paper focused on characterization of on Kapton®CS material that was exposed to LEO space weather conditions. The material was aged under simulated space weather in the laboratory and on-orbit on the ISS. From the preliminary characterization it is apparent that AO exposure causes significant damage whereas electron and VUV exposure has minimum impact both in laboratory experiments and on-orbit samples.

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