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Contamination Results of MISSE 8 Wake and Nadir Samples After 2 Years of Space Exposure on the International Space Station

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May 2024

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Abstract

Spacecraft in low Earth orbit (LEO) are subjected to harsh environmental conditions, including radiation (cosmic rays, ultraviolet, x-ray and charged particle radiation), micrometeoroids and orbital debris, temperature extremes, thermal cycling, and atomic oxygen. In addition, on-orbit spacecraft contamination is a serious spaceflight issue, with silicone contamination being a particular concern. In an effort to understand on-orbit contamination of Materials International Space Station Experiment 8 (MISSE 8) flight samples and other International Space Station (ISS) payloads, contamination studies were conducted post-flight on retrieved Teflon fluorinated ethylene propylene (FEP) samples flown in the wake and nadir orientations on MISSE 8. The wake sample was Teflon FEP (M8-W11) flown as part of the Glenn MISSE 8 Polymers Experiment on the Optical Reflector Materials Experiment-III Ram/Wake (ORMatE-III R/W) tray and exposed to the LEO wake environment for 2.0 years. The nadir samples were silver-Teflon FEP radiator pieces taken from the MISSE 8 Single Events Upset Xilinx-Sandia Experiment II (SEUXSE II) Power Box and exposed to the LEO nadir environment for 2.14 years. The Teflon FEP flight materials were analyzed for changes in surface morphology and chemistry as compared to pristine control samples. The wake samples were also analyzed for changes in optical properties. There was no evidence of a molecular contamination layer present on the surface of the MISSE 8 wake or nadir facing Teflon FEP flight samples, although both the wake and nadir flight samples contained particulate contamination in some regions. The analyzed nadir particles were primarily

*Currently retired.

zinc rich. The majority of analyzed wake particles were comprised of oxidized aluminum with small amounts of zinc and magnesium. The wake particles appear to have arrived early in the mission (or pre-flight) during a single event. This paper provides details of the MISSE 8 sample contamination analyses.

1.0 Introduction

Spacecraft in low Earth orbit (LEO) are subjected to harsh environmental conditions, including radiation (cosmic rays, ultraviolet (UV), x-ray and charged particle radiation), micrometeoroids and orbital debris, temperature extremes, thermal cycling, and atomic oxygen (AO). These environmental exposures can result in erosion, embrittlement and optical property degradation of susceptible materials threatening spacecraft performance and durability. In addition, on-orbit spacecraft contamination is a serious spaceflight issue, with silicone contamination being a particular concern.

Silicones are used as adhesives, potting compounds, and lubricants for various spacecraft components. An example of this is the adhesive for bonding cover glass to solar cells. Although spacecraft designers generally use silicones that meet outgas requirements, silicone fragments can still be evolved in the vacuum environment in LEO and the process is enhanced with AO and/or radiation-induced bond breaking.[1] Silicone fragments that deposit on surfaces with accompanying AO exposure are oxidized, lose hydrocarbons, and convert to a hardened, often crazed, silica-based oxide layer.[2,3] In addition, UV can react with silicone fragments, causing a polymerized contaminant layer to build up. If the silicone deposition is also accompanied by hydrocarbon deposition, a far more optically absorbing coating can result.[1] Examples of darkened silicone contamination have been reported on the Long Duration Exposure Facility (LDEF)[4,5] and the Russian space station Mir[6]. Figure 1 shows an example of AO and UV radiation darkened silicone contamination from LDEF's Passive Solar Array Materials Experiment.[4,5]

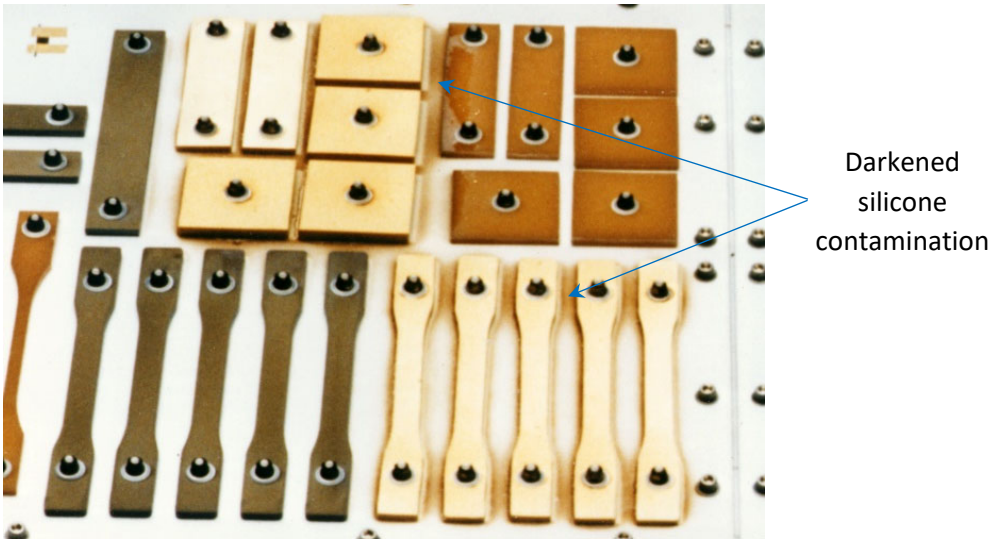


Figure 1. Atomic oxygen and UV darkened silicone contamination on LDEF (arrows point to areas with the dark contamination layer).[4,5]

The Materials International Space Station Experiment 8 (MISSE 8) experiment carriers and neighboring Alpha Magnetic Spectrometer-02 (AMS-02) were planned for launch and installation on ISS during the STS-134 mission in May 2011. In 2010, pre-flight model predictions conducted by the Boeing Space Environments Team indicated that there could be high levels of induced molecular contamination on the MISSE 8 wake and nadir surfaces from the newly deployed AMS-02 facility materials outgassing.[7] The main outgas contaminants were expected to be a mix of silicones (siloxanes and other organic silicones) and hydrocarbons. Due to the AMS-02 materials outgas concern the MISSE 8 ram/wake tray, the Optical Reflector Materials Experiment-III Ram/Wake (ORMatE-III R/W), containing sensitive optical materials was planned to be deployed approximately 2 months after the AMS-02 installation.

The STS-134 mission was launched on May 16, 2011 and the AMS-02 was installed on ISS's upper Payload Attach Point on the S3 Truss on May 19, 2011.[8] The MISSE 8 zenith/nadir Passive Experiment Container (PEC) was installed on the ISS EXPRESS Logistics Carrier 2 (ELC 2) Site 3 and deployed (opened to space) by astronauts during an extravehicular activity (EVA) spacewalk on May 20, 2011. And, the ORMatE-III R/W was installed and deployed in a ram/wake orientation on ELC 2 Site 3 during the STS-135 mission on July 12, 2011. Figure 2 provides an on-orbit photograph of the MISSE 8 zenith/nadir PEC on the ELC 2 and the AMS-02 as photographed during the STS-134 mission May 20, 2011 EVA.

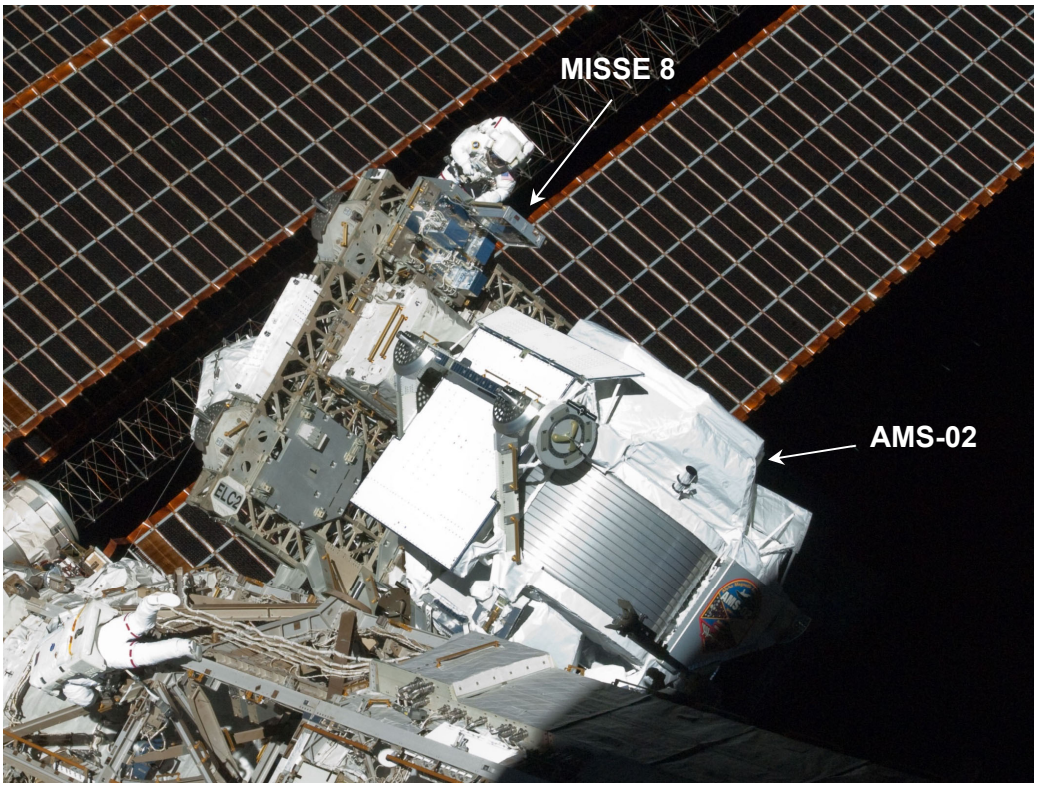


Figure 2. On-orbit photograph of the MISSE 8 zenith/nadir PEC and the AMS-02 taken during the MISSE 8 PEC installation EVA on May 20, 2011.

In an effort to understand on-orbit contamination on MISSE 8 flight samples and other ISS payloads, contamination studies were conducted post-flight on retrieved Teflon™ fluorinated ethylene propylene (FEP) samples flown in the wake and nadir orientations on MISSE 8. The analyzed wake sample was clear FEP (M8-W11) flown in the ORMatE-III R/W tray and exposed to the wake space environment for 2.0 years. The analyzed nadir material was silver-Teflon FEP radiator material from the MISSE 8 Single Events Upset Xilinx-Sandia Experiment II (SEUXSE II) Power Box. The SEUXSE II Power Box was exposed to the nadir space environment for 2.14 years. Nadir surfaces typically do not receive direct solar exposure, hence UV enhanced fixing of molecular contamination (which would contribute to contamination build-up) would not be expected to occur. But, the wake surface does receive direct solar exposure with corresponding low erosion, therefore it would be expected that any arriving molecular contamination would be fixed, or adhered, in place in the wake direction. The Teflon FEP flight materials were analyzed for changes in surface morphology and chemistry as compared to pristine control samples. The wake samples were also analyzed for changes in optical properties. This paper provides details of the MISSE 8 sample contamination analyses.

2.0 Materials International Space Station Experiment 8 (MISSE 8) Overview

The MISSE project is a series of spaceflight experiments flown on the exterior of the ISS to test the performance and durability of materials and devices exposed to the LEO space environment. In the MISSE 1-8 missions, individual flight experiments were flown in suitcase-like containers called PECs that provided exposure to the space environment. The exception was the flight of the smaller MISSE 8 ram/wake ORMatE-III R/W tray. The PECs were closed during launch to protect the samples. Once on orbit, the PECs were attached to the exterior of the ISS during an EVA in either a ram/wake or a zenith/nadir orientation and opened exposing the experiments to the space environment for the duration of the mission. 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 surfaces receive a high flux of directed AO and sweeping (moderate) solar exposure. Zenith facing surfaces receive a low flux of grazing arrival AO and the highest solar exposure. Wake surfaces receive very low AO flux and moderate solar radiation levels similar to ram experiments. Nadir surfaces 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 the 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 especially for AO exposure of zenith and nadir surfaces.

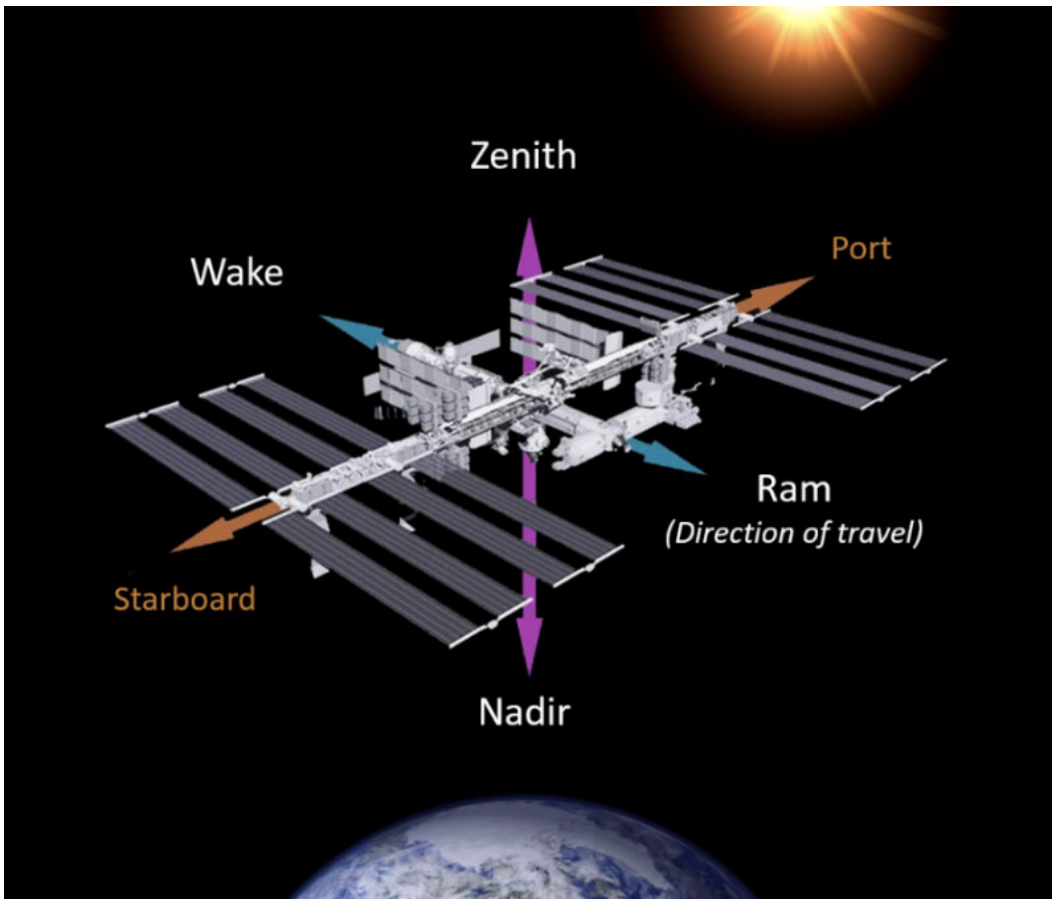


Figure 3. Diagram showing ram, wake, zenith, and nadir flight directions on the International Space Station.

The MISSE 8 mission consisted of the zenith/nadir PEC and the smaller ORMatE-III R/W tray. As stated previously, the MISSE 8 PEC was attached to the exterior of the ISS on the ELC 2 Site 3 in a zenith/nadir orientation during an EVA as part of the STS-134 Shuttle mission on May 20, 2011. The ORMatE-III R/W was deployed in a ram/wake orientation during the STS-135 Shuttle mission on July 12, 2011, approximately two months after deploy of the MISSE 8 PEC. Once positioned on ELC 2, the MISSE 8 PEC and ORMatE-III samples remained exposed to the LEO space environment until they were retrieved on July 9, 2013 after 2.14 and 2.00 years of space exposure, respectively, and returned to Earth in the SpaceX-3 Dragon (CRS-3). Figure 4 shows the location of the MISSE 8 PEC and ORMatE-III R/W on the ISS ELC-2. Figure 5 shows on-orbit images of the PEC (Figure 5a) and ORMatE-III (Figure 5b) as imaged during the STS-135 ORMatE-III R/W deploy mission in July 2011.

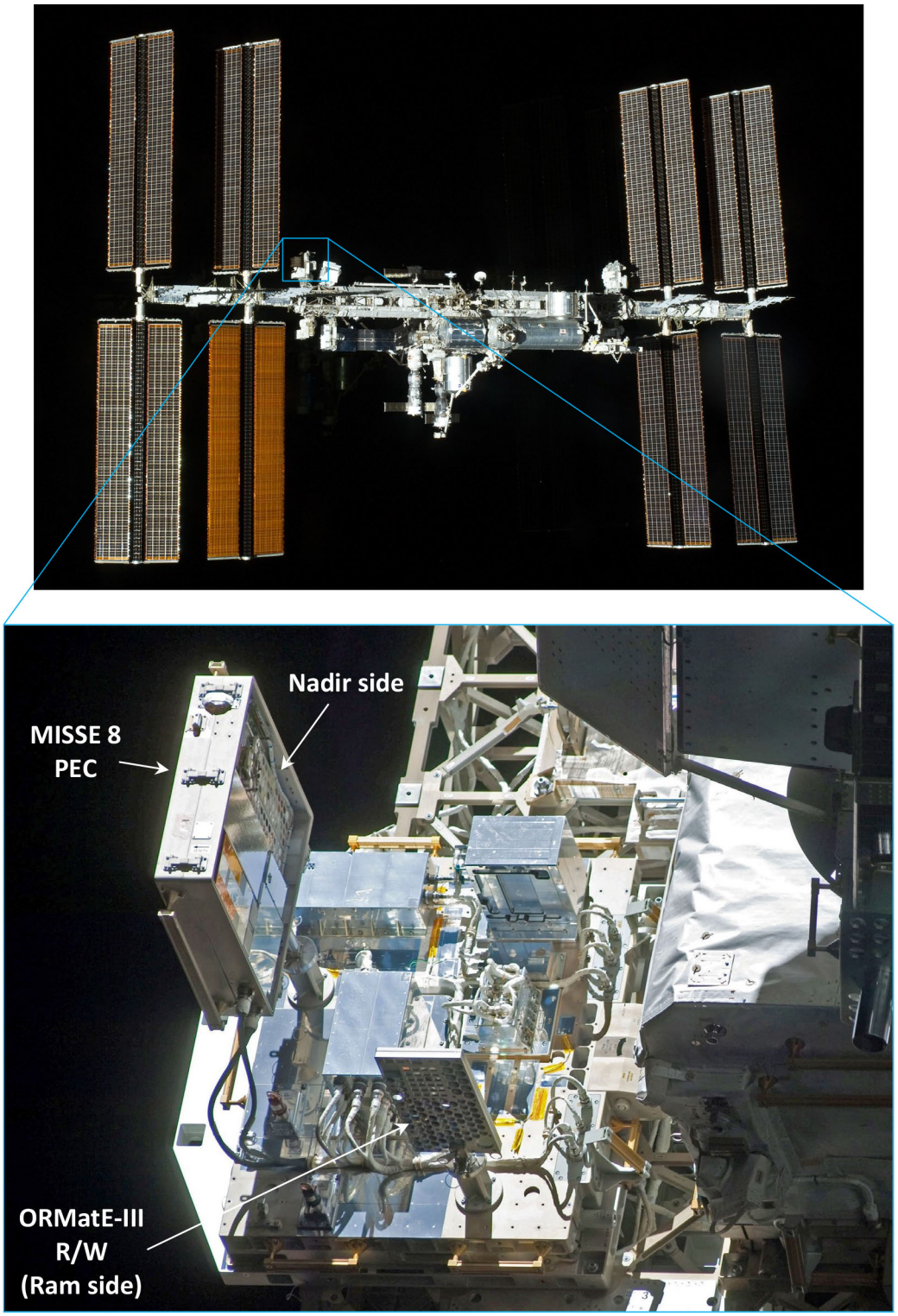
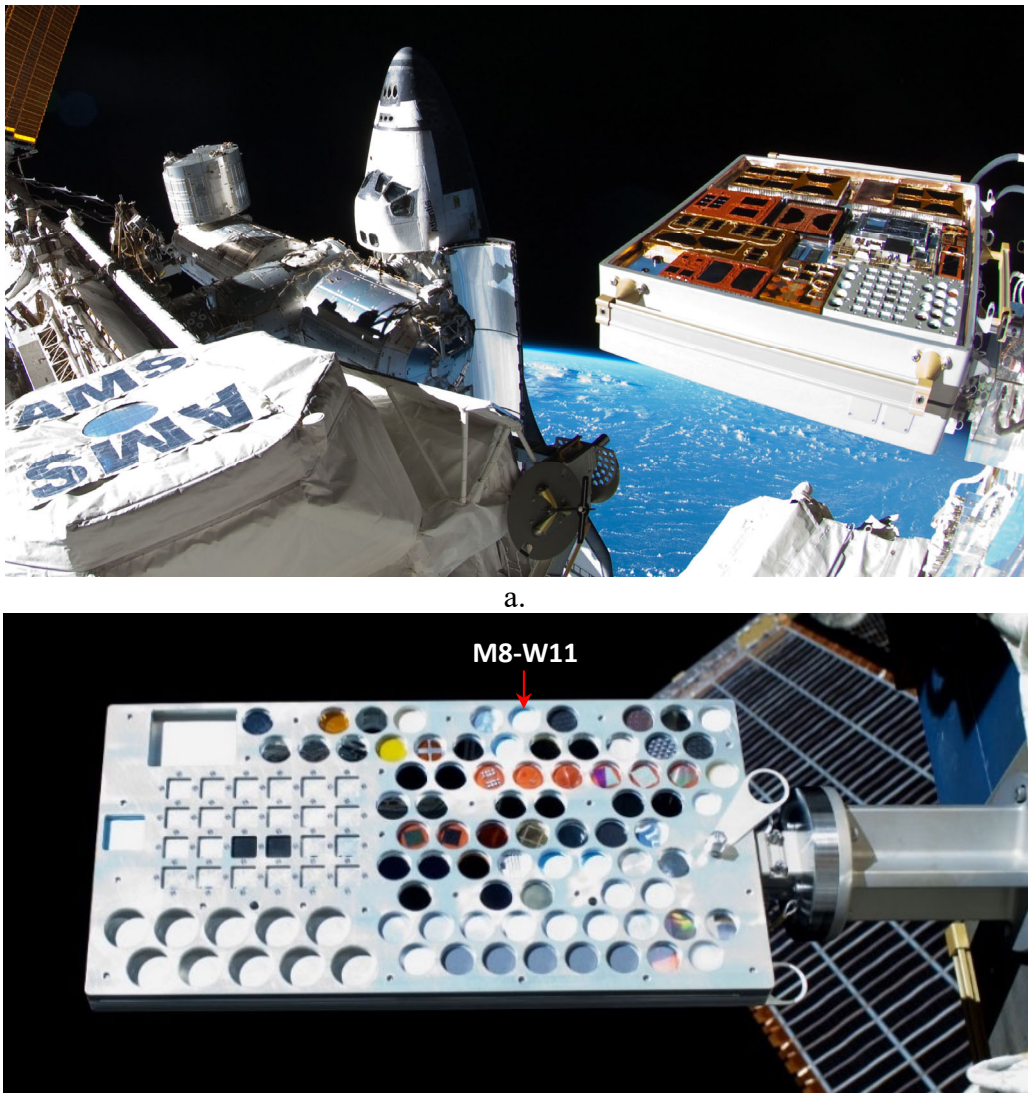


Figure 4. Location of MISSE 8 PEC and ORMatE-III R/W on the ISS ELC-2 as imaged during the STS-135 shuttle mission in July 2011 shortly after deployment of ORMatE-III R/W.



a.

b.

Figure 5. On-orbit images of the MISSE 8 PEC and ORMatE-III R/W as imaged during the STS-135 mission in July 2011: a). The zenith surface of the PEC is visible on the right side of the image and the AMS-02 is visible on the left, and b). The wake surface of ORMatE-III R/W.

3.0 MISSE 8 Contamination Materials

3.1 MISSE 8 Wake Flight Sample

The MISSE 8 wake sample was 2 mil (50.8 µm) thick Teflon FEP. The 1-inch (2.54 cm) diameter flight sample (M8-W11) was flown as part of NASA Glenn Research Center's MISSE 8 Polymers Experiment.[9,10] The Polymers Experiment was a passive experiment that included 42 samples that were flown in ram (8 samples), wake (11 samples) or zenith (23 samples) orientations during the MISSE 8 mission.[9,10] The primary objective of the Polymers Experiment was to determine the effect of solar exposure on the AO erosion yield (E_y, given in cm^3/atom) of fluoropolymers. The Polymers Experiment ram and wake samples were flown on the ORMatE-III

R/W tray. The positon of M8-W11 is shown in Figure 5b. Additional details on the MISSE 8 Polymers Experiment along with the AO E_y results are provided by de Groh in Reference 9.

3.2 MISSE 8 Nadir Flight Sample

The MISSE-8 nadir contamination test materials were silver-Teflon radiator material from the nadir surface of the SEUXSE II Power Box. The MISSE-8 SEUXSE-II experiment was designed to measure Single Event Upset (SEU) radiation effects on field programmable gate arrays (FPGAs).[11] Figure 6 provides an on-orbit photograph of the nadir side of the MISSE 8 PEC with the SEUXSE II Power Box highlighted. The silver-Teflon was 10 mil (254 μm) thick Teflon FEP that is back-surface coated with thin coatings of silver and Inconel (FEP/Ag/Inconel). The silver-Teflon film was adhered to the SEUXSE II Power Box with Y966 acrylic adhesive. The SEUXSE II Power Box was provided to Glenn from the Naval Research Laboratory (NRL) MISSE-8 principal investigator. Two test samples, one for X-ray Photoelectron Spectroscopy (XPS) analyses and one for Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) analyses, were carefully sectioned from the nadir surface (top) of the SEUXSE II Power Box in an area where there was no observable AO effects, as shown in Figures 7 and 8.

3.3 MISSE 8 Control Samples

The MISSE 8 wake control sample (M8-R9-B) was 2 mil (50.8 μm) thick clear Teflon FEP from the same batch of material as the flight sample (M8-W11). The MISSE 8 nadir control sample was silver-Teflon tape (10 mil Teflon FEP/Ag/Inconel/Y966 acrylic adhesive) provided by NRL.

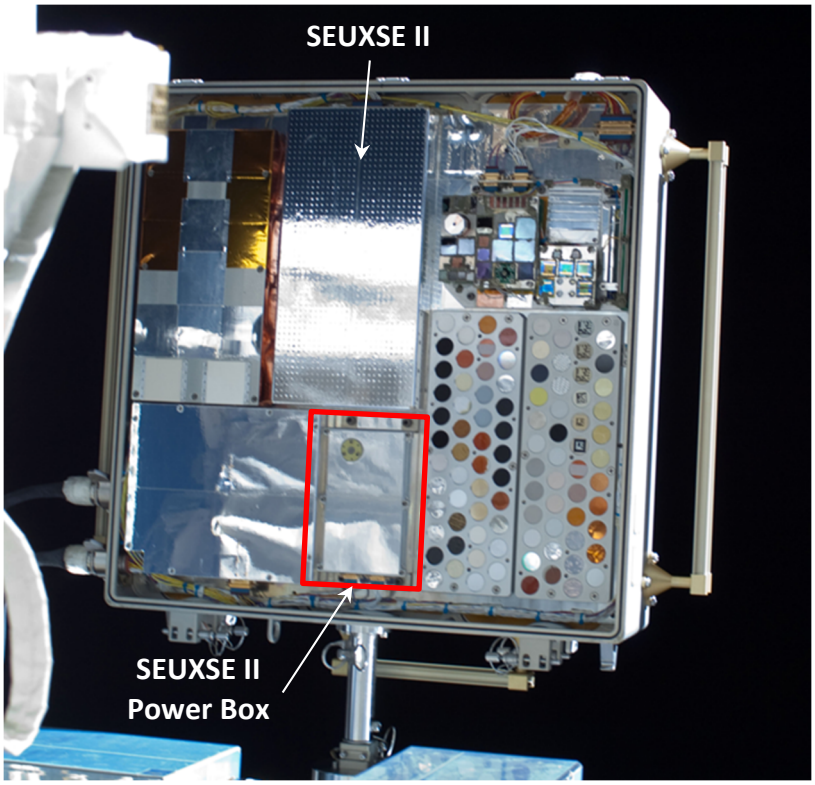


Figure 6. The SEUXSE II Power Box (red box) on the nadir side of the MISSE 8 PEC.

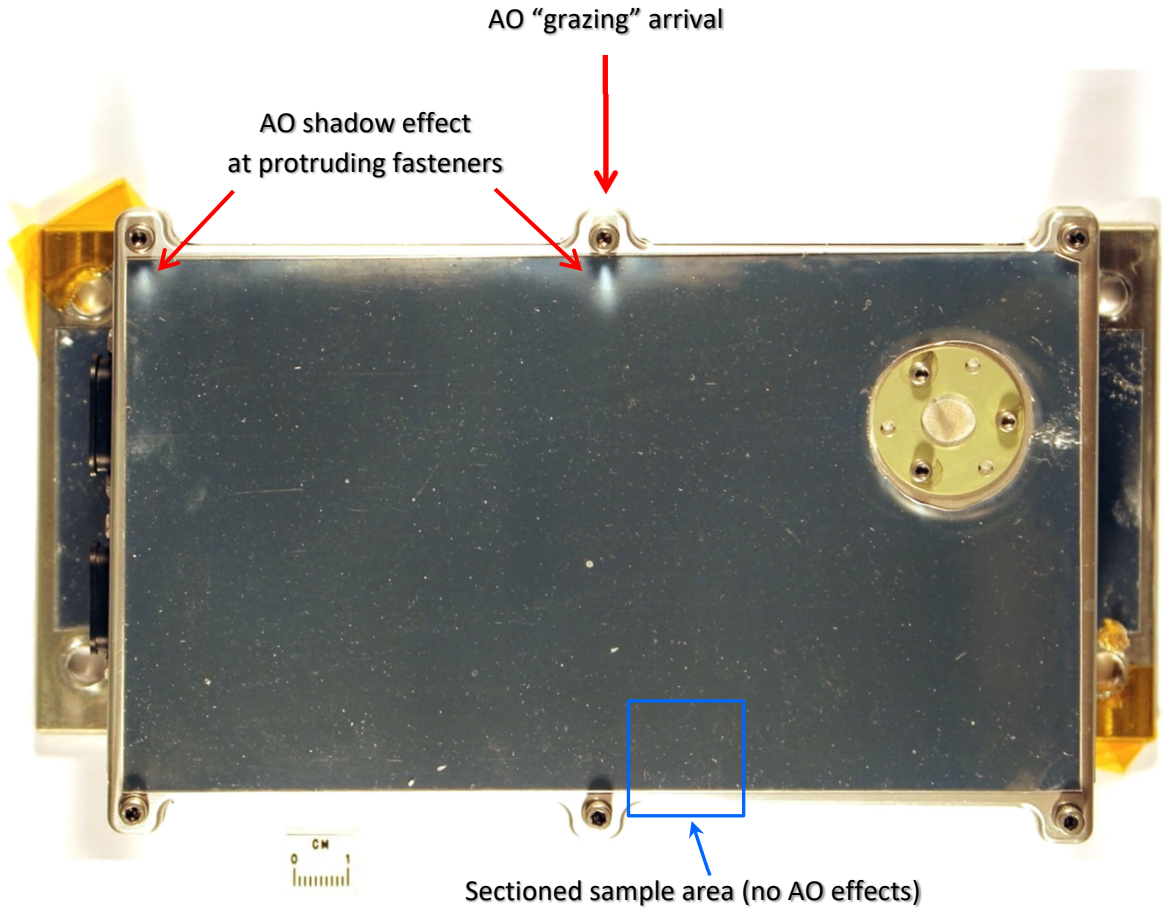


Figure 7. Post-flight photograph of the SEUXSE II Power Box showing AO effects at fasteners and the location of the MISSE 8 nadir contamination samples before sample removal.

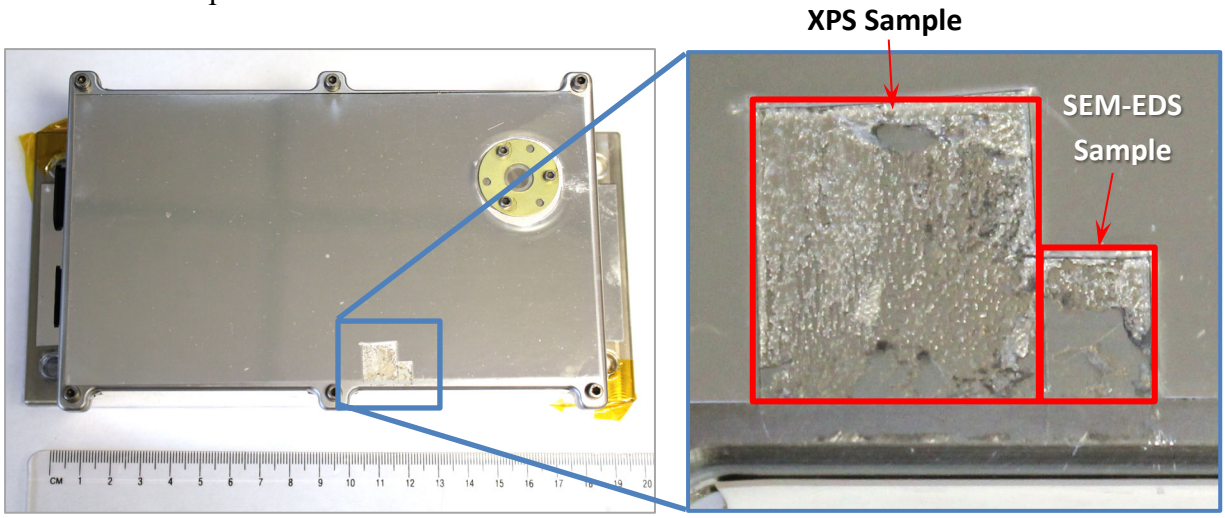


Figure 8. Location of the two sectioned SEUXSE II Power Box samples after sample removal (XPS and SEM-EDS samples).

4.0 Experimental Procedures

4.1 Optical Properties

Optical properties were obtained using a Cary 5000 UV-Vis-NIR Spectrophotometer operated with a DRA 2500 integrating sphere. Spectral total and diffuse reflectance (TR_λ and DR_λ), and spectral total and diffuse transmittance (TT_λ and DT_λ) were obtained from 250 to 2,500 nm wavelengths. Spectral specular reflectance and transmittance (SR_λ and ST_λ) were obtained by subtracting the diffuse values from the total values ($SR_\lambda = TR_\lambda - DR_\lambda$ and $ST_\lambda = TT_\lambda - DT_\lambda$, respectively).

A NASA Glenn Research Center Cary 5000 Excel® Macro was used to compute the spectral absorptance (α_λ), which was determined using the equation $\alpha_\lambda = 1 - (TR_\lambda + TT_\lambda)$. The Excel® Macro was also used to integrate each spectral curve with respect to the air mass zero (AM0) solar spectrum (the spectrum of the solar radiation outside the Earth's atmosphere, shown in Figure 9) over the spectral range (250 to 2500 nm) to obtain the total AM0 integrated values for: total reflectance (TR), diffuse reflectance (DR), specular reflectance (SR), total transmittance (TT), diffuse transmittance (DT), specular transmittance (ST) and solar absorptance (α_s) for the MISSE 8 wake flight and control samples.

4.2 Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)

Surface morphology images (250X to 20kX magnification) and corresponding chemistries were obtained using a Hitachi S-4700 II field emission scanning electron microscopy (FESEM) operated with an IXRF energy dispersive spectroscopy (EDS) system. Samples were coated with a thin platinum (Pt) coating (5 nm) to prevent charging during imaging. Images and EDS spectra were obtained with operating voltages of 6 kV, 10 kV or 15 kV. Iridium Ultra software was used to obtain and analyze the EDS spectra.

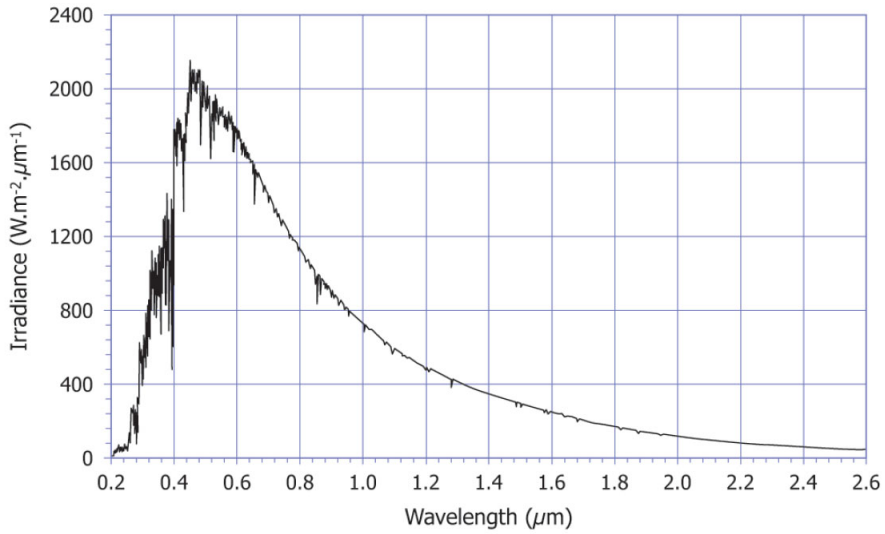


Figure 9. Air mass zero (AM0) solar spectrum irradiance.[11]

4.3 X-ray Photoelectron Spectroscopy (XPS) Analyses

X-ray Photoelectron Spectroscopy is a surface sensitive technique that allows for the identification and quantification of elements and trace contaminants found on a surface. Analysis depth is between 3 to 5 nm (30 to 50 Å) and detection limits are parts per thousand. XPS is sensitive to the electronic environment of each atom, allowing for chemical state information to be obtained about any elements detected. It is also possible to evaluate deeper areas below a surface contamination layer using depth profiling. In this case, an argon ion gun is used to generate ions and information is obtained by alternating sputtering and measurement.

X-ray Photoelectron Spectroscopy measurements were performed on a PHI 5000 Versaprobe (ULVAC-PHI) using monochromatic microfocused Al x-rays (200 μm, 43.4 W) with a photoelectron takeoff angle of 45°. Survey scans using a 117.4 eV pass energy were initially taken to identify all components, followed by higher resolution individual region scans using a pass energy of 58.7 eV. Atomic concentrations were calculated based on the individual peaks. Sputtering was then done on each sample using a 3 kV Ar+ beam to remove 10 nm of material. For the control samples, this was done to remove any adventitious carbon (C) that comes from normal air exposure. For the actual flight samples, sputtering was done in 1-minute increments (removing 10 nm) until a clean substrate surface was obtained. This way it was possible to determine the thickness of the contamination layer on the flight exposed surfaces.

5.0 MISSE 8 Atomic Oxygen Fluence and Solar Exposure

The MISSE 8 wake AO fluence was determined to be 8.80 x 10^{19} atoms/cm^2 based on Kapton H dehydrated mass loss measurements made by de Groh for a beveled tray sample flown as part of the MISSE 8 Polymers Experiments.[9] Thus, the ORMatE-III R/W wake surface received some AO exposure during the MISSE 8 mission. The nadir AO fluence was determined to be 3.6 ± 0.1 x 10^{19} atoms/cm^2 based on mass and thickness loss measurements of Kapton HN made by Finckenor of NASA Marshall Space Flight Center (MSFC) using beveled tray samples.[12] This fluence is very close to that for the zenith beveled tray (4.04 x 10^{19} atoms/cm^2), as would be expected as both surfaces receive grazing AO exposure.[9] It should be noted that the AO fluence for a zenith facing Kapton H sample mounted in a thin aluminum foil holder (versus a standard MISSE beveled tray) was found by de Groh to have a significantly higher AO fluence (1.96 x 10^{20} atoms/cm^2) than the recessed zenith sample.[9] This would be expected to be true for the nadir samples. Hence, the AO fluence to nadir surfaces that receive gazing AO arrival is dependence on mounting and shadowing. A summary of the MISSE 8 AO fluence values is provided in Table 1.

Table 2 provides the solar exposures in equivalent sun hours (ESH) for MISSE 8. The MISSE 8 ram and wake solar exposures were approximated using the ratios of the MISSE 7 ram and wake to zenith ratios, respectively, because MISSE 8 was flown in the same location on ISS as MISSE 7 (ELC-2 Site 3).[9] The MISSE 8 nadir solar exposure was estimated at NASA MSFC based on Teflon erosion.[12] Computations of the zenith solar exposure have been conducted by the Naval Research Laboratory (NRL) and were determined to be 6,100 ± 1,000 ESH.[13]

Table 1. MISSE 8 Kapton Atomic Oxygen Fluence Determination[9,12]

MISSE 8 Orientation	Sample ID	Holder Style	Mass Loss (g)	Surface Area (cm^2)	Density (g/cm^3)	MISSE 8 Fluence ($atom/cm^2$)
Ram	M8-R1 Kapton H	Beveled Tray	0.079104	3.995	1.4273	4.62E+21
Wake	M8-W1 Kapton H	Beveled Tray	0.001523	4.041	1.4273	8.80E+19
Nadir	MSFC Kapton HN	Beveled Tray	----------	-------	--------	3.6 ± 0.1E+19
Zenith	M8-Z1B Kapton H	Beveled Tray	0.000670	3.877	1.4273	4.04E+19
Zenith	M8-1 Kapton H	Thin Al Foil	0.002330	2.782	1.4273	1.96E+20

Table 2. MISSE 8 Ram and Wake Solar Exposure Approximations[9,12,13]

Flight Orientation	MISSE 7 Exposure (Years)	MISSE 7 Solar Exposure (ESH)	MISSE 7 ESH Relative to Zenith	MISSE 8 (ESH)
Zenith	2.14	4,300	1	6,100 ± 1,000
Ram	2.00	2,400	0.56	3,200*
Wake	2.00	2,000	0.47	2,700*
Nadir	2.14	<<2,000	-----	800 ± 300

*Ram and wake also multiplied by the ram-wake to zenith duration ratio of 2.00/2.14 (0.93)

6.0 Results and Discussion

6.1 MISSE 8 Wake Flight Sample—Optical Properties

Post-flight photographs of the wake FEP flight (M8-W11) and control (M8-R9-B) samples are provided in Figures 10 and 11. As mentioned previously, the MISSE-8 wake flight samples were exposed to a low AO fluence of 8.80 x 10^{19} atoms/cm^2 and approximately 2,700 ESH. The flight sample is slightly wavy as compared to the control sample. It also has a few hazy regions, which appear a little darker than the rest of the sample (or the control sample) in the photographs in Figures 10 and 11. The optical microscope image on the right side of Figure 11 shows the hazy region of the flight sample contains particulate contamination. The AO E_y of the clear FEP wake sample was determined to be 1.08 x 10^{-24} cm^3/atom based on mass loss of 827 mg.[9]

Figures 12 to 18 provide the TR, DR, SR, TT, DT, ST and α_S spectral curves for the flight (M8-W11) and control (M8-R9-B) samples, respectively. As can be seen in Figures 12 to 14, there was insignificant change in the TR spectra but there was an observable increase in the DR, and decrease in the SR, across all wavelengths of the flight samples as compared to the control sample. The TT of the flight sample decreased most notably in the UV region of the spectra, as shown in Figure 15. Whereas the DT increased, and the ST decreased, across most wavelengths, as shown in Figures 16 and 17 respectively. The diffuse reflectance and transmittance increase is likely contributed to by the hazy regions. Table 3 provides a summary of the AM0 integrated optical properties for the MISSE 8 wake flight and control samples. Overall, the α_s of the flight sample increased 0.008 as compared to the control sample, mostly in the UV region (see Figure 18).

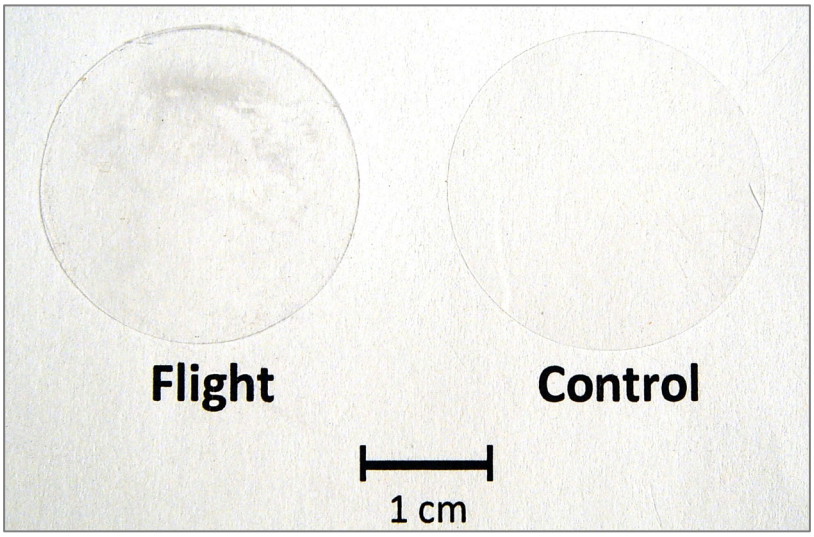


Figure 10. Post-flight photograph of the MISSE 8 wake FEP flight (M8-W11) and corresponding control (M8-R9-B) samples.

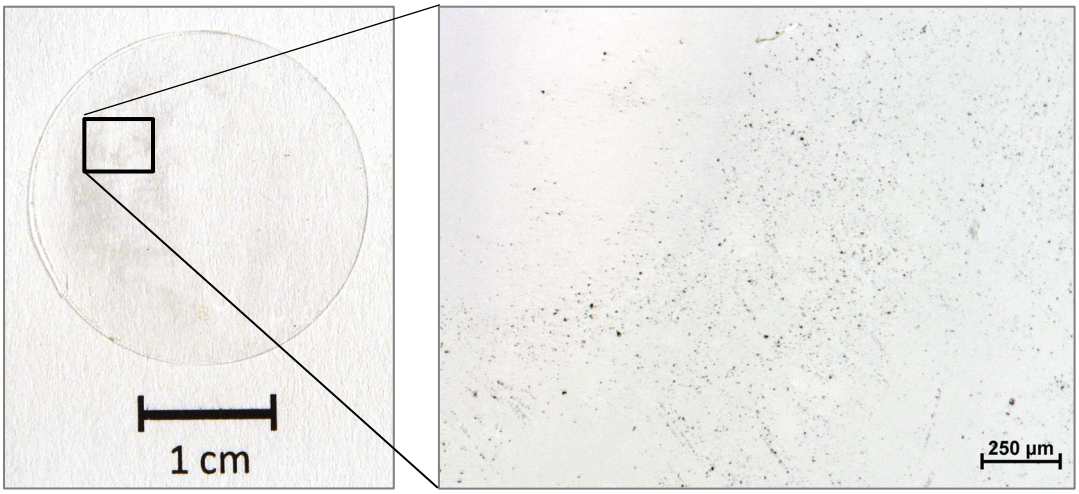


Figure 11. Post-flight photo of the wake FEP flight sample (M8-W11) with a close-up optical microscope image of the particulate-rich hazy area.

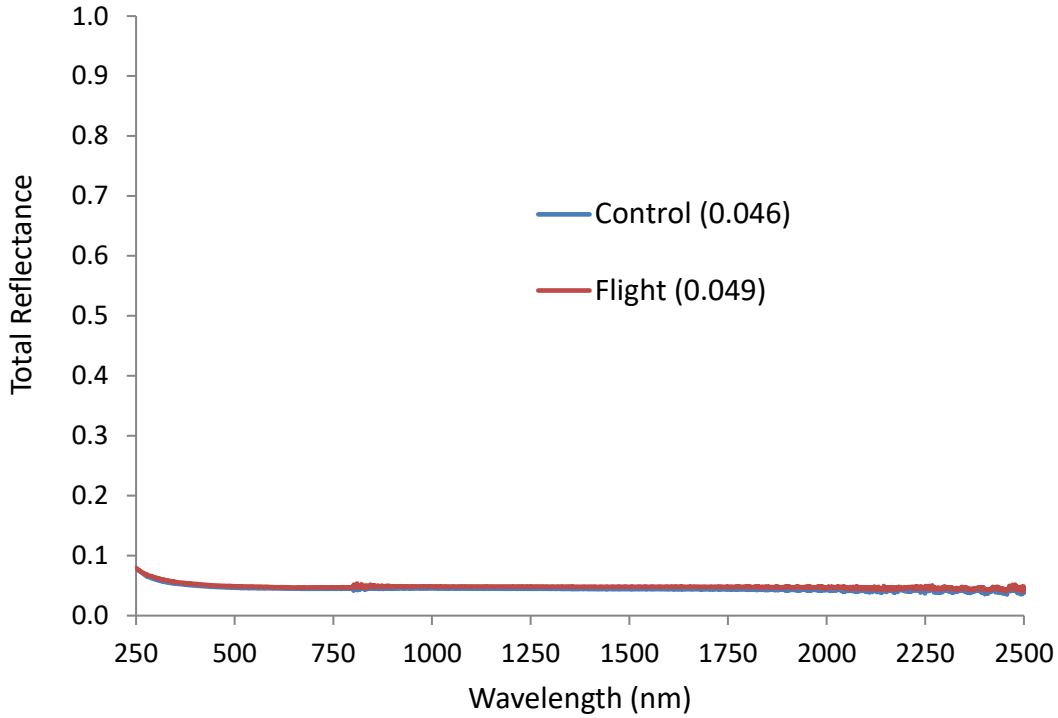


Figure 12. Total reflectance spectra for the MISSE-8 wake Teflon FEP flight (M8-W11) and control (M8-R9-B) samples.

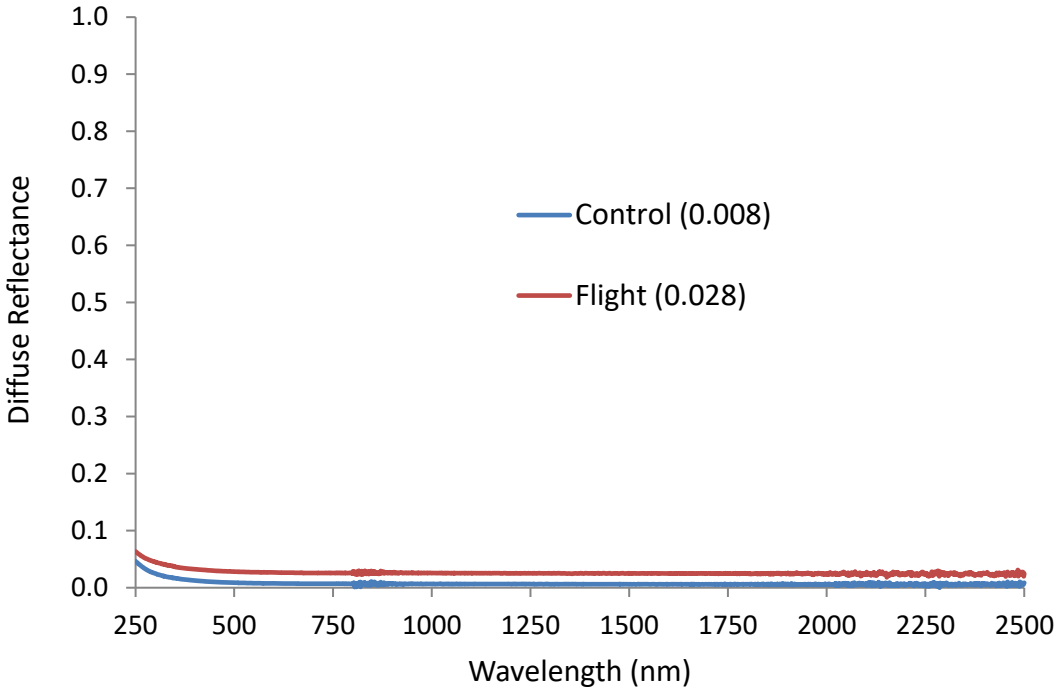


Figure 13. Diffuse reflectance spectra for the MISSE-8 wake Teflon FEP flight (M8-W11) and control (M8-R9-B) samples.

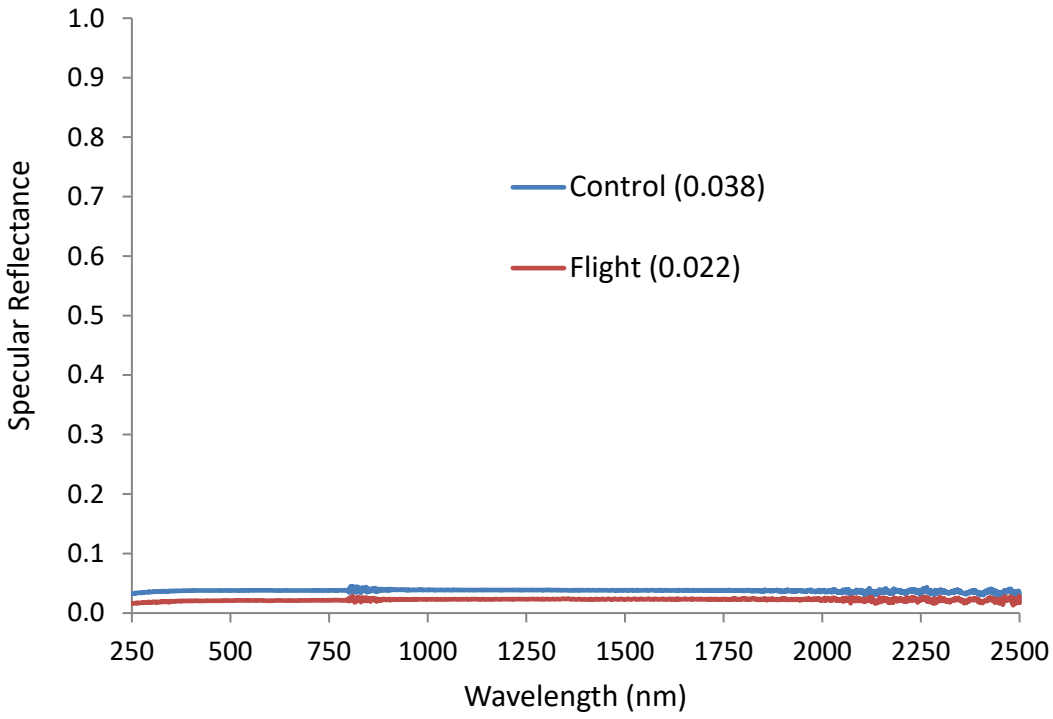


Figure 14. Specular reflectance spectra for the MISSE-8 wake Teflon FEP flight (M8-W11) and control (M8-R9-B) samples.

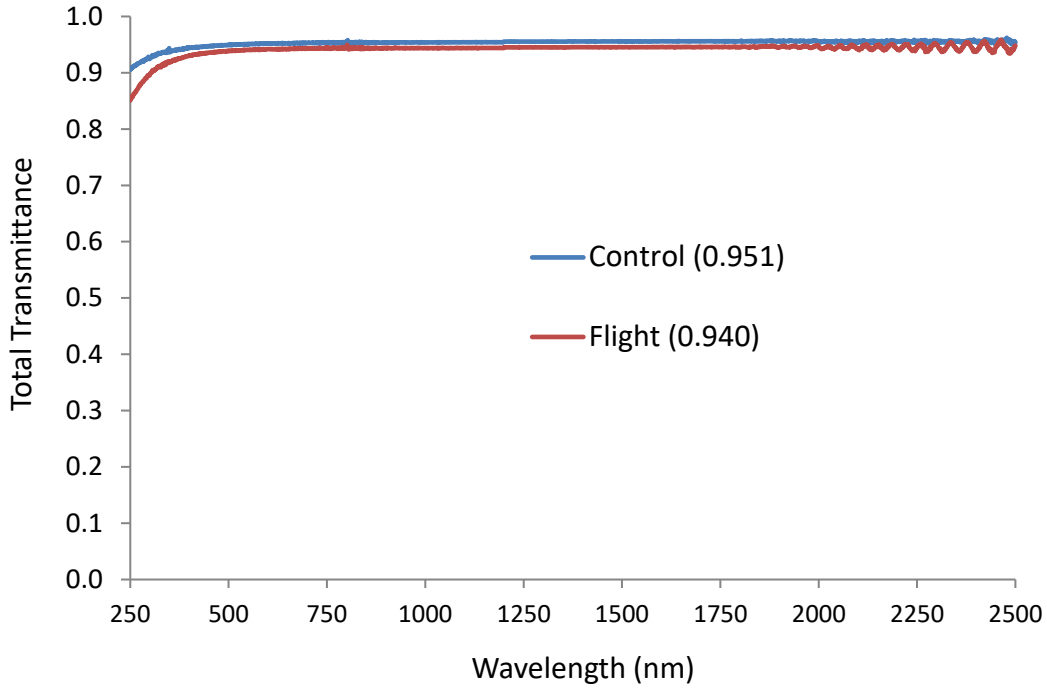


Figure 15. Total transmittance spectra for the MISSE-8 wake Teflon FEP flight (M8-W11) and control (M8-R9-B) samples.

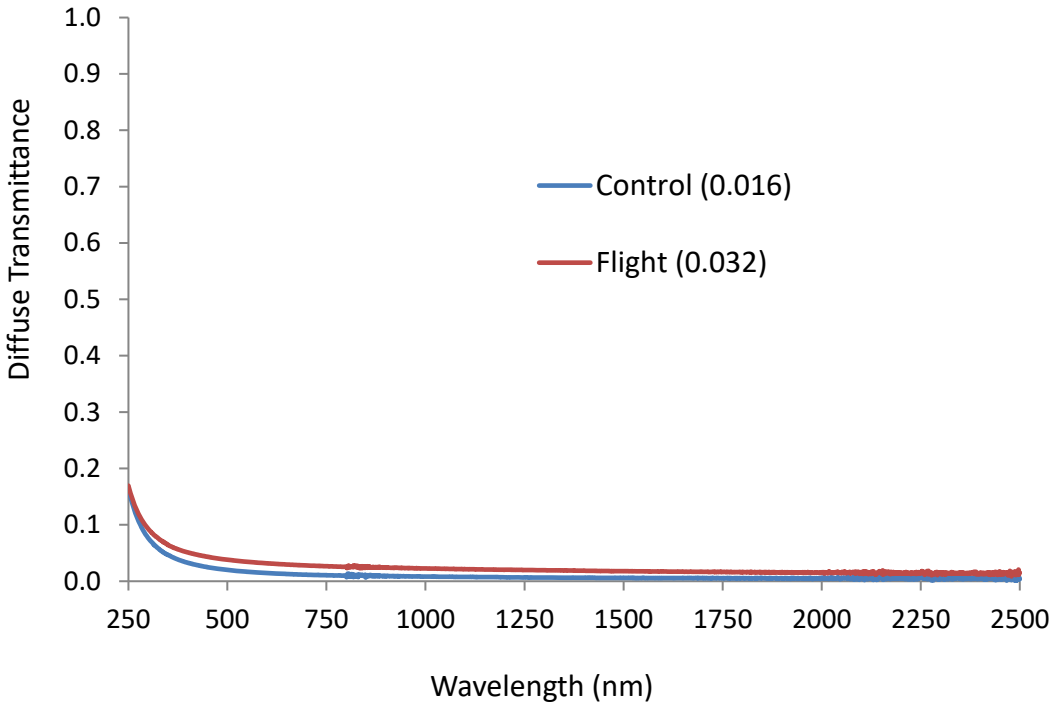


Figure 16. Diffuse transmittance spectra for the MISSE-8 wake Teflon FEP flight (M8-W11) and control (M8-R9-B) samples.

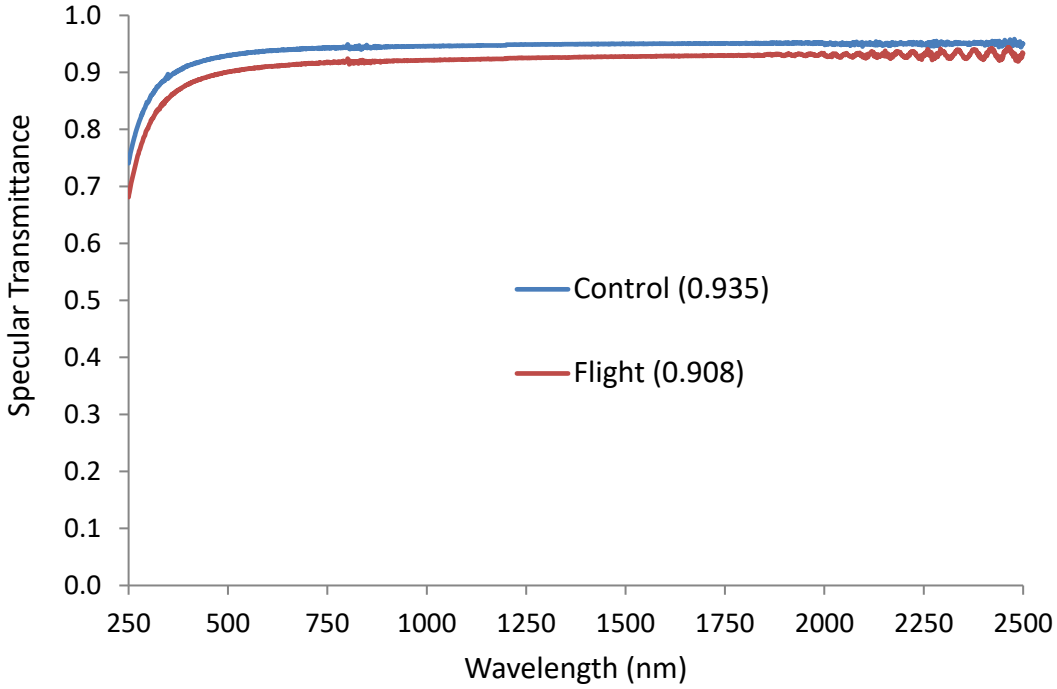


Figure 17. Specular transmittance spectra for the MISSE-8 wake Teflon FEP flight (M8-W11) and control (M8-R9-B) samples.

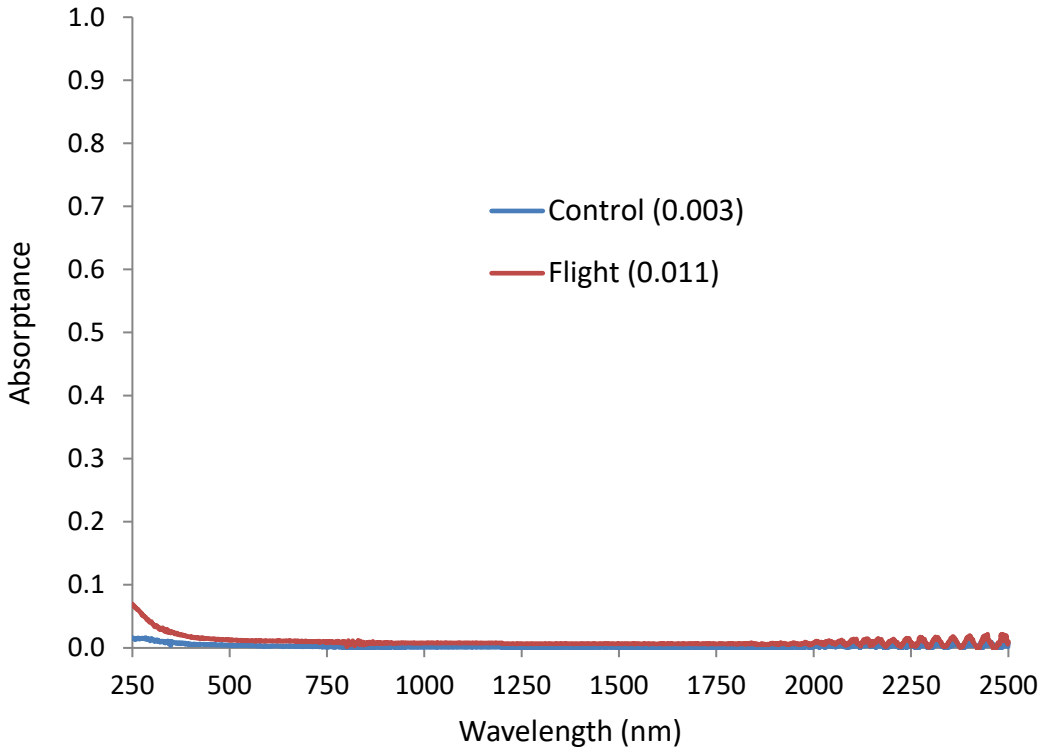


Figure 18. Absorptance spectra for the MISSE-8 wake Teflon FEP flight (M8-W11) and control (M8-R9-B) samples.

Table 3. Optical Properties for MISSE 8 Wake Flight and Control Samples

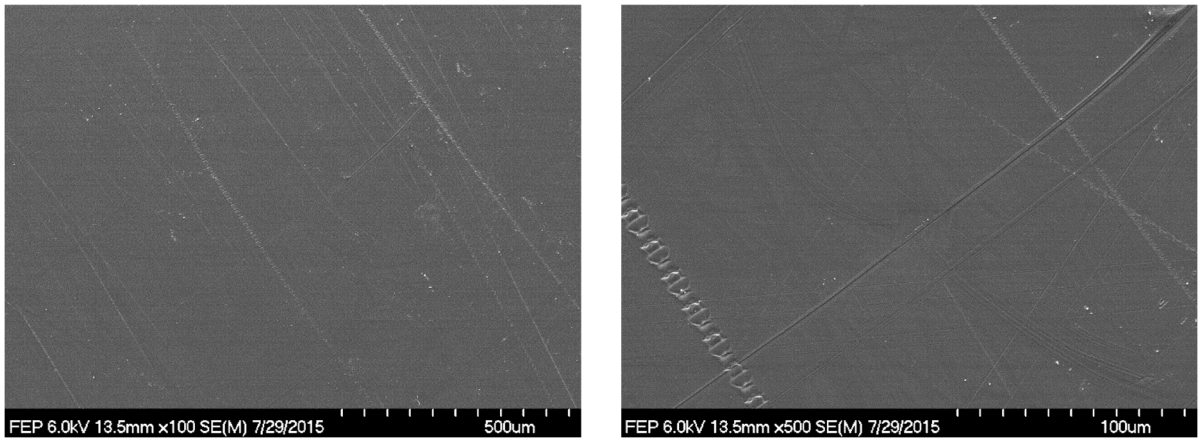
MISSE Flight Sample	Material	Flight or Control	TR	DR	SR	TT	DT	ST	α_s	$\Delta \alpha_s$
M8-W11	FEP	Flight	0.049	0.028	0.022	0.940	0.032	0.908	0.011	0.008
		Control	0.046	0.008	0.038	0.951	0.016	0.935	0.003	

6.2 MISSE 8 Wake Control Sample—FESEM, EDS, and XPS Analyses

Figures 19 and 20 provide FESEM images and corresponding EDS area analysis spectra, respectively, for the MISSE 8 wake Teflon FEP control sample (M8-R9-B). The FESEM images in Figure 19 show a very smooth surface with some lines that are likely roll processing lines. The EDS spectra in Figure 20 show fluorine (F) and C peaks for FEP and the Pt peak for the applied conductive coating. The EDS spectra for image area FEP-01 (Figure 19a) taken at 6 kV also shows a very small oxygen (O) peak, which is not seen in the 15 kV EDS spectra (Figure 19b). Figure 21 provides XPS spectra the MISSE 8 wake control sample (M8-R9-B). The XPS spectra corresponds with the EDS data. The surface atomic concentration (at.%) for F was 67.04 at.% and for C was 32.8 at.%. The control sample also had a small amount of O (0.09 at.%).

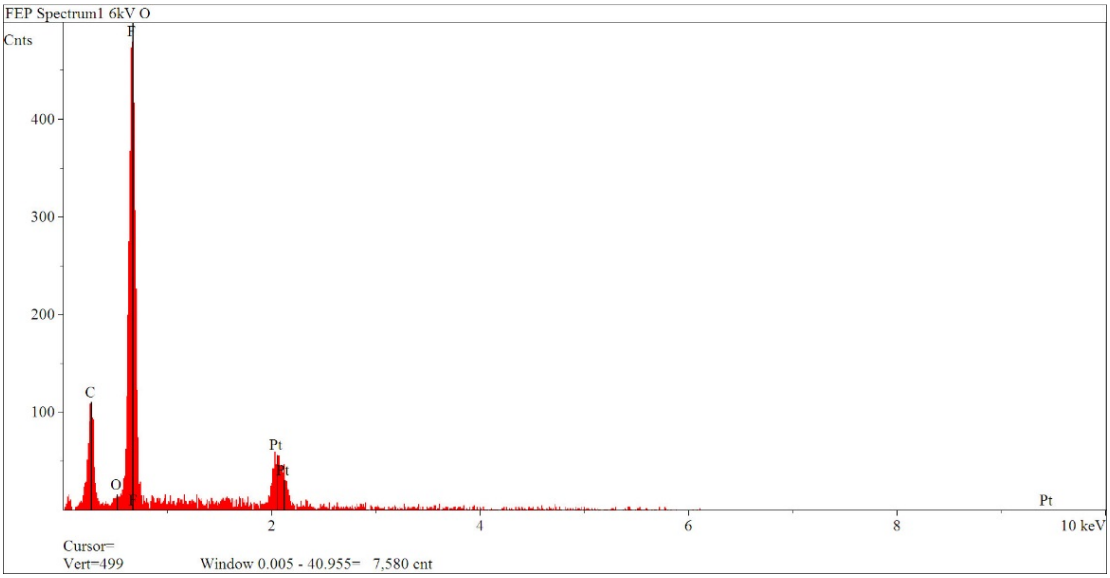
6.3 MISSE 8 Wake Flight Sample—FESEM, EDS, and XPS Analyses

Figures 22 and 23 provide FESEM images and corresponding EDS area analysis spectra, respectively, for the MISSE 8 wake flight sample (M8-W11) in "cleaner" space exposed regions. The FESEM images show a smooth surface with a few bright particles. The EDS spectra show the F and C peaks for FEP and the Pt peak for the conductive coating that was applied. The EDS spectra also show very small aluminum (Al) and O peaks, which are believed to be associated with the particles. Figure 24 provides XPS spectra of the space exposed area of the wake flight sample in a cleaner region. The XPS spectra corresponds with the EDS data. The surface atomic concentration for F was 64.65 at.% and for C was 33.55 at.%. These are slightly less than the F and C atomic concentrations for the FEP control sample (67.04 and 32.88 at.%, respectively). The cleaner region of the flight sample also had small peaks for O (1.30 at.%) and Al (0.49 at.%). An insignificant amount of silica (Si) was also detected (0.02 at.%). The at.% of O increased from 0.09 to 1.30 at.%, so oxidation of the flight sample has occurred. And, the flight sample has Al, likely from the particles. The EDS and XPS spectra do not provide evidence of Si (i.e. silicone) molecular contamination on the flight sample.

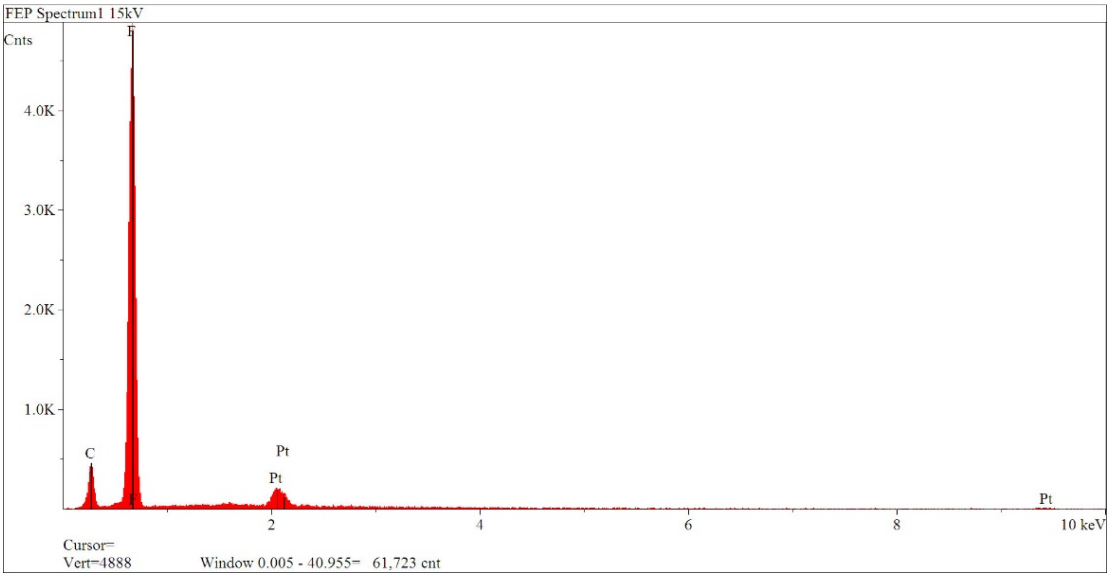


a. b.

Figure 19. FESEM images of the MISSE 8 wake FEP control sample (M8-R9-B): a). Image FEP-01 taken at 100X and 0° tilt, b). Image FEP-02 taken at 500X and 0° tilt.



a.



b.

Figure 20. EDS area analysis spectra of the FEP control sample from: a). FESEM image area in Figure 19a taken at 6 kV, and b). FESEM image area in Figure 19a taken at 15 kV.

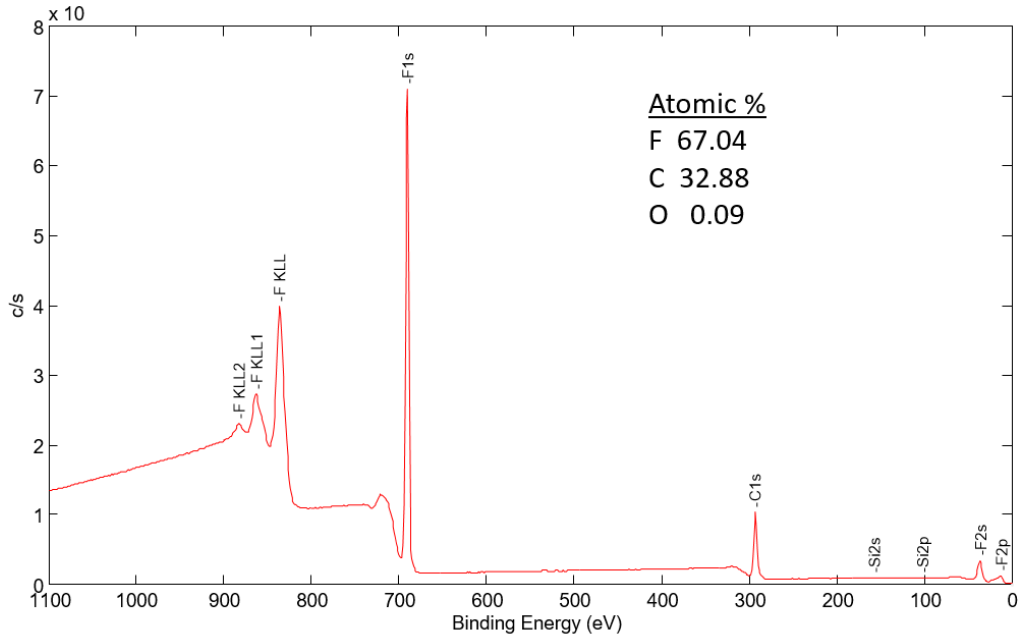
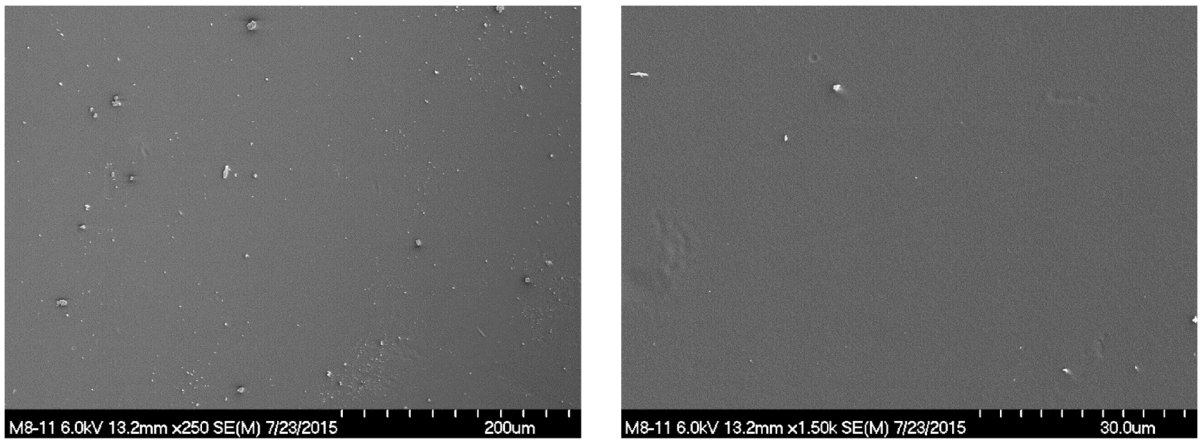
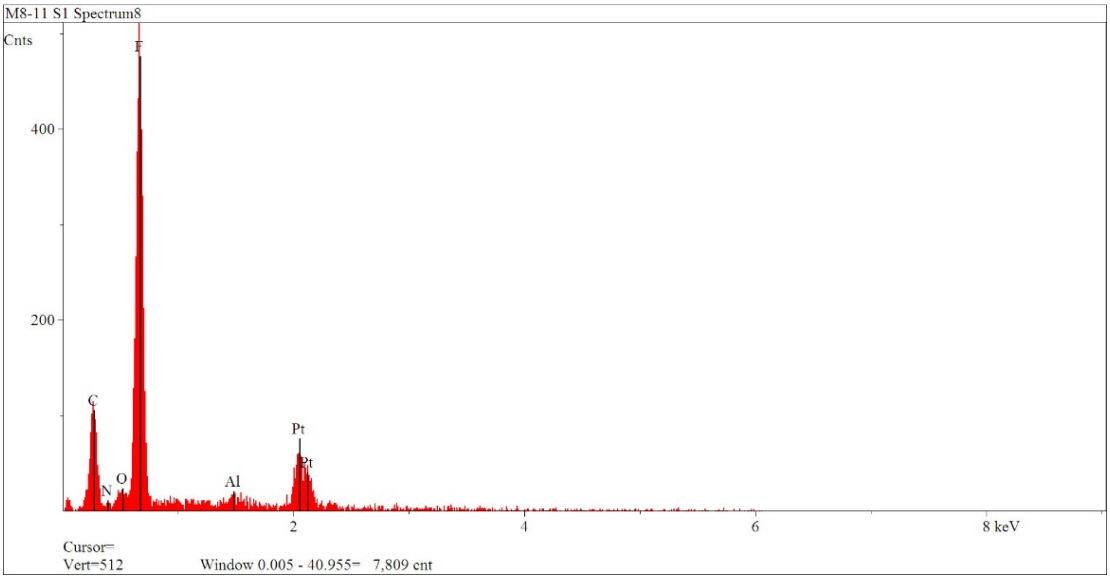


Figure 21. The XPS binding energy data for the FEP control sample (M8-R9-8).

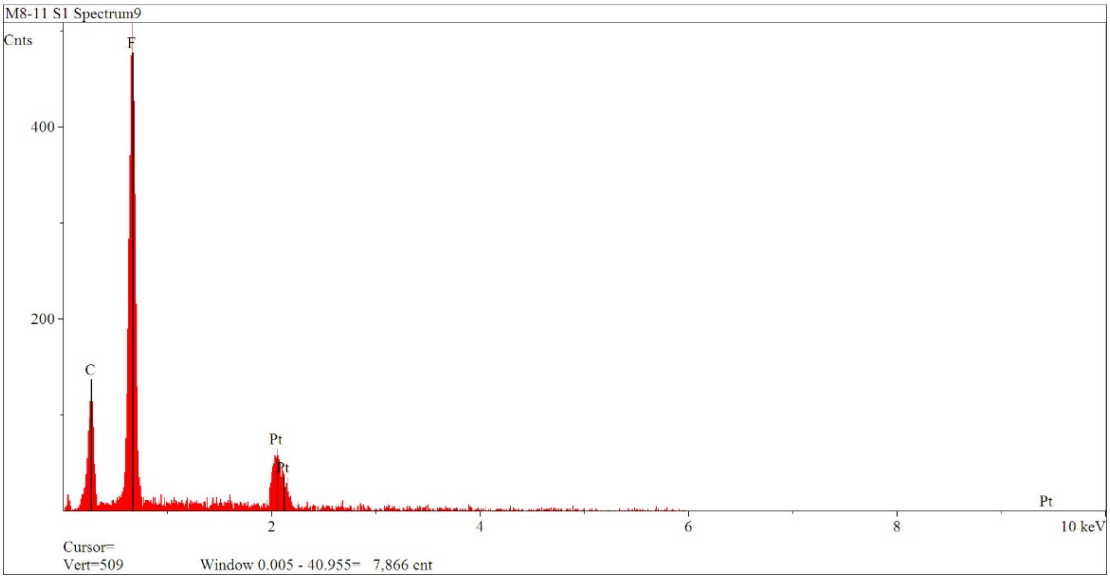


a. b.

Figure 22. FESEM images of the MISSE 8 wake flight sample (M8-W11) in space exposed "cleaner" regions: a). Image S1-06 taken at 250X and 0° tilt, b). Image S1-07 taken at 1.5kX and 0° tilt.



a.



b.

Figure 23. EDS area analysis spectra of M8-W11 in cleaner regions from: a). FESEM image area in Figure 22a (6 kV), and b). FESEM image area in Figure 22b (6 kV).

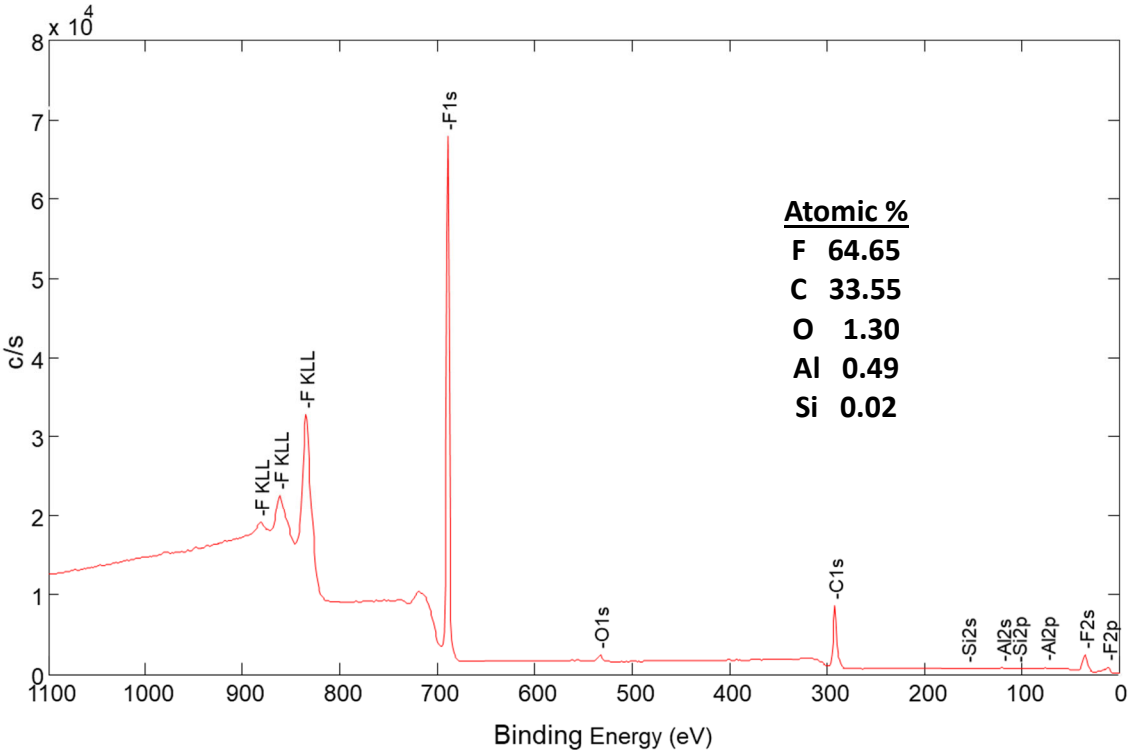
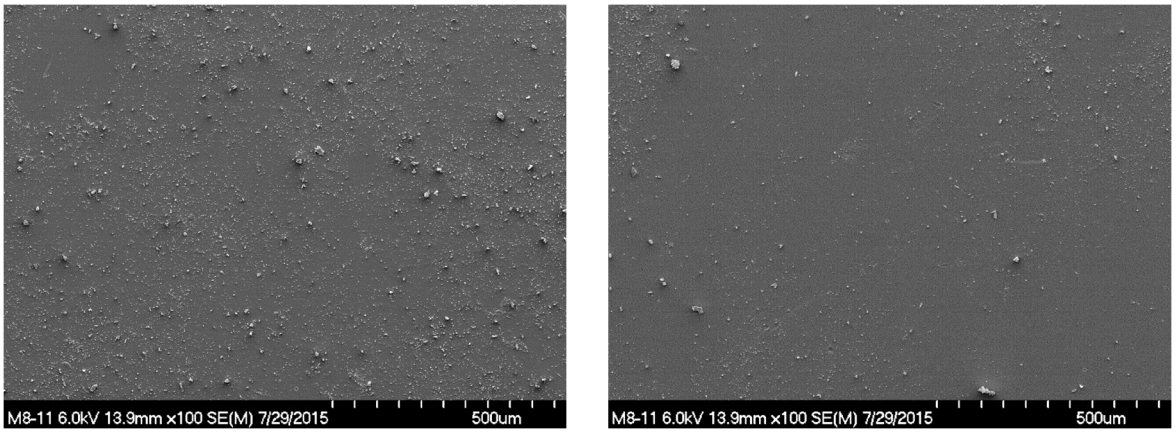


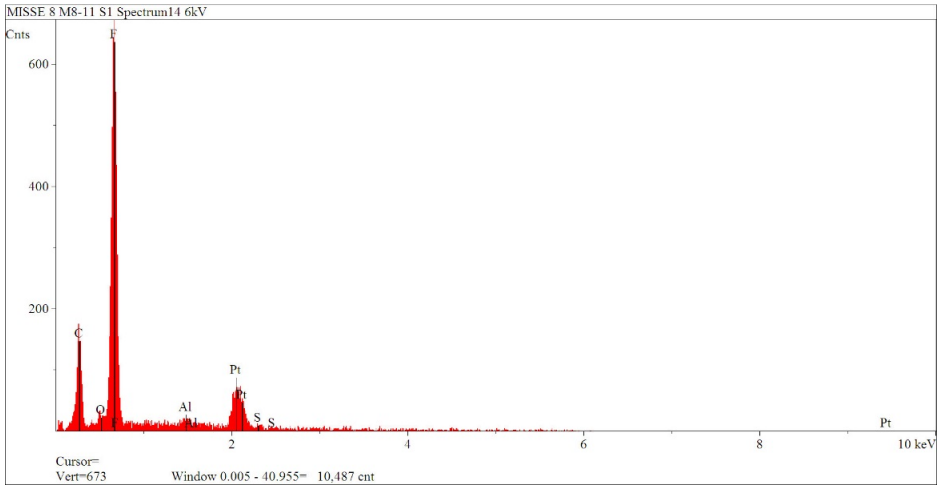
Figure 24. The XPS binding energy data for the MISSE 8 wake flight sample (M8-W11) in a "cleaner" space exposed region.

Figures 25 and 26 provide FESEM images and corresponding EDS area analysis spectra, respectively, for the MISSE 8 wake flight sample (M8-W11) in "hazy" space exposed regions. The FESEM images show more small particles than seen in the cleaner regions shown in Figure 22. The EDS spectra show the F and C peaks for FEP and the Pt peak for the conductive coating that was applied. The EDS spectra also show very small Al, O and sulfur (S) peaks. No additional elements were identified using the higher accelerating voltage of 15 kV, as shown in Figure 26b, and the very small S peak was not present at 15 kV. Figure 27 provides XPS spectra of the space exposed area of the wake flight sample in a hazy region. The XPS spectra corresponds with the EDS data. The surface atomic concentration for F was 60.88 at.% (down from 64.65 at.% in the clean region) and for C was 38.36 at.% (up from 33.55 at.% in the clean region). There were also small peaks for O (0.61 vs. 1.30 at.%) and Al (0.11 vs. 0.49 at.%). And, insignificant amount of Si (0.02 at.%) and lead (Pb) (0.02 at.%) were also detected. Thus, the hazy region had fairly significant increase in C (38.36 vs. 32.88 at.% for the control sample) and decrease in F (60.88 vs. 67.04 at. % for the control sample). The EDS and XPS spectra do not provide evidence of Si (i.e., silicone) molecular contamination on the flight sample.

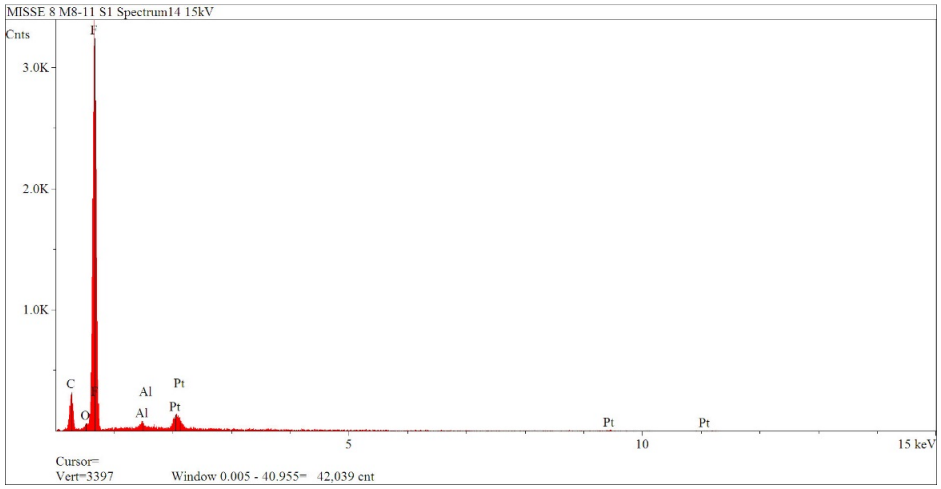


a. b.

Figure 25. FESEM images of the MISSE 8 wake flight sample (M8-W11) in space exposed "hazy" regions: a). Image (S1-14) taken at 100X and 0° tilt, b). Image (S1-15) taken at 100X and 0° tilt.



a.



b.

Figure 26. EDS area analysis spectra of M8-W11 in hazy regions from: a). FESEM image area in Figure 25a taken at 6 kV, and b). FESEM image area in Figure 25a taken at 15 kV.

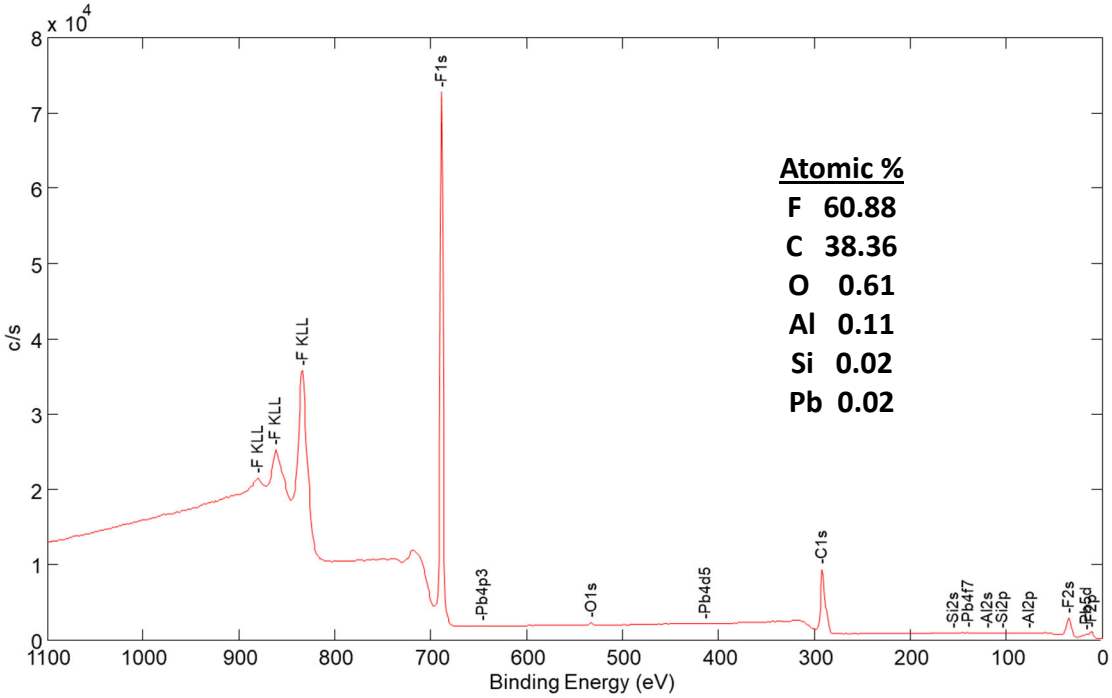
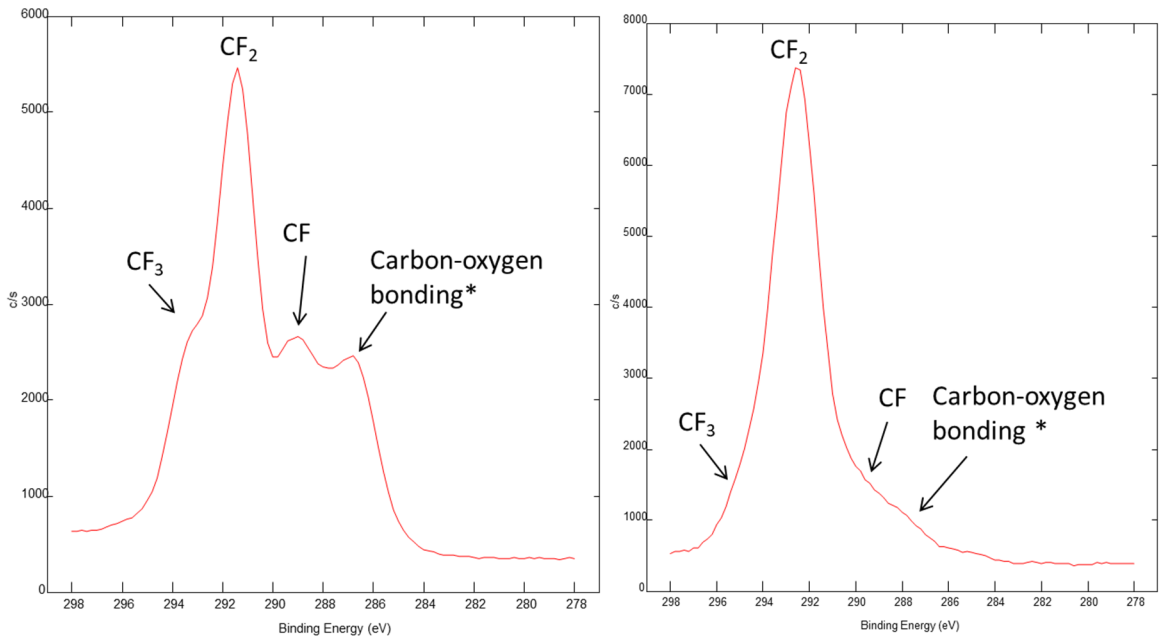


Figure 27. The XPS binding energy data for the wake flight sample (M8-W11) in a hazy space exposed region.

The XPS analyses of the MISSE 8 wake FEP flight sample (M8-W11) also indicated visible (optical camera) evidence of erosion. Figure 28 provides expanded XPS C binding energy spectra for the MISSE 8 wake flight sample space exposure surface (Figure 28a) and the control sample (Figure 28b). There could be some degradation of the polymer C-O bonding caused by x-ray irradiation during analysis. This is highlighted with an asterisk (*) in the two graphs shown in Figure 28. The comparisons also show that the flight sample has more degradation, which is most likely caused by AO oxidation.

Figure 29 provides FESEM images of the space exposed surface of the MISSE 8 wake flight sample (M8-W11) including a higher magnificent image of the two central particles. EDS spot analyses were conducted on each of the two particles at the locations indicated with a plus sign, "+". The corresponding EDS spot analysis spectra for the particles are provided in Figures 30 and 31. The EDS spectra for both particles show large peaks of A1 and O, along with small peaks of zinc (Zn) and possibly magnesium (Mg). Small amounts of F and C from the substrate are also present.



a. b.

Figure 28. Expanded C regions of the XPS binding energy data for: a). MISSE 8 wake FEP flight sample (M8-W11), and b). Control FEP sample (M8-R9-B) surface.

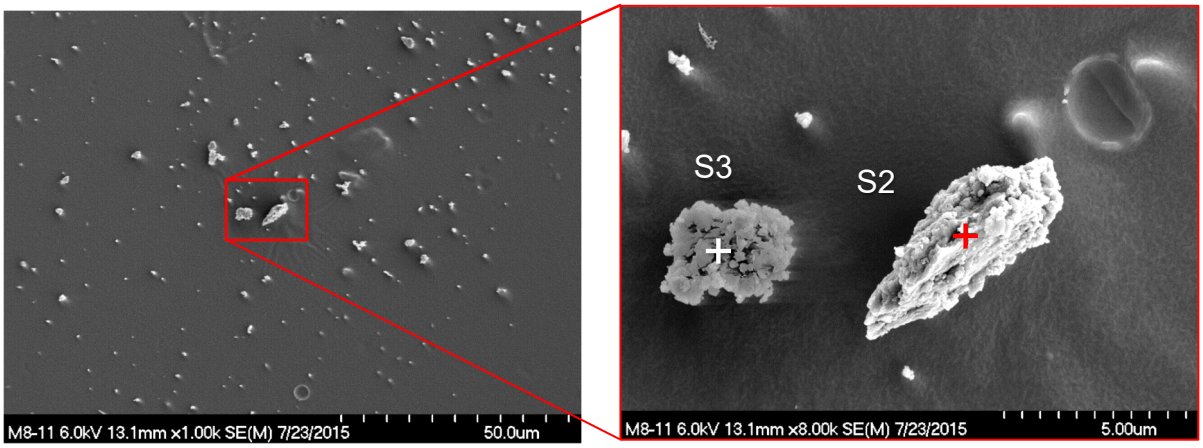


Figure 29. FESEM image of the space exposed area of the MISSE 8 wake flight sample (M8-W11) with a higher magnification image (right) showing the spot analysis locations (S2 and S3) of the central two particles.

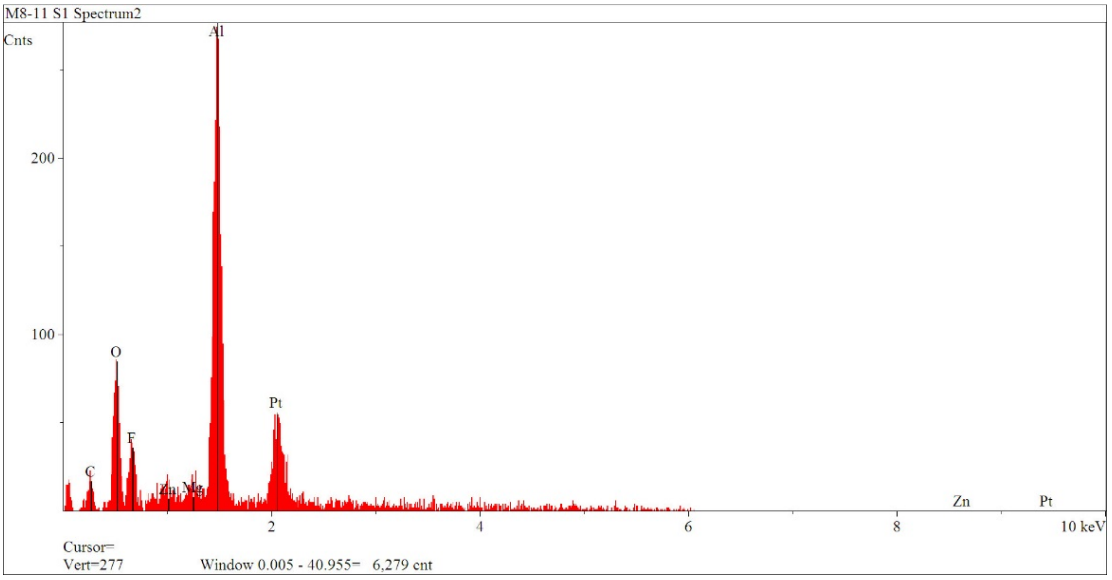


Figure 30. EDS spot analysis spectra (S2) of the particle on the right in the higher magnification image in Figure 29.

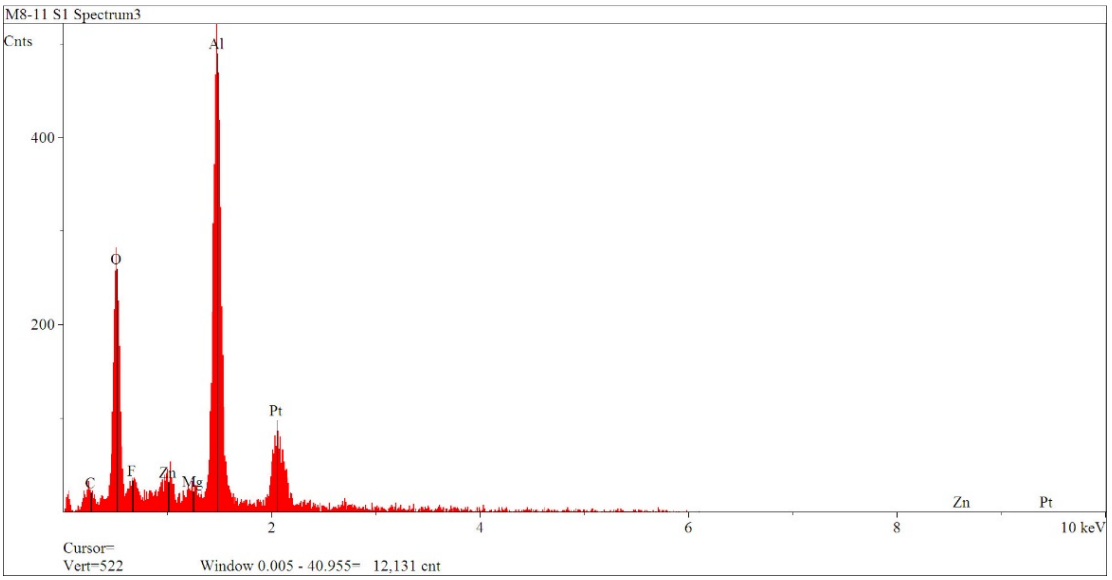


Figure 31. EDS spot analysis spectra (S3) of the particle on the left in the higher magnification image in Figure 29.

Figures 32 to 41 provide FESEM images and corresponding EDS spectra of particles on the space exposed region of the MISSE 8 wake flight sample. EDS spot analyses were conducted at higher magnifications (1 to 15 kX) and indicate the particles are Al and O rich with small peaks of Zn and possibly Mg. A few particles also have very small S peaks.

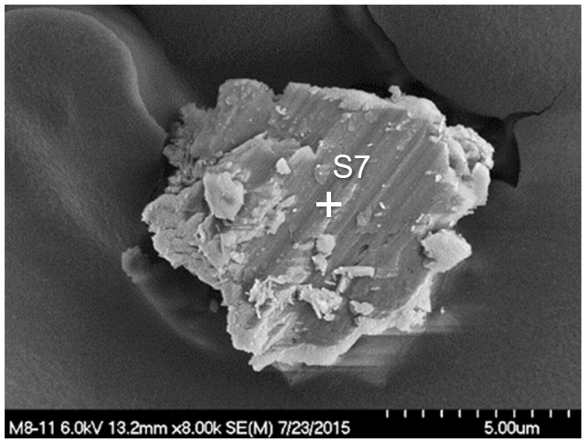


Figure 32. FESEM image of a particle on the space exposed surface of the MISSE 8 wake flight sample (M8-W11) with the location identified for the EDS spot analysis (S7).

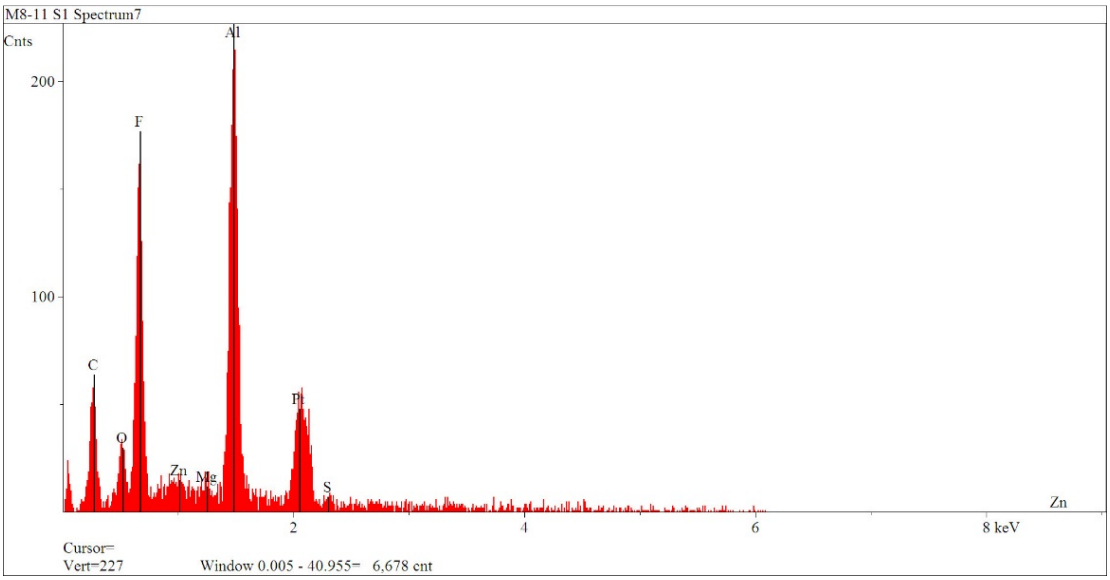


Figure 33. EDS spot analysis spectra (S7) of the particle shown in Figure 32 with Al, O, Zn and Mg peaks in addition to F, C and Pt (conductive coating).

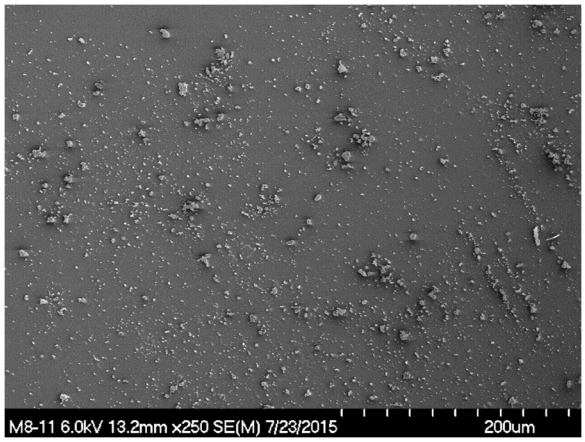


Figure 34. FESEM image of the space exposed surface of the wake flight sample (M8-W11).

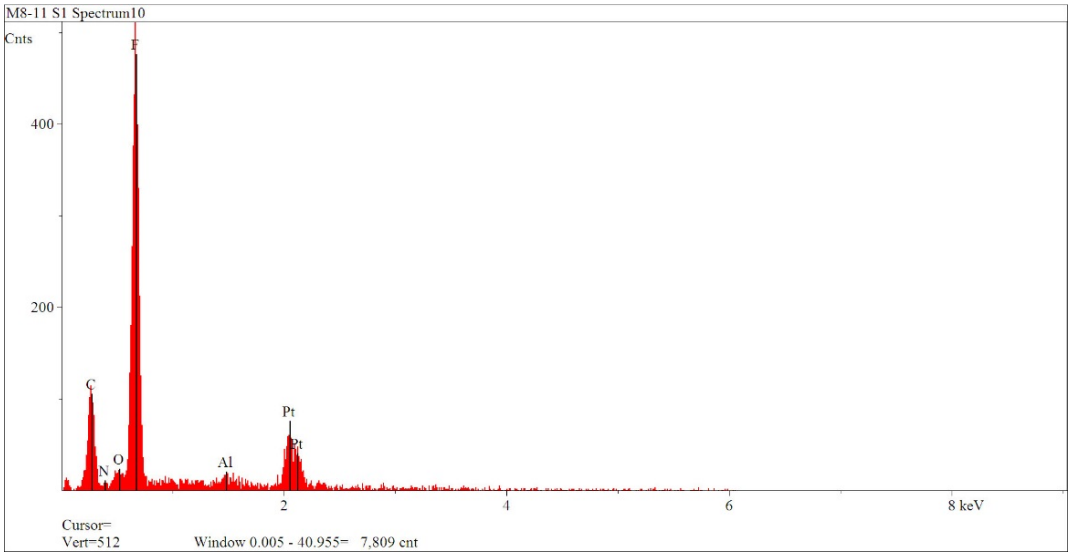


Figure 35. EDS area analysis spectra (S10) of the wake flight sample (M8-W11) for the area shown in Figure 34 with Al and O in addition to F, C and Pt (conductive coating).

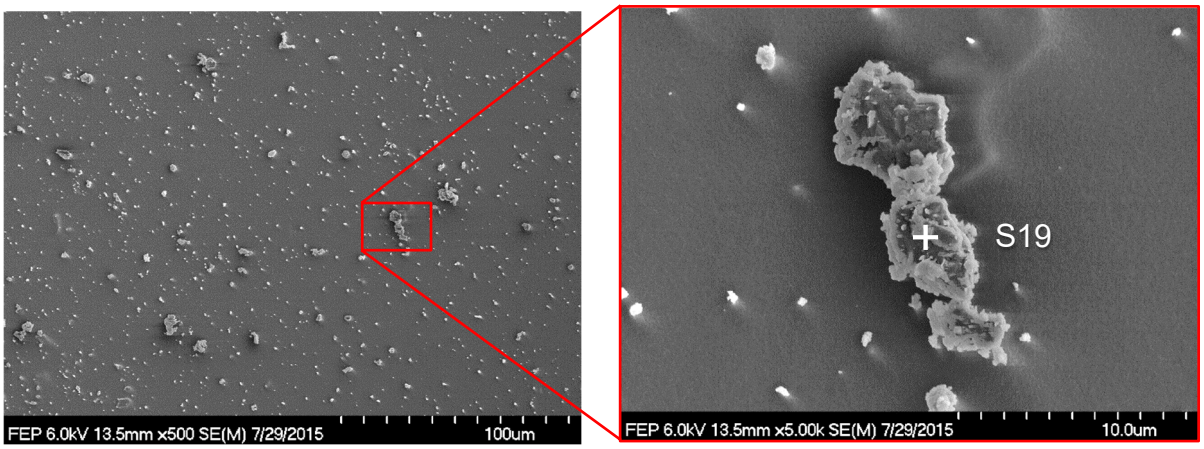
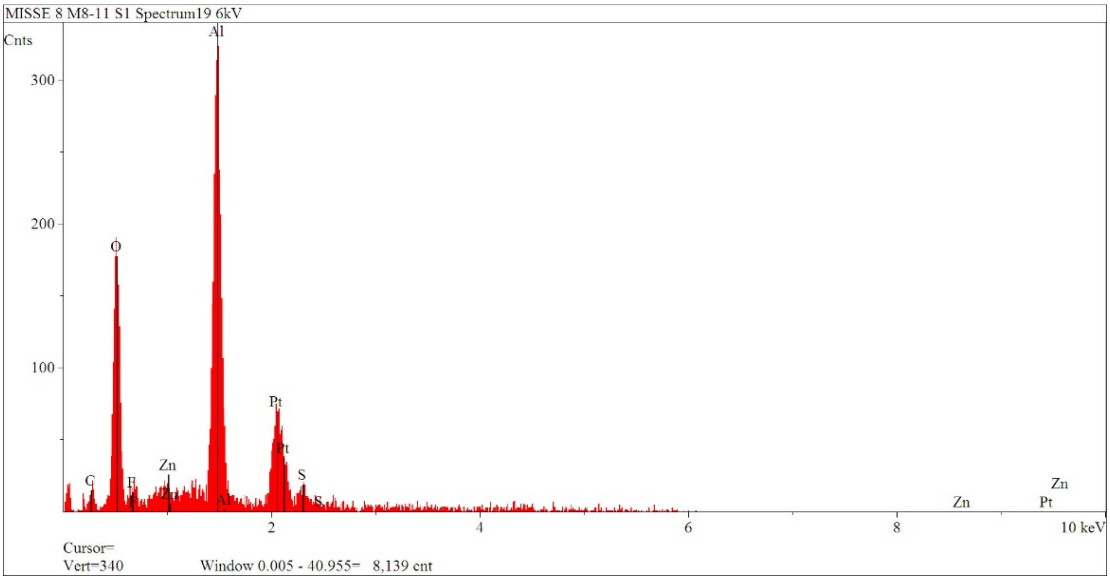
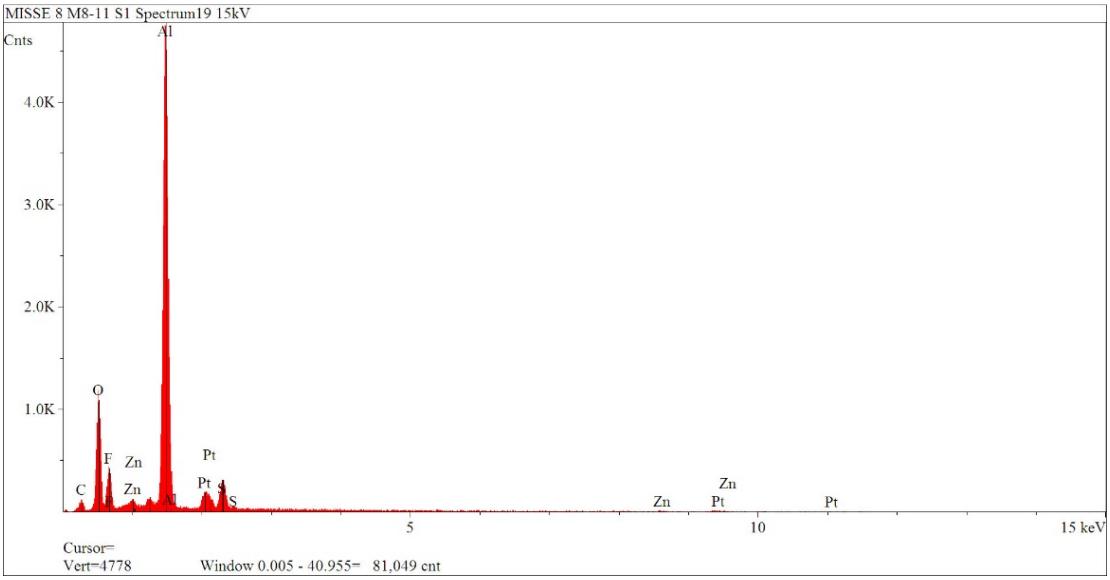


Figure 36. FESEM image of the space exposed area of the wake flight sample (M8-W11) with a higher magnification image (right) showing the spot analysis location (S19) of one of the particles.



a.



b.

Figure 37. EDS spot analysis spectra (S19) of the particle shown in Figure 36: a). EDS collected at 6 kV, and b). EDS spectra collected at 15 kV. The EDS spectra show peaks of Al, O, Zn and S in addition to F, C and Pt (conductive coating).

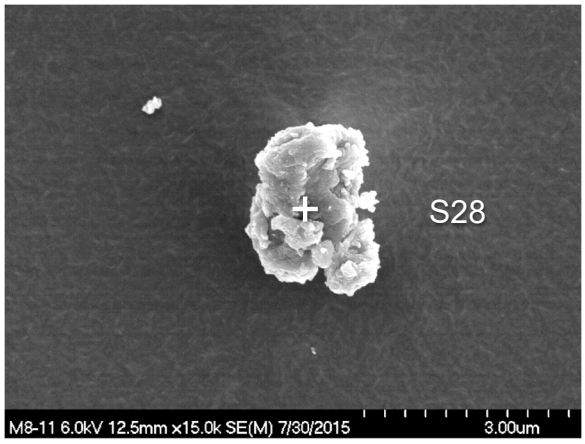


Figure 38. FESEM image of a particle on the space exposed surface of the wake flight sample (M8-W11) with the location identified for EDS spot analyses (S28).

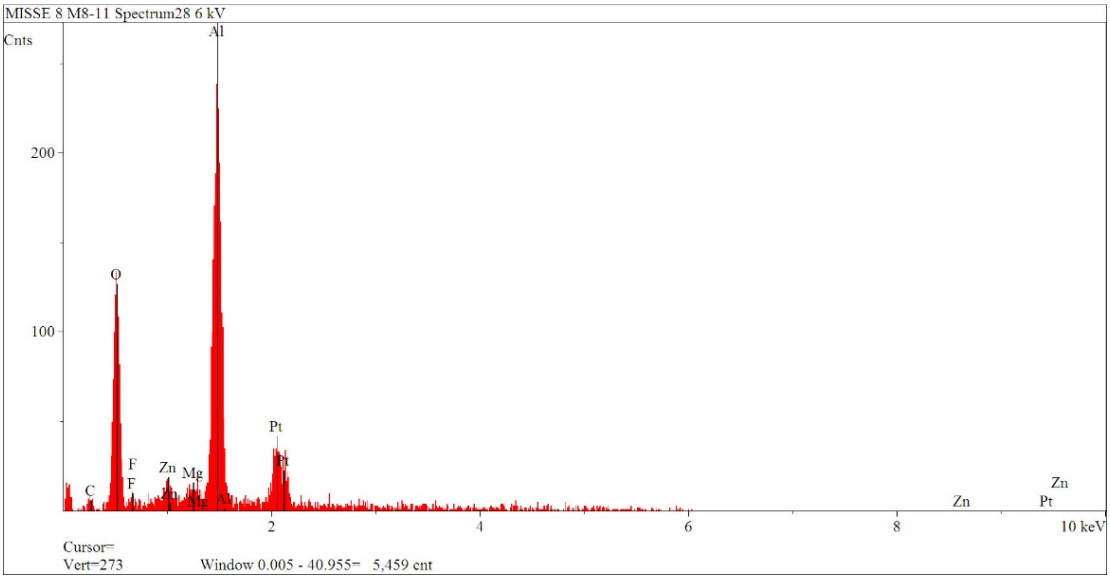


Figure 39. EDS spot analysis spectra (S28) of the particle shown in Figure 38 with Al, O, Zn and Mg peaks in addition to F, C and Pt (conductive coating).

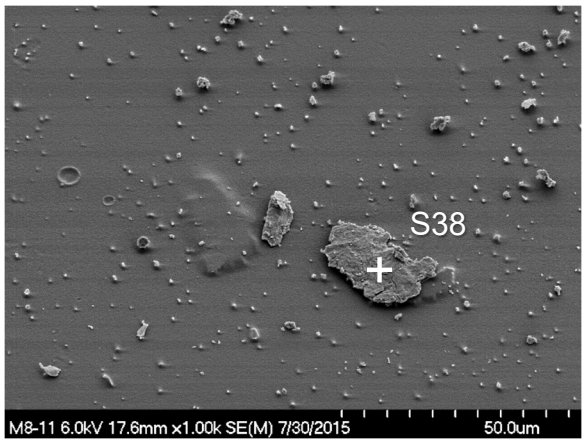


Figure 40. FESEM image of a particle on the space exposed surface of the wake flight sample (M8-W11) with the location identified for EDS spot analyses (S38).

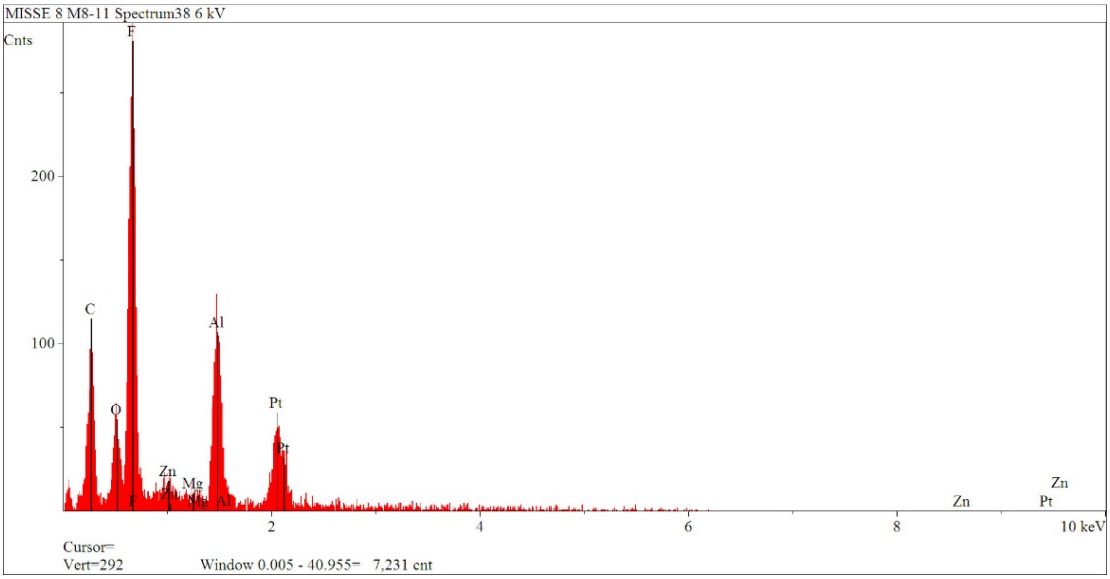


Figure 41. EDS spot analysis spectra (S38) of the particle shown in Figure 40 with Al, O, Zn and Mg peaks in addition to F, C and Pt (conductive coating).

Figure 42 provides FESEM images of the MISSE-8 wake flight sample particles as taken at a 45° tilt angle. As can be seen in these images, some AO erosion of the wake Teflon FEP surface has occurred during the MISSE 8 mission. As stated previously, the AO fluence for the wake ORMatE-III R/W tray surface was determined to be 8.80×10^{19} atoms/cm^2. The particles appear to be AO durable and have protected the underlying area from erosion. The particles appear to have arrived early in the mission (or pre-flight) during a single event based on similar erosion around particles. The AO arrival appears to be somewhat off-normal to the wake surface. The very small amount of erosion may have helped reduce the build-up of a Si contaminant layer during the mission.

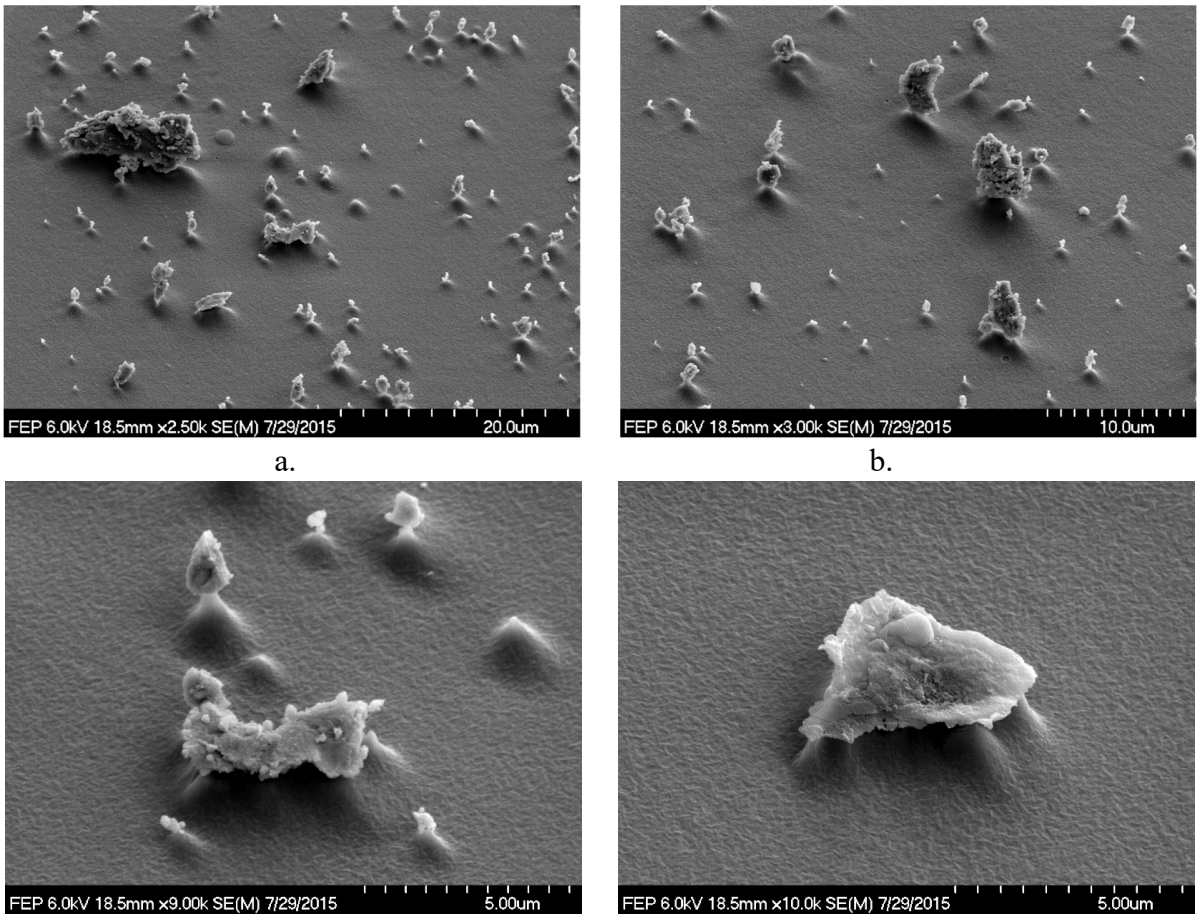
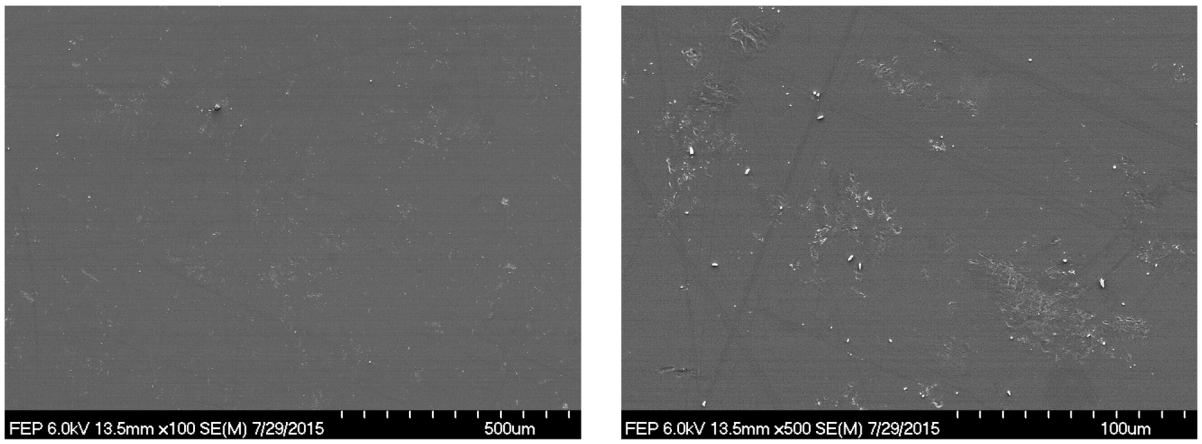


Figure 42. FESEM images of the MISSE-8 wake flight sample (M8-W11) particles taken at a 45° tilt angle: a). Image taken at 2.5kX, b). Image taken at 3kX, c). Image taken at 9kX, and d). Image taken at 10kX.

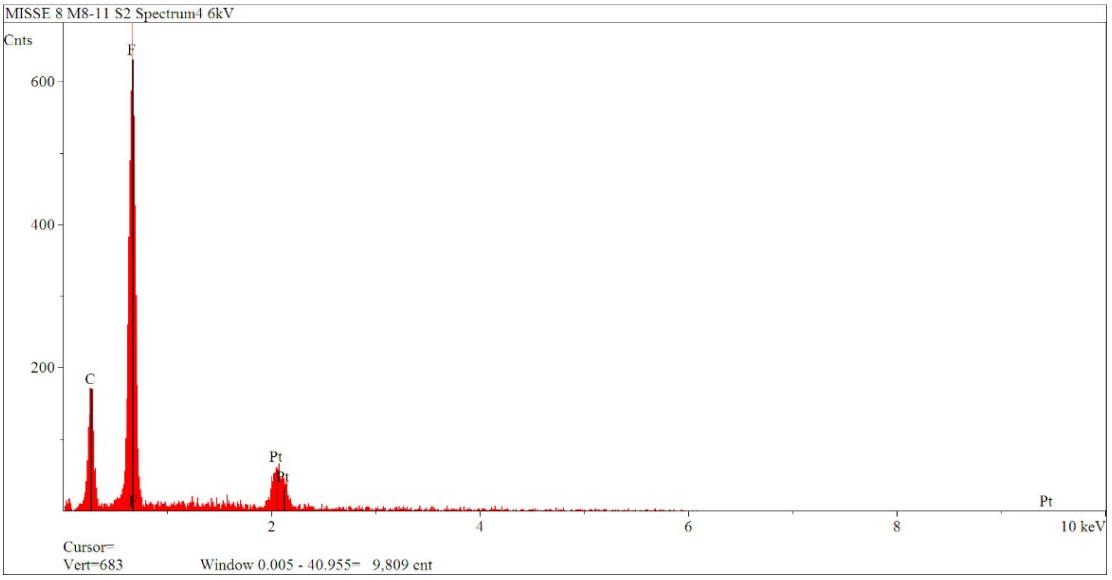
Figure 43 provides FESEM images of the **backside surface** of the MISSE 8 wake flight sample. As can be seen, if compared to the FESEM images of the control sample in Figure 19, the surface is similar to the control but appears to have a very small amount of particles in some areas such as shown in Figure 43b. EDS area analyses of the area shown in Figure 43a taken at both 6 and 15 kV are provided in Figure 44a and 44b, respectively. The only elements detected are F, C and the applied conductive coating Pt. Figure 45 provides XPS spectra of the backside of the wake flight sample. The XPS spectra corresponds with the EDS data. The surface atomic concentration for F was 69.13 at.%, which is slightly higher than the control sample (67.04 at.%), C was 30.82 at.%, which was slightly lower than the control sample (32.8 at.%) and there was a very small amount of O (0.05 vs. 0.09 at.% for the control sample). Thus, the XPS analysis of the backside of the flight sample does not show Al, greater amounts of O, or trace amounts of Si like the flight sample.

Figure 46 provides FESEM images of the cut edge of the MISSE 8 wake flight sample sectioned for FESEM imaging. The top surface of the space exposed surface is embrittled, as can be seen in these images. Teflon is known to be come embrittled with radiation and thermal exposure in the space environment.[14-16] Figure 47 provides FESEM images of the cut edge of the MISSE 8 control sample sectioned for FESEM imaging. These images show that the FEP control sample does not display the same cracking as the embrittled space exposed flight sample.

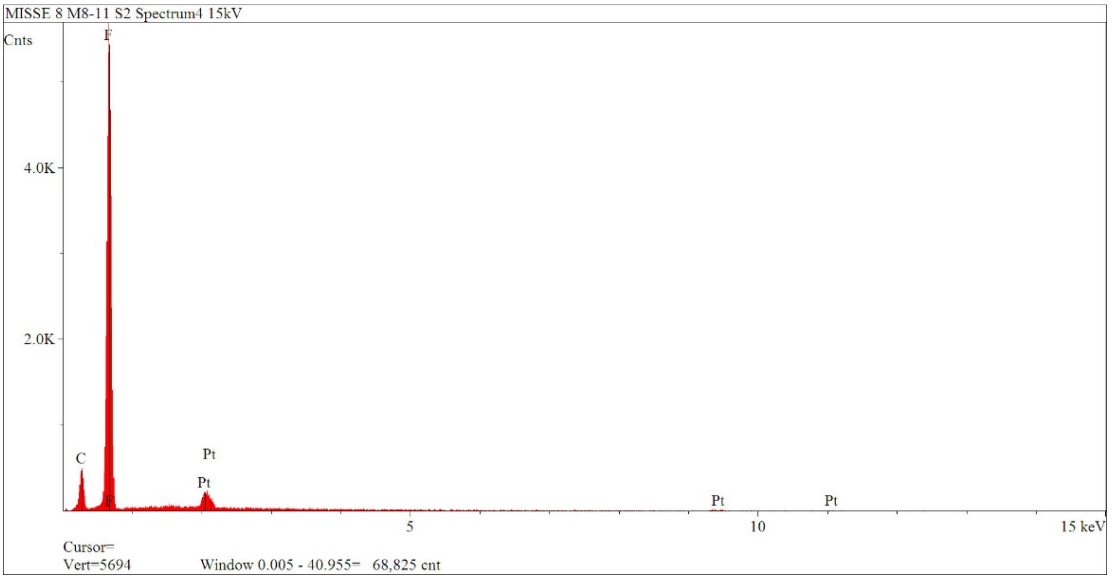


a. b.

Figure 43. FESEM images of the backside of the MISSE 8 wake flight sample (M8-W11): a). Image S2-04 taken at 100X and 0° tilt, b). Image S2-03 taken at 500X and 0° tilt.



a.



b.

Figure 44. EDS area analysis spectra of the backside of wake flight sample (M8-W11) from: a). FESEM image area in Figure 43a taken at 6 kV, and b). FESEM image area in Figure 43a taken at 15 kV.

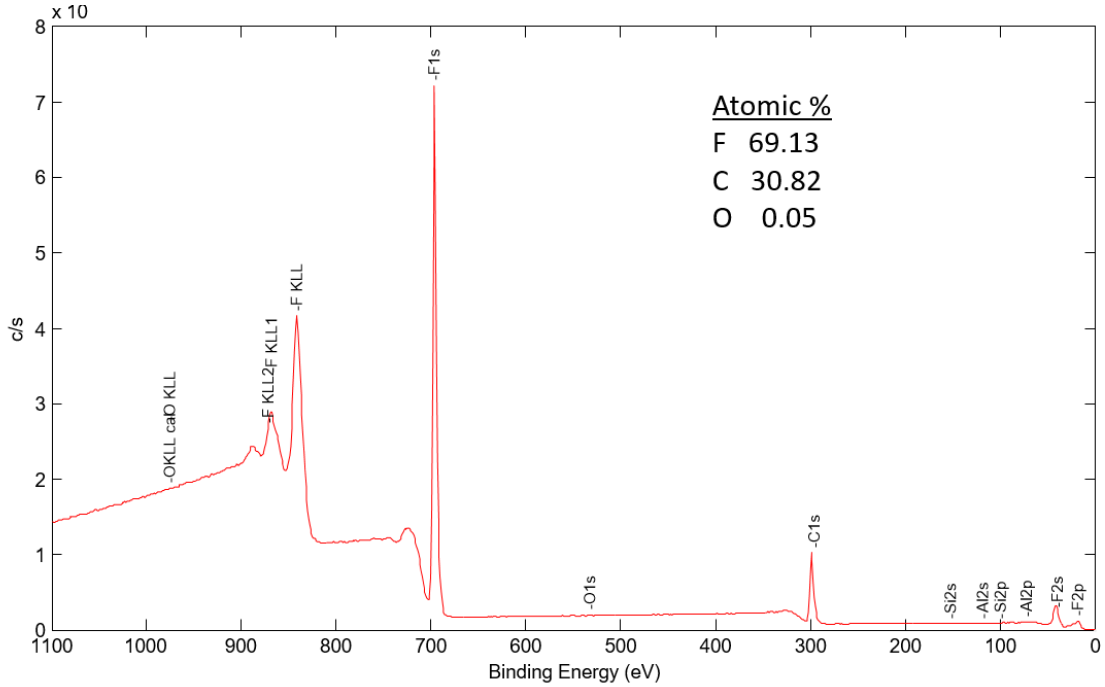


Figure 45. The XPS binding energy data for the backside of wake flight sample M8-W11.

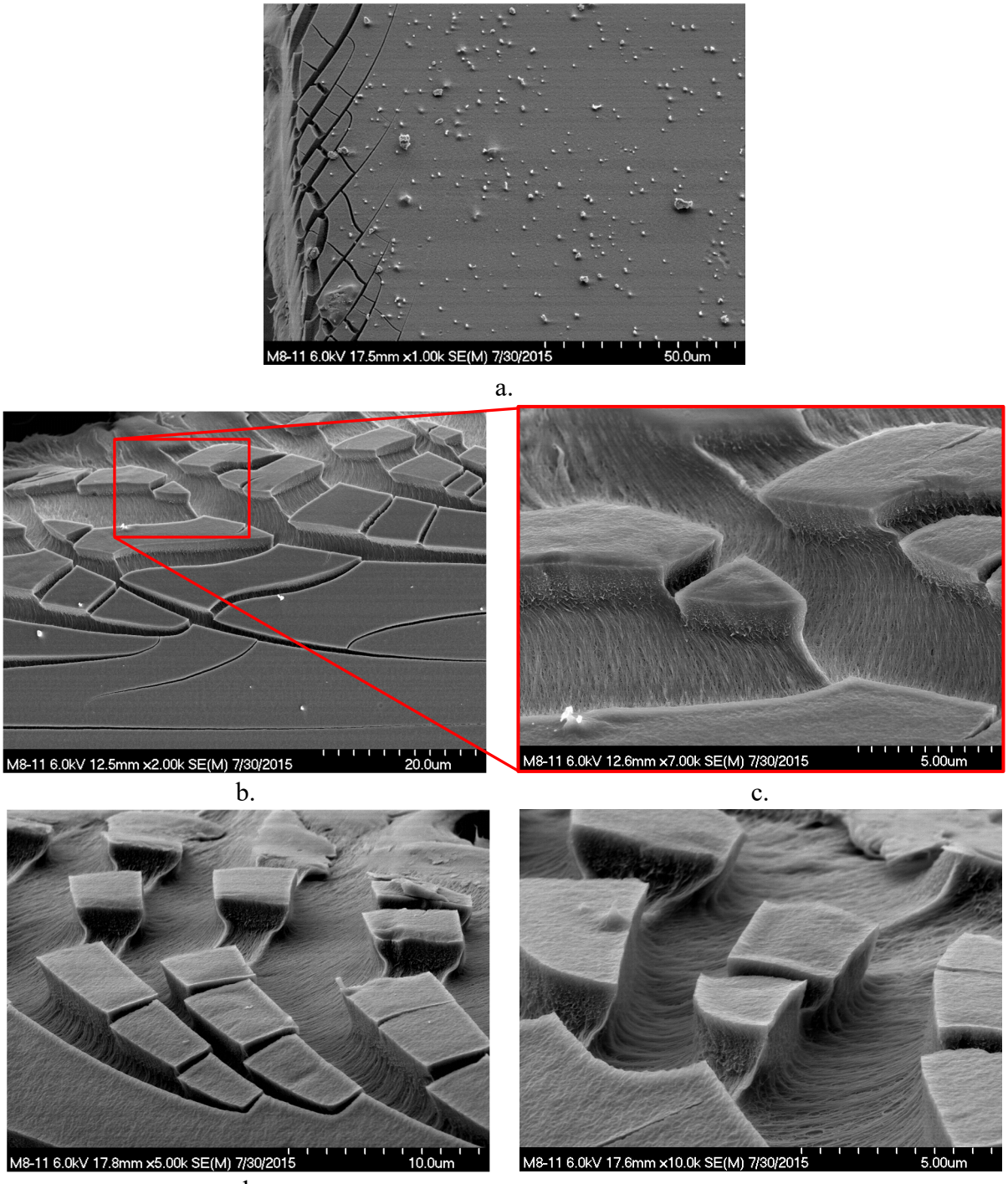
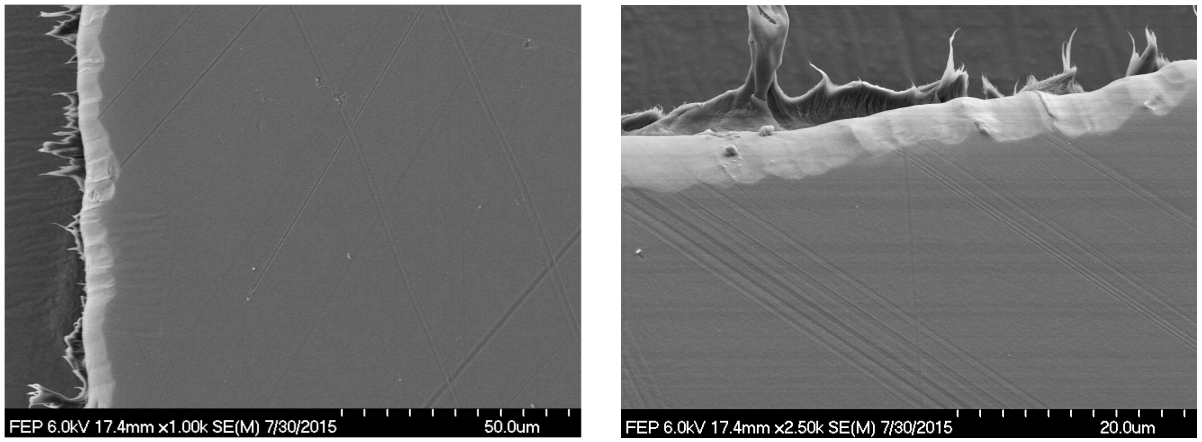


Figure 46. FESEM images of the cut edge of the MISSE 8 wake flight sample (M8-W11) in the space exposed area: a). Image taken at 1kX (0° tilt), b). Image taken at 2kX (0° tilt), c). Image taken at 7kX (0° tilt), d). Image taken at 5kX (40° tilt), and e). Image taken at 10kX (40° tilt).



a. b.

Figure 47. FESEM images of the cut edge of the MISSE 8 wake control sample (M8-R9-B): a). Image taken at 1kX (40° tilt), and b). Image taken at 2.5kX (40° tilt).

6.4 MISSE 8 Nadir Control Sample—FESEM, EDS, and XPS Analyses

Figure 48 provides FESEM images for the MISSE 8 nadir silver-Teflon control sample. The FESEM images show a very smooth surface, even at 3.5kX as shown in Figure 48c. Figures 49 to 51 provide corresponding EDS area analysis spectra for the image areas shown in Figure 48a-c, respectively. The EDS spectra show F and C peaks for FEP and the Pt peak for the applied conductive coating. Figure 52 provides XPS spectra the MISSE 8 nadir silver-Teflon control sample. The XPS spectra corresponds with the EDS data. The surface atomic concentration for F was 68.6 at.% and for C was 31.3 at.%. The control sample also had a small amount of O (0.1 at.%).

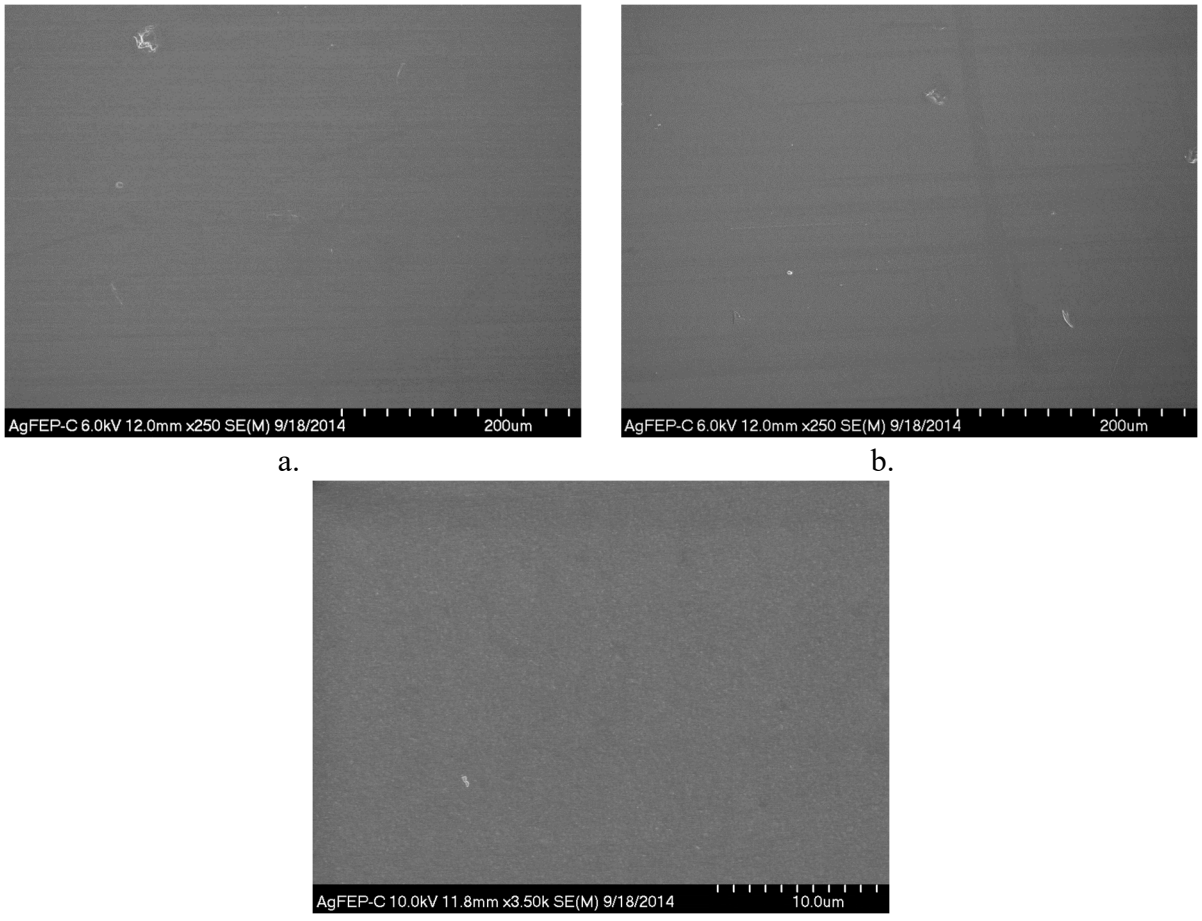


Figure 48. FESEM images of the silver-Teflon control sample (0° tilt): a). Image taken at 250X and 6 kV (C01), b). Image taken at 250X and 6 kV (C11), and c). Image taken at 3.5kX and 10 kV (C13).

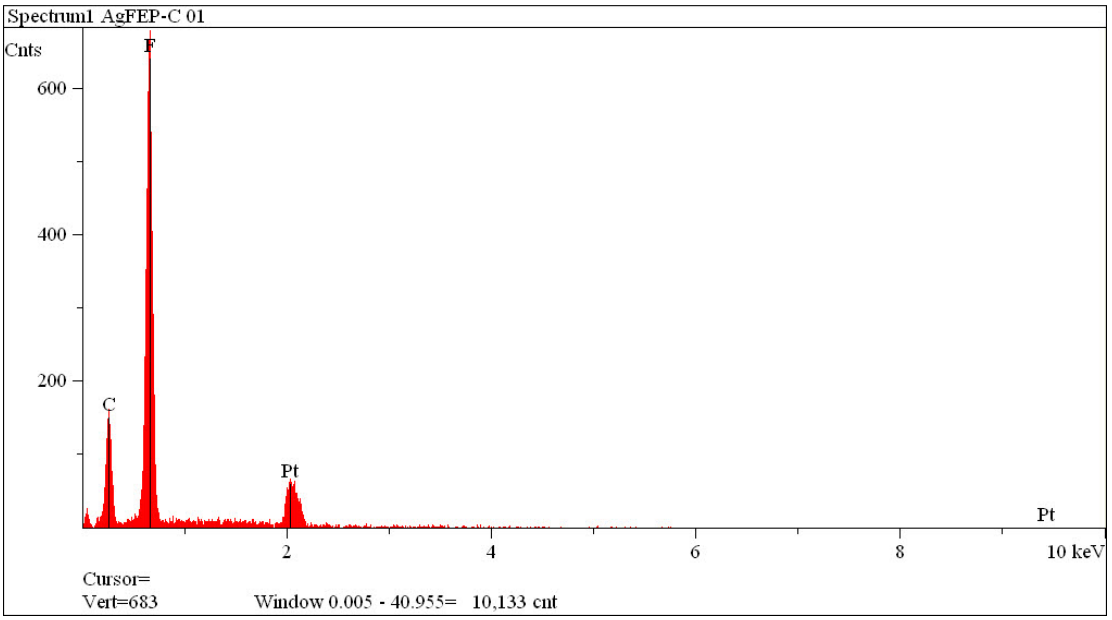


Figure 49. EDS area analysis spectra of the silver-Teflon control sample from FESEM image area in Figure 48a taken at 6 kV.

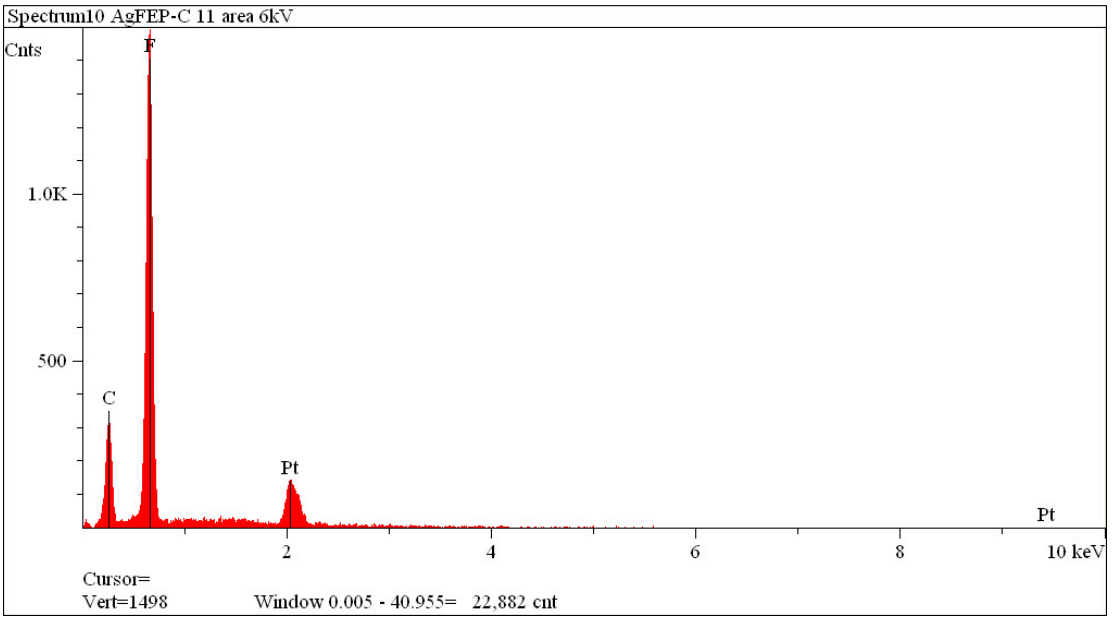


Figure 50. EDS area analysis spectra of the silver-Teflon control sample from FESEM image area in Figure 48b taken at 6 kV.

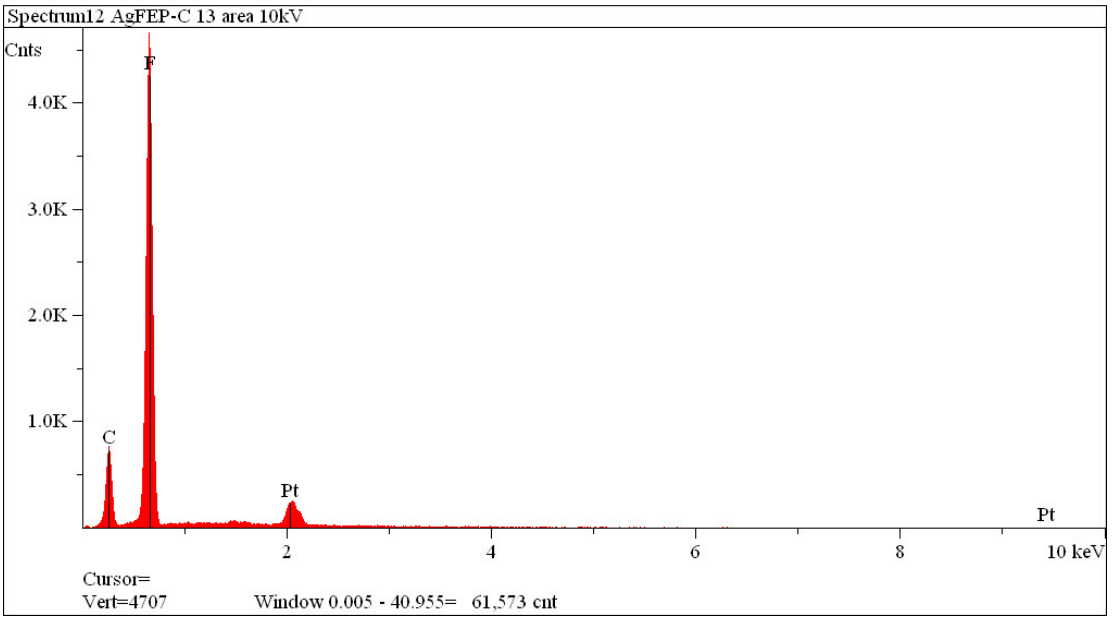


Figure 51. EDS area analysis spectra of the silver-Teflon control sample from FESEM image area in Figure 48c taken at 10 kV.

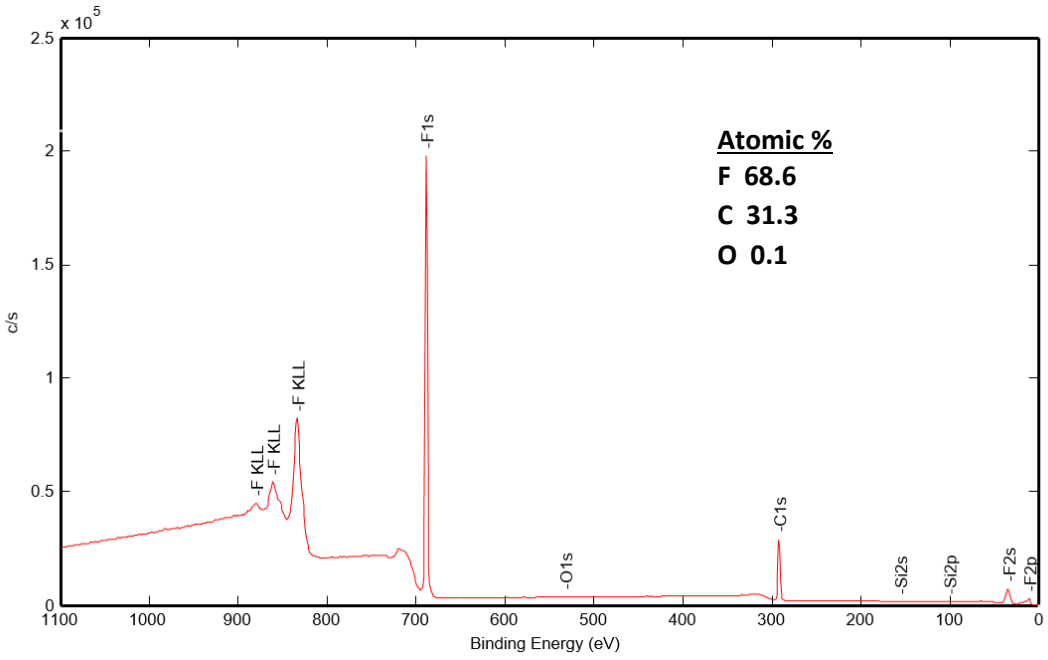


Figure 52. The XPS binding energy data for the silver-Teflon control sample.

6.5 MISSE 8 Nadir Flight Sample—FESEM, EDS, and XPS Analyses

Figure 53 provides FESEM images for the MISSE 8 nadir SEUXSE Power Box silver-Teflon radiator sample. The FESEM images show a smooth surface, although higher magnification images at some of the particle sites, such as shown in Figure 53d, show a very small amount of "grazing AO" erosion. As stated previously, the AO E_y for MISSE 8 nadir samples was determined to be 3.6×10^{19} atoms/cm^2.[12] Figures 54 to 57 provide corresponding EDS area analysis spectra for the image areas shown in Figures 53a-d, respectively. The EDS spectra shown in Figures 53a-d all show F and C peaks for FEP and the Pt peak for the applied conductive coating. No other elements are present in the EDS spectra. Figure 58 provides XPS spectra the nadir SEUXSE Power Box silver-Teflon radiator flight sample. The XPS spectra corresponds with the EDS data. The surface atomic concentration for F was 68.5 at.% and for C was 31.1 at.%. The control sample also had a small amount of O (0.4 at.%).

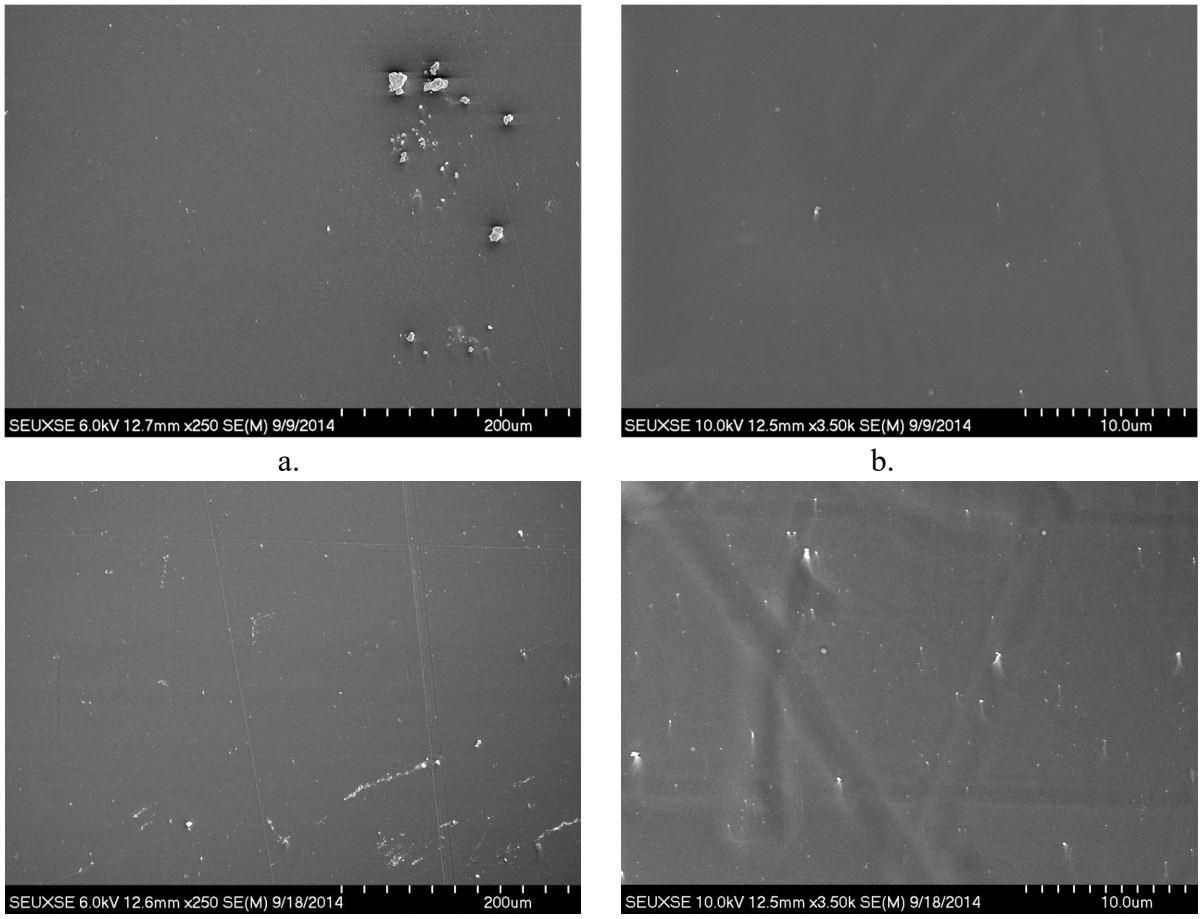


Figure 53. FESEM images of the SEUXSE flight sample (0° tilt): a). Image taken at 250X and 6 kV (02), b). Image taken at 3.5kX and 10 kV (19), c). Image taken at 250X and 6 kV (25), and d). Image taken at 3.5kX and 10 kV (27).

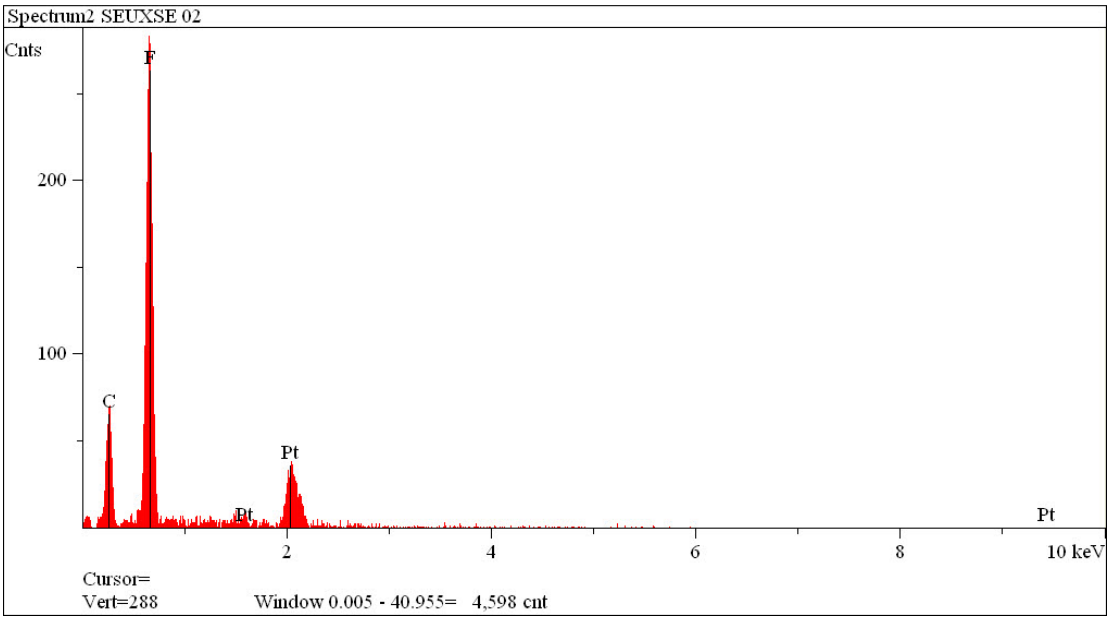


Figure 54. EDS area analysis spectra of the nadir SEUXSE silver-Teflon flight sample from FESEM image area in Figure 53a taken at 6 kV.

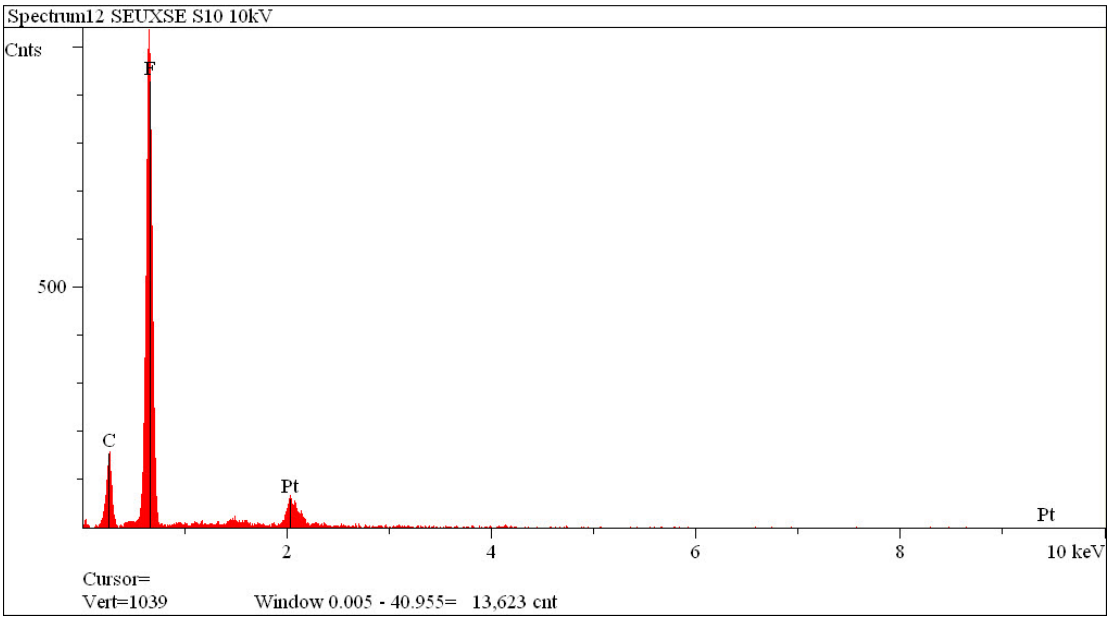


Figure 55. EDS area analysis spectra of the nadir SEUXSE silver-Teflon flight sample from FESEM image area in Figure 53b taken at 10 kV.

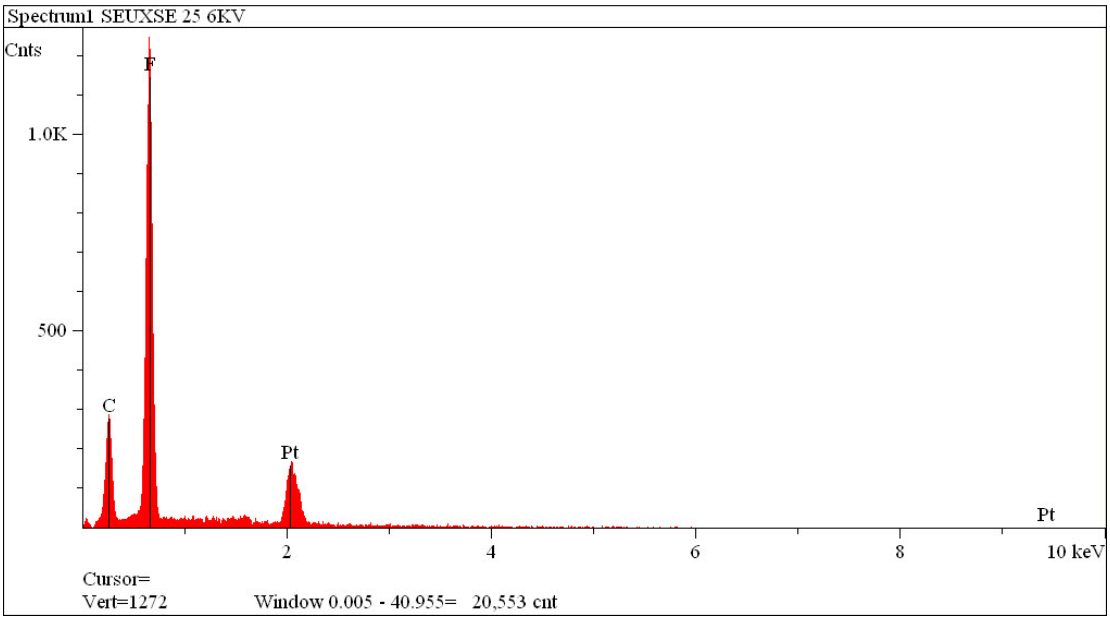


Figure 56. EDS area analysis spectra of the nadir SEUXSE silver-Teflon flight sample from FESEM image area in Figure 53c taken at 6 kV.

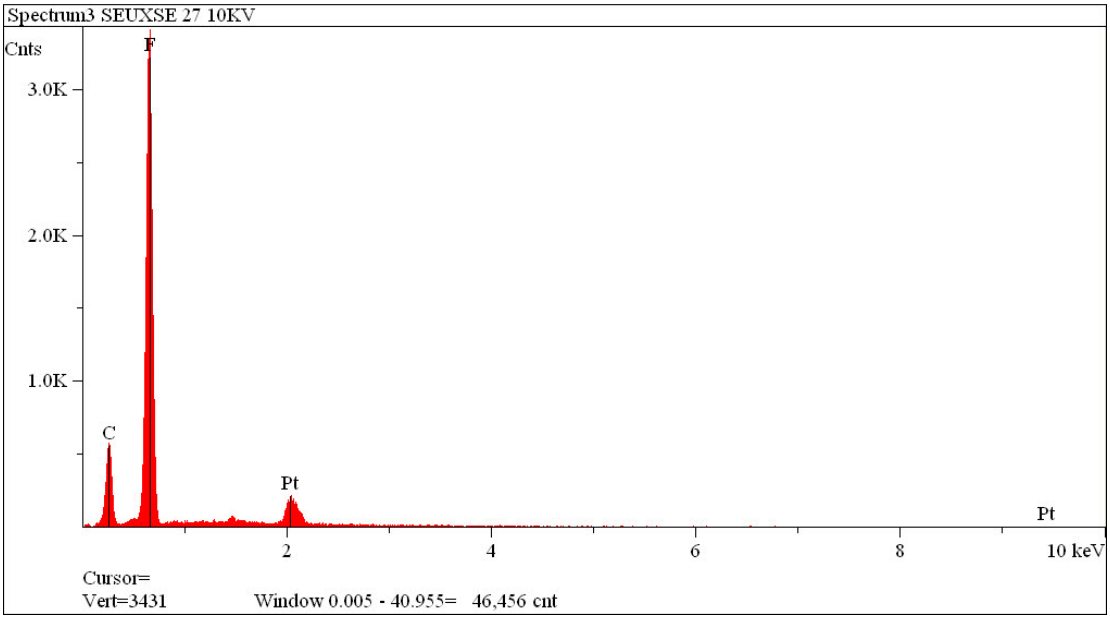


Figure 57. EDS area analysis spectra of the nadir SEUXSE silver-Teflon flight sample from FESEM image area in Figure 53d taken at 10 kV.

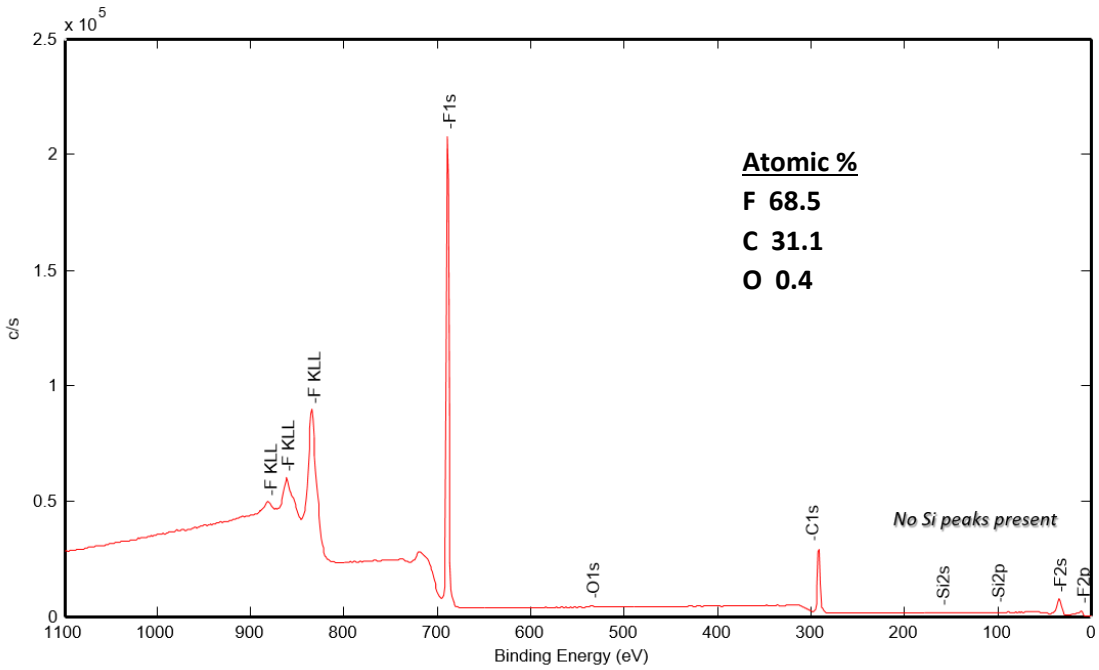
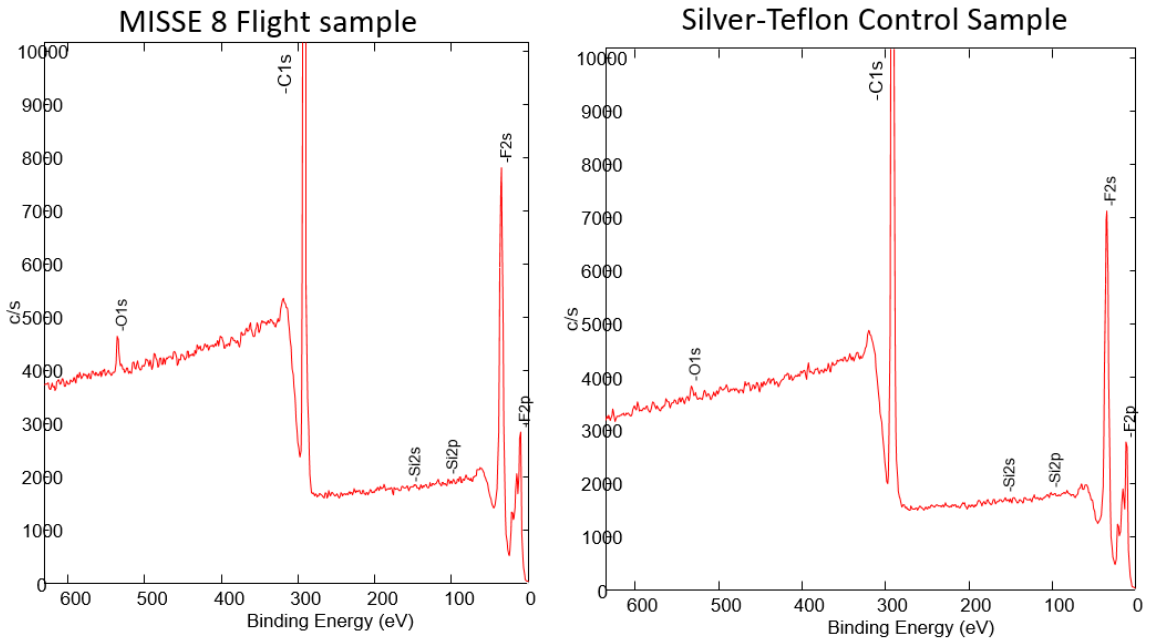


Figure 58. The XPS binding energy data for the nadir SEUXSE silver-Teflon flight sample.



a. b.

Figure 59. Expanded regions of the XPS binding energy data for: a). MISSE 8 nadir SEUXSE silver-Teflon flight sample, and b). Silver-Teflon control sample.

Figure 59 provides expanded regions of the XPS binding energy data for the MISSE 8 nadir SEUXSE silver-Teflon flight sample (Figure 59a) and the silver-Teflon control sample surface (Figure 59b). The expanded region indicates that there is no Si molecular contamination, but some oxidation is observed.

Figures 60 to 63 provides a FESEM image and corresponding EDS spot analysis spectra of two particles on the MISSE 8 nadir SEUXSE silver-Teflon flight sample. The EDS spot analyses were conducted at high magnification imaging (10 kX) and the electron beam accelerating voltage was 6 and 15 kV. The spectra in Figures 61 to 63 (S3, S4, and S6, respectively) indicate that the two particles in Figure 60 are Zn-oxide rich particles. Smaller Al and Mg peaks are also present for the EDS spectra S4 taken at 15 kV and for the EDS spectra S6 taken at 6 kV.

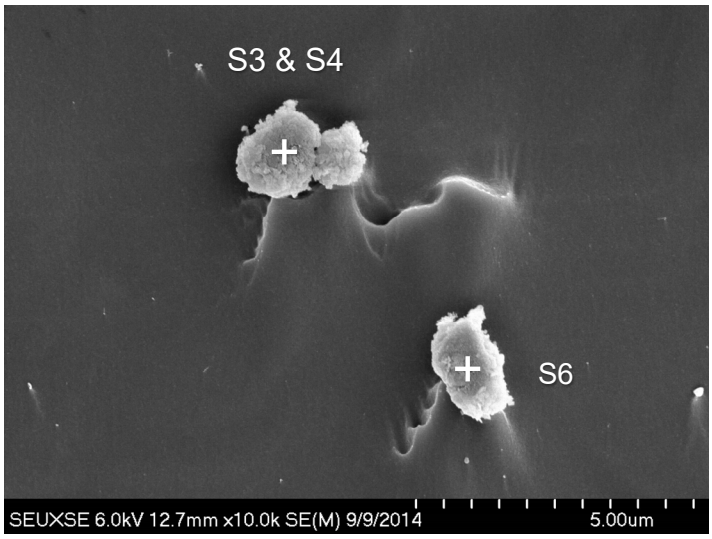


Figure 60. FESEM image of particles on the space exposed surface of the MISSE 8 nadir SEUXSE silver-Teflon flight sample with the locations identified for EDS spot analyses (S3, S4, and S6).

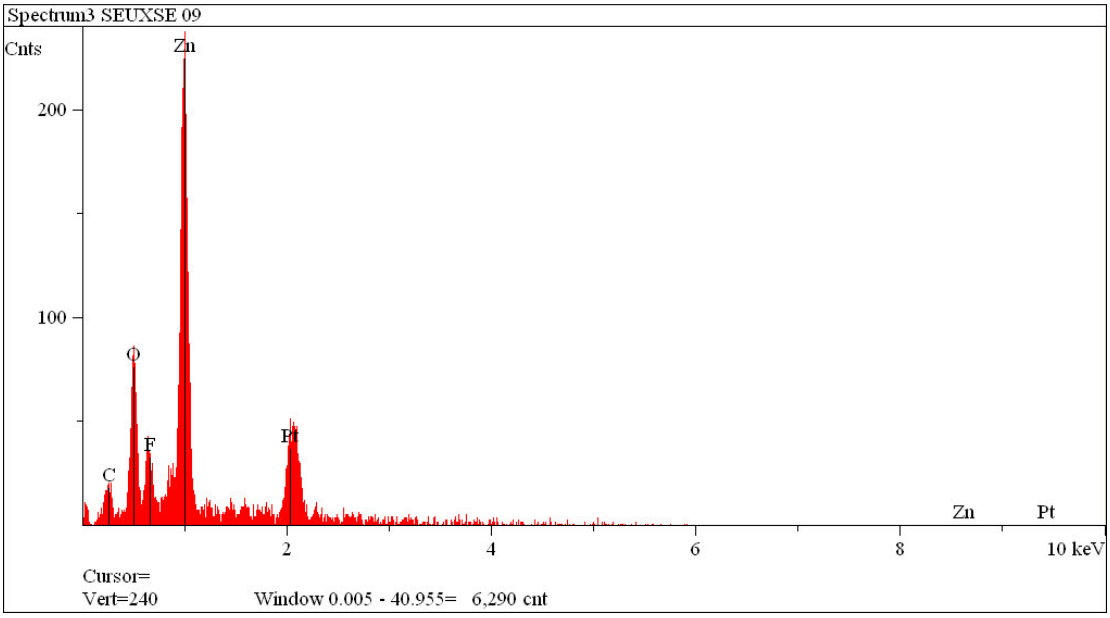


Figure 61. EDS spot analysis spectra (S3) of the particle shown in Figure 60 taken at 6 kV.

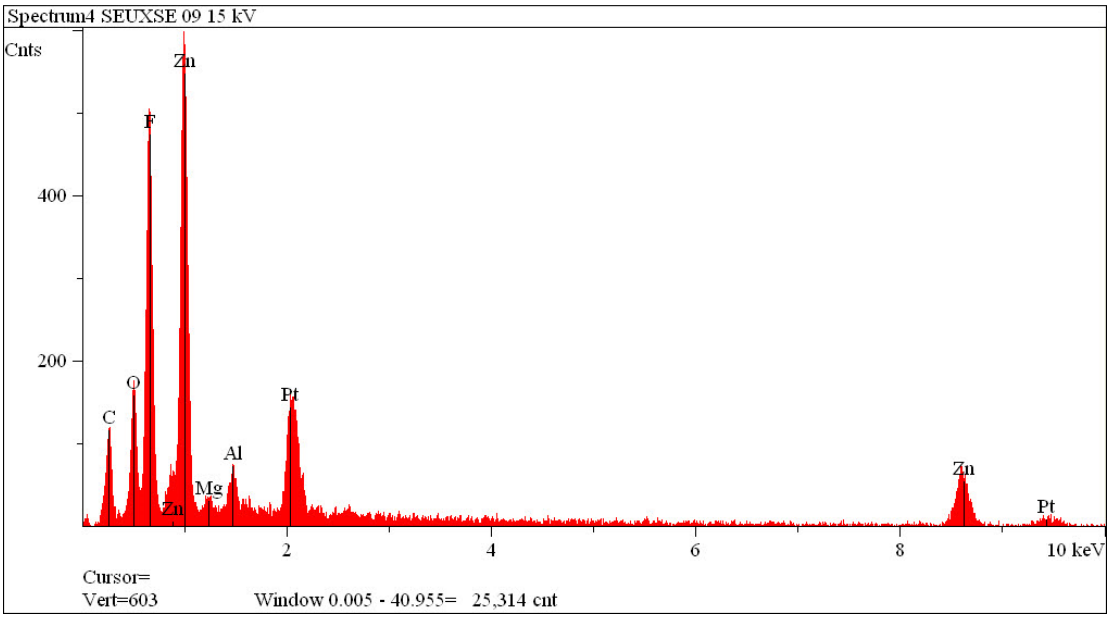


Figure 62. EDS spot analysis spectra (S4) of the particle shown in Figure 60 taken at 15 kV.

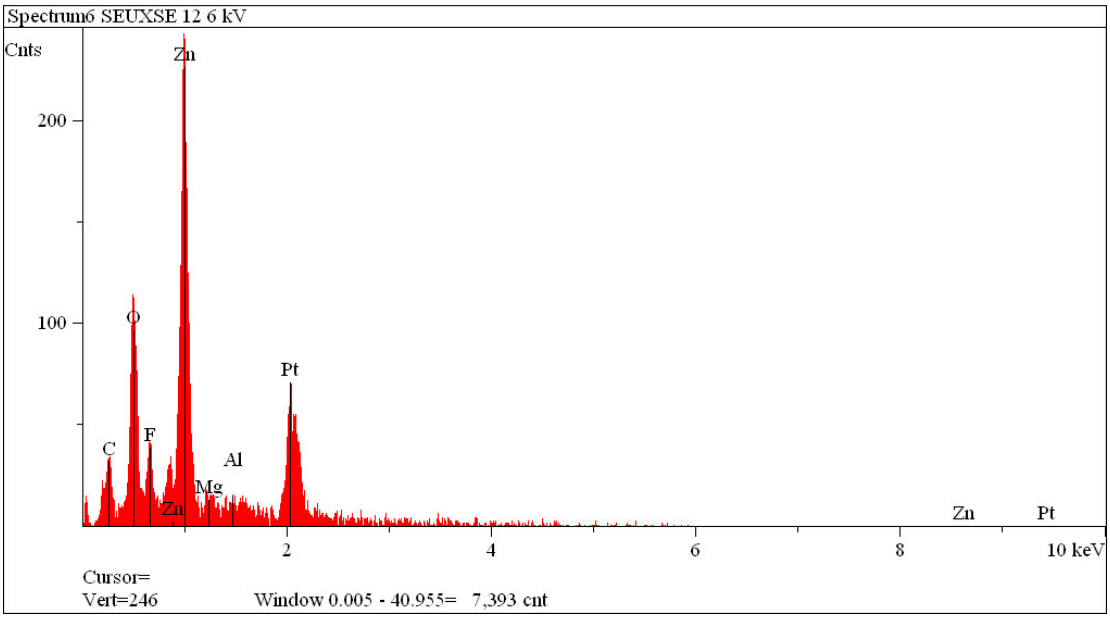


Figure 63. EDS spot analysis spectra (S6) of the particle shown in Figure 60 taken at 6 kV.

Figure 64 provides XPS spectra for a large particle on the MISSE 8 nadir SEUXSE silver-Teflon flight sample. The XPS spectra is similar to the EDS spectra for the Zn-oxide particles. In addition to F (36.6 at.%) and C (26.8 at.%), the particle has 17.6 at.% O, 5.7 at.% sodium (Na), 5.0 at.% Zn, 4.7 at.% Si, 1.5 at.% nitrogen (N), 1.4 at.% Al and 0.7 at.% chlorine (Cl).

Figure 65 provides FESEM images of a "cluster" of fine particles on the MISSE 8 nadir SEUXSE silver-Teflon flight sample. Corresponding EDS spot analysis spectra in Figure 66 (S10) indicates that the particle cluster shown in Figure 65 is Fe-oxide rich. The spectra also shows peaks for Al, Zn, Cl and a very small potassium (K) peak.

Possible sources of Zn-rich particles include the AMS white silicone paint (SG121GD) which contains ZnO and Na. The bottom side of the MISSE 8 SEUXSE Power Box (see Figure 67) contains a white thermally conductive heat-sink silicone grease (Nusil CV-9341) that was applied pre-flight. Figure 68 provides XPS spectra for a sample sectioned from the bottom side of the MISSE 8 SEUXSE Power Box (sample location shown in Figure 67). The sample was sputter cleaned with Ar+ ion beam to remove ~200 nm of surface contamination. The heat-sink silicone grease contains ZnO, as confirmed in the XPS spectra. Other sources of Zn-rich particles could be other MISSE experiments, or other ELC-2 or ISS payloads.

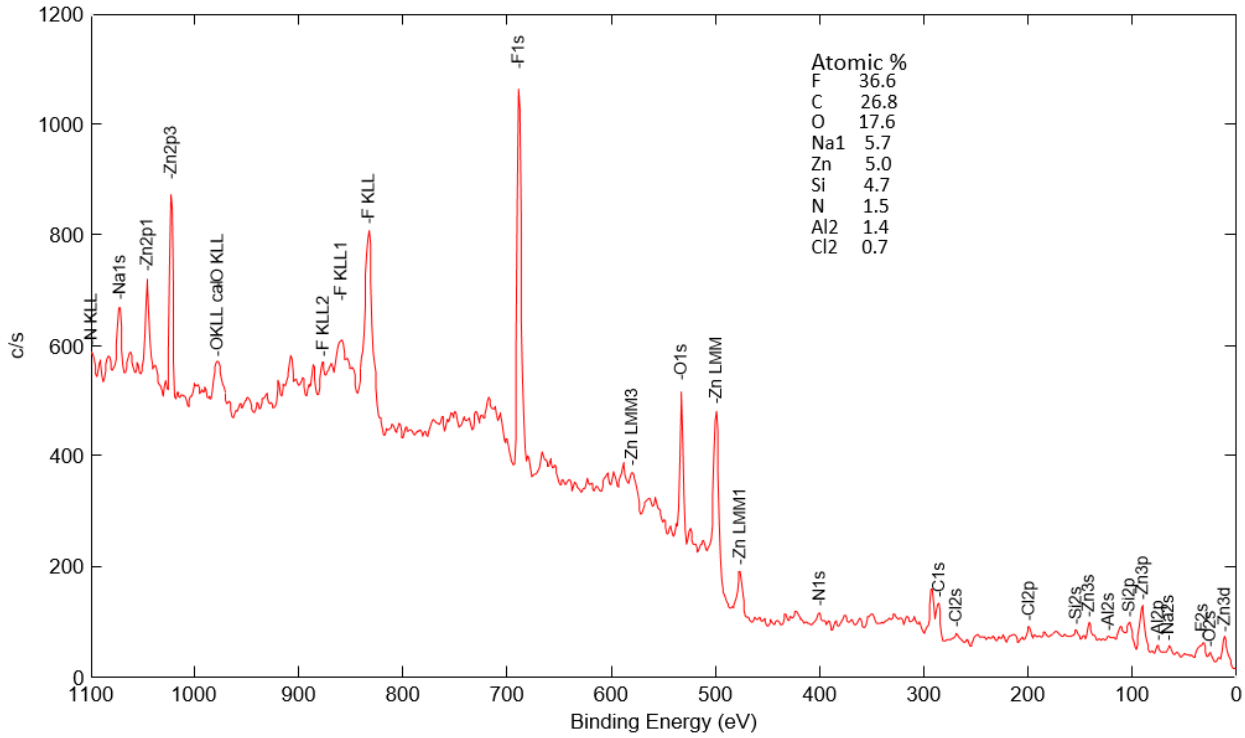


Figure 64. The XPS binding energy data for a large particle on the space exposed surface of nadir SEUXSE silver-Teflon flight sample.

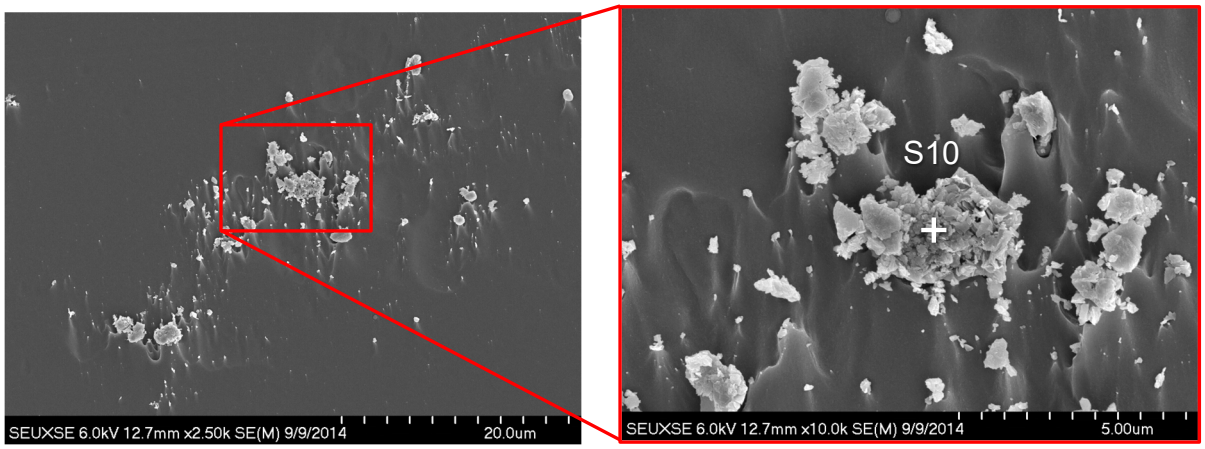


Figure 65. FESEM image of the MISSE 8 nadir SEUXSE silver-Teflon flight sample with a higher magnification image (right) showing the spot analysis location (S10) of a particle cluster.

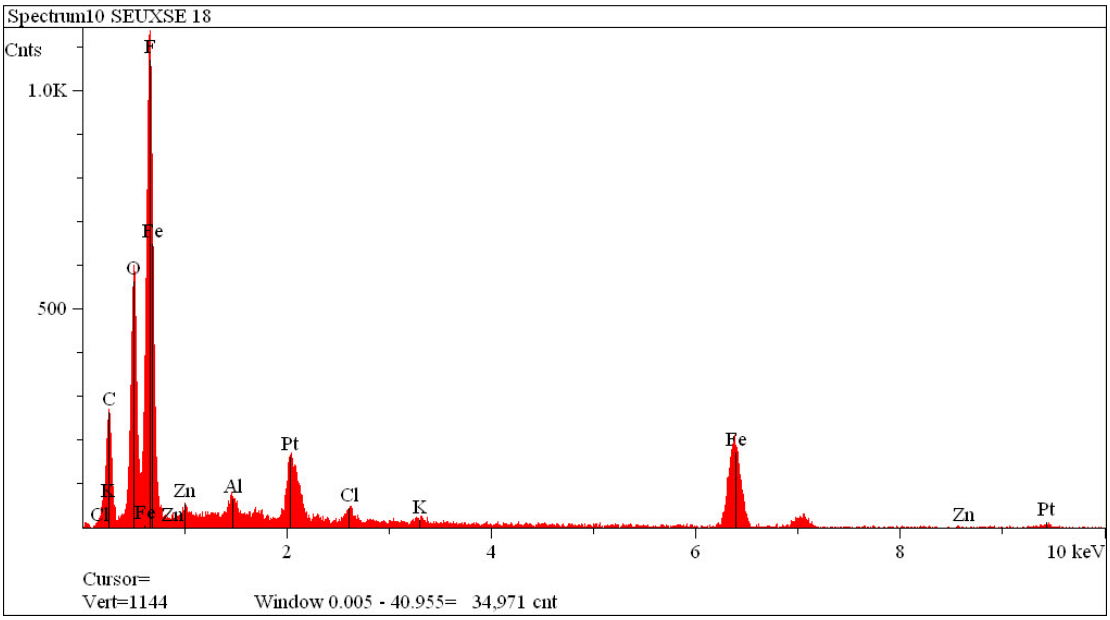


Figure 66. EDS spot analysis spectra (S10) of the particle shown in Figure 65 taken at 15 kV.

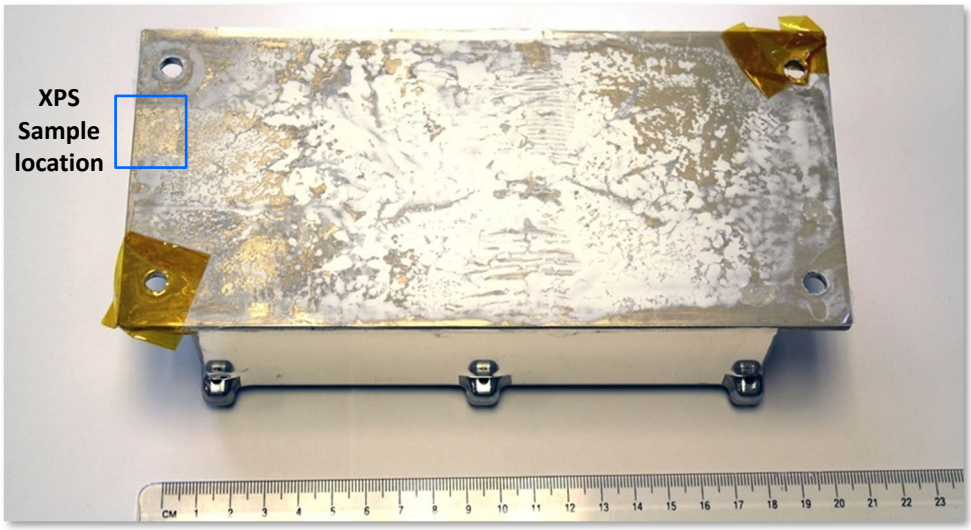


Figure 67. The bottom side of the MISSE 8 SEUXSE Power Box showing the white Nusil CV-9341 thermally conductive silicone grease that was applied pre-flight.

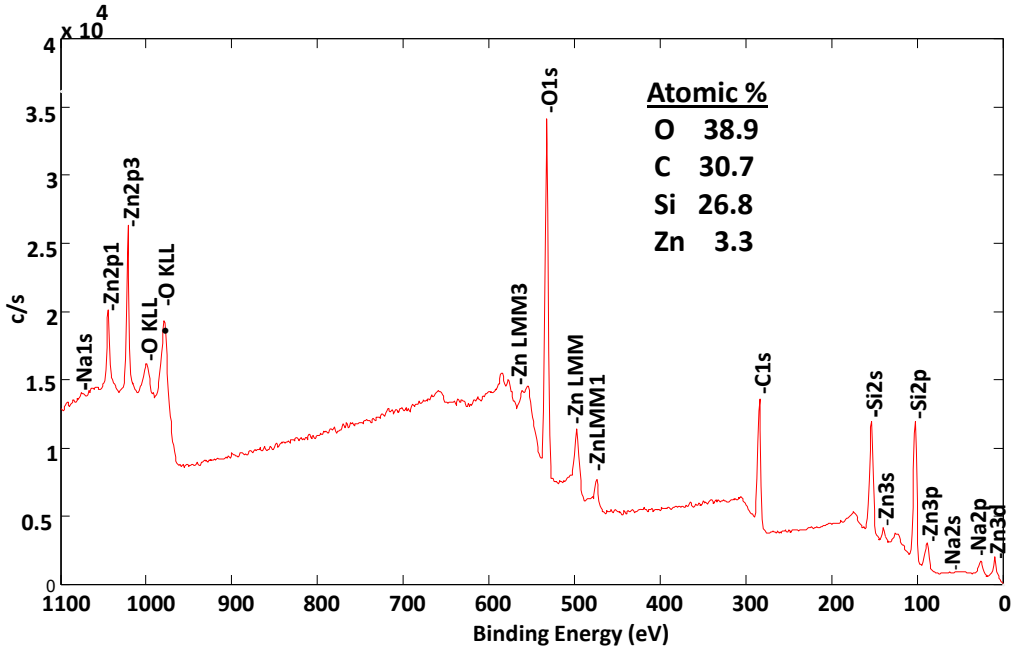


Figure 68. The XPS binding energy data of the sample sectioned from the bottom side of the MISSE 8 SEUXSE Power Box showing the white Nusil CV-9341 thermally conductive silicone grease that was applied pre-flight.

Figure 69 provides FESEM images showing the topography of the MISSE 8 nadir SEUXSE silver-Teflon flight sample surface. These images show a smooth surface with a small amount of particles. High magnification images show that a very small amount of erosion occurred around the particles (< 0.5 μm) and that the erosion was very directional, consistent with grazing AO arrival. The amount of erosion for the MISSE 8 nadir Teflon surface was less than that observed for the MISSE 8 wake Teflon surface, as shown in Figure 42. This is consistent with the AO

fluence for the two surfaces (3.6×10^{19} atoms/cm^2 for the nadir surface vs. 8.80×10^{19} atoms/cm^2 for the wake surface). This very small amount of erosion was not likely to have impacted the build-up of a molecular contaminant layer on-orbit.

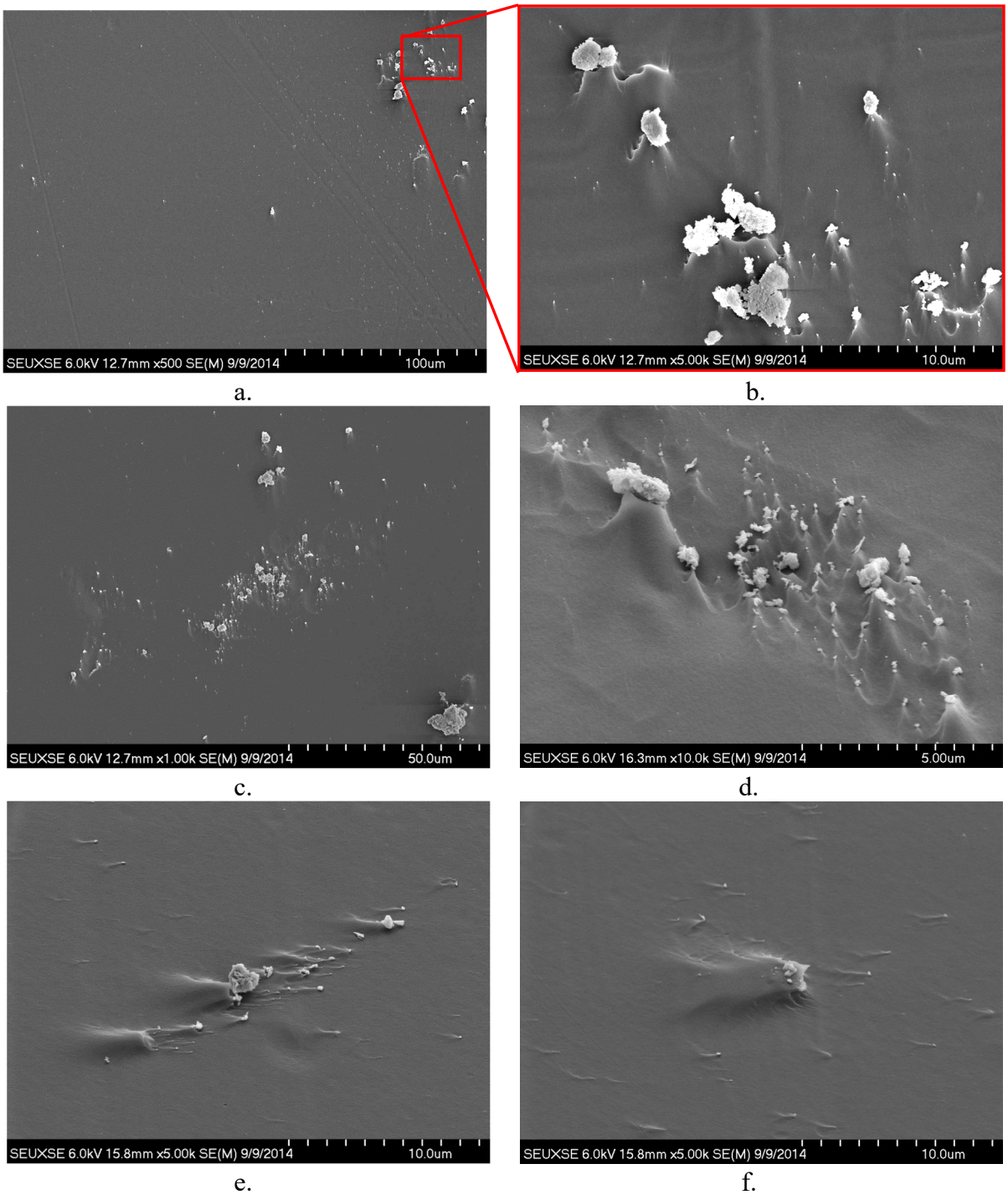


Figure 69. FESEM images of MISSE-8 nadir SEUXSE Power Box silver-Teflon sample: a). Image taken at 500X (0° tilt), b). Image taken at 5kX (0° tilt), c). Image taken at 1kX (0° tilt), d). Image taken at 10kX (45° tilt), e). Image taken at 5kX (45° tilt), and e). Image taken at 500X (45° tilt).

7.0 Summary and Conclusions

Samples of Teflon FEP flown in the wake and nadir directions during the MISSE 8 mission were analyzed for on-orbit contamination. The wake sample was Teflon FEP (M8-W11) flown as part of the Glenn MISSE 8 Polymers Experiment on the ORMatE-III R/W tray and exposed to LEO wake environment for 2.0 years. The nadir samples were silver-Teflon radiator pieces taken from the MISSE 8 SEUXSE Power Box and exposed to LEO nadir environment for 2.14 years. Flight and corresponding control samples were analyzed for surface morphology (FESEM) and chemistry (EDS and XPS). The wake sample was also analyzed for changes in solar absorptance as compared to a control sample.

The MISSE 8 wake Teflon FEP flight sample had a slightly hazy appearance in some regions. The total reflectance of the flight sample did not change significantly as compared to the control sample, but the total transmittance decreased from 0.951 to 0.940. The flight sample had increased diffuse reflectance and transmittance, and corresponding decreased specular reflectance and transmittance. The AM0 integrated solar absorptance of the flight sample increased by 0.008 as compared to the control sample.

The hazy section of the MISSE 8 wake Teflon FEP flight sample had particulate contamination, as seen in optical and FESEM images. High magnification FESEM images showed a small amount of erosion of the wake sample around the particles, which appear to be AO durable and have protected the underlying area from erosion. The AO arrival appears to have been off-normal to the wake surface. In addition, the particles appear to have arrived early in the mission (or pre-flight) during a single event based on similar erosion around all the particles. The EDS analyses indicated that the particles are Al and O rich, with very small amounts of Zn and Mg. The EDS data also indicated no evidence of a molecular contamination layer (no Si detected) on the flight sample.

The XPS analyses of the MISSE 8 wake Teflon FEP flight sample also indicated visible (optical camera) evidence of erosion and there was some corresponding degradation of the polymer as evidenced in the XPS carbon spectrum due to AO oxidation. High resolution scans were obtained on C, F, O, Al, and Si regions. Barely detectable amounts of Si (0.02 at.% after overnight scan) were observed on the exposed FEP surfaces indicating no molecular contaminant layer. No Si was observed on the backside of the flight sample or the control sample. Also, no hydrocarbon molecular contamination was detected on the flight sample. Trace amounts of Al were detected (0.05 to 0.1 at.%), which are most likely due to the particulates on the surface.

High magnification FESEM images showed evidence of surface erosion of the MISSE 8 nadir silver-Teflon material. The FESEM images indicated that the extent of AO erosion was very small (< 0.5 μm) and the texture was consistent with grazing AO arrival. The FESEM images also showed some random particulate contamination. But, much less than on the wake sample.

Results from EDS and XPS analyses for the MISSE 8 nadir silver-Teflon material were very consistent. Both found no evidence of general molecular contamination (no silicone or

hydrocarbon contamination). High resolution XPS scans were obtained on C, F, O and Si regions. The C region showed a slight increase in the amount of O incorporation over time (as expected due to AO interaction). However, there was not any evidence of C-Si bonds or hydrocarbons. The Si regions for both the flight and control samples showed only noise. Particles on the surface were found to be composed mainly of Zn, and also contained Na, Al, Cl and Si. One particulate area analyzed by EDS was found to be Fe-rich. Heat-sink grease on the bottom of the SEUXSE Power Box was also analyzed and found to contain Zn and Si.

There was no evidence of a molecular contamination layer present on the surface of either the MISSE 8 wake or MISSE 8 nadir facing Teflon FEP flight samples (within XPS instrument detection limits, 0.1 at.%). Although, the wake and nadir flight samples did contain particulate contamination in some regions. The nadir particles analyzed were primarily Zn-rich. The majority of analyzed wake particles appear to be oxidized Al with small amounts of Zn and Mg. The wake particles appear to have arrived early in the mission (or pre-flight) during a single event.

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