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Degradation of Intercalated Graphite Epoxy Composites Exposed to Low Earth Orbit

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Degradation of Intercalated Graphite Epoxy Composites Exposed to Low Earth Orbit

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Abstract

Four different graphite fiber/epoxy composites were exposed to the wake-side low Earth orbit (LEO) environment on the Optical Reflector Materials Experiment III (ORMatE-III) platform mounted on the exterior of the ISS for two years in order to determine their long term durability in the space environment. Three of the composite samples used bromine intercalated P100 fibers and one used pristine P100 fibers. One of the P100-Br samples was coated with a protective SiO_2 layer, and half of another was coated with SiO_2. Results were compared with the EOIM-III experiment which exposed the same materials in LEO in the ram direction for 42 h on the Space Shuttle. Although the atomic oxygen (AO) fluence of the ORMatE-III samples was one-third of the EOIM-III exposure, the resulting effects were qualitatively the same. Both found that SiO_2 coated intercalated graphite composites showed no AO erosion, verifying that conventional protection strategies are applicable to bromine intercalated composites. Both found that bromine intercalation does not alter the AO erosion rates of graphite fiber/epoxy composites. Both found no bromine was detected to have migrated into the surrounding epoxy, even for highly eroded fibers and epoxy, allaying fears that bromination could compromise the properties of the epoxy. And both found no corrosive bromine was detected to have been released by the fibers allaying fears that outgassing could degrade sensitive electronics. The uncoated EOIM-III samples suffered slightly greater erosion resulting in a slightly higher solar absorptance. The general conclusion is that ram AO exposure had very similar effects on graphite fiber epoxy composites in both the EOIM-III and ORMatE-III experiments, and in neither case did bromine intercalation of the fibers substantially influence that degradation.

Introduction

Because of their low density and high strength and stiffness, carbon fiber composites are being used extensively as structural materials in modern spacecraft. Recent examples include the aeroshell for the Curiosity rover, (Ref. 1) the primary bus structure for the Maven Mars orbiter, (Ref. 2) and the Space-X Falcon 9 launch vehicle (Ref. 3). However, because carbon fibers have only moderately low electrical resistivity (220 to 2000 μΩ-cm) they have not reached their full potential as multifunctional materials. A proven way to lower the resistivity of the more ordered carbon fibers, referred to here as graphite fibers, is by the process of intercalation. Intercalation is the insertion of guest atoms or molecules between the layers of laminar materials. For this study the laminar material was Thornel P-100 graphite fiber (Amoco)* and guest molecules were Br_2. The P-100/Br system has been studied extensively and found to have a good mix of mechanical, thermal, and electrical properties with a reasonable cost (Ref. 4). The baseline application for P-100/Br epoxy composites is for electromagnetic interference (EMI) shielding boxes, (Ref. 5) but many other multifunctional applications have been proposed (Ref. 6).

*Since the composites were made for this study Amoco has sold their Carbon Fiber business to Cytec.

It is important to evaluate the degradation of spacecraft materials that might be exposed to the space environment. In the case of P-100/Br epoxy composites, it has been well established that other carbon and epoxy polymers are oxidized by atomic oxygen (AO) in the low Earth orbit (LEO) environment (Ref. 7). The third Evaluation of Oxygen Interaction with Materials (EOIM-III) experiment included six samples meant to evaluate the durability of bromine intercalated graphite fiber/epoxy composites in LEO. EOIM-III was part of the payload for the Space Shuttle Discovery STS-46 mission launched on July 31, 1992. On August 6, at 5:30 a.m. EST the experiment was initiated where the materials were exposed to ram AO for 42 h at an altitude of 230 km. The resulting AO fluence was determined to be 2.3×10^{20} atoms/cm^2 (Ref. 8).

Three of the EOIM-III samples were P-100/Br epoxy composites. One was exposed unprotected to the AO, a second was protected with a thin film (few tens of nm) of SiO_2, and a third had half of the face covered with SiO_2 and half left bare. This third sample was covered with a copper foil screen so that it might react with any Br_2 that might escape as the fiber was etched away by the AO. A fourth sample was an unprotected composite of P-100 epoxy composite to judge the effects of intercalation on the degradation rate. There were two more samples made up of composites with intercalated graphite fibers less graphitic than P100 (P-75/Br epoxy and P55/Br epoxy) that were also half protected with SiO_2 and covered with a copper foil screen. Details of the fabrication of the samples and the results of their exposure are found in an earlier report (Ref. 8).

The principal conclusions drawn from that study were:

- SiO_2 protected intercalated graphite composites showed no AO erosion, verifying that conventional protection strategies are applicable to bromine intercalated composites.
- Unprotected samples showed that bromine intercalation does not alter the AO erosion rates of graphite fiber/epoxy composites.
- No bromine was detected to have migrated into the surrounding epoxy, even for highly eroded fibers and epoxy, allaying fears that bromination could compromise the properties of the epoxy.
- No corrosive bromine was detected to have been released by the fibers allaying fears that outgassing could be disruptive to the sensitive electronics the EMI shield is meant to protect.
- No effect of the crystalline order within a graphite fiber on the AO erosion rate was observed.

This was but a single test and it is possible that a lower flux exposure over a longer time could yield different results. An opportunity to gather more data on the effects of the space environment on intercalated graphite composites space presented itself when four sample opportunities opened up on the Optical Reflector Materials Experiment III (ORMatE-III) which flew on the ISS with Materials International Space Station Experiment-8 (MISSE-8) in 2011-13. The back-up samples for all four of the EOIM-III P-100 based composites, which were machined from the same composite sheets, were flown on ORMatE-III enabling direct comparison of the results.

Space Exposure

ORMatE-III was launched aboard the final flight of the Space Shuttle Endeavor (STS-134) on May 16, 2011 as part of the MISSE-8 mission. The ORMatE-III wake exposure trays were placed on ExPRESS Logistics Carrier-2 by ISS Expedition 28 NASA Astronaut Ron Garan during a spacewalk on July 12, 2011. A photograph of the ORMatE-III on orbit is shown in Figure 1 with the samples discussed in this paper highlighted. They were retrieved 728 days later by ISS Expedition 36 ESA Astronaut Luca Parmitano via EVA on July 9, 2013, and returned to Earth inside the SpaceX Dragon capsule on May 18, 2014 as part of the SpX-3 Mission. The ORMatE-III samples were part of the down-mass cargo from the mission that was returned to the Port of Long Beach via marine vessel on May 20, 2014, two days after splashdown. Sea water was found inside the Dragon capsule, possibly related to the fact that it was in the ocean 11 h before it was recovered, but preliminary checks indicated that no scientific equipment had been damaged (Ref. 9). The capsule was transferred to NASA at the SpaceX McGregor test facility in Texas (Ref. 10), where the samples were bagged in Kapron® and returned to the NASA Glenn Research Center.

The fluence of AO the samples were exposed to was determined by the erosion of Kapton® H witness coupons using well documented standard procedures (Ref. 11). As expected, the wake surface received a relatively low fluence of 8.80×10^{19} atoms/cm^2, about 1.9 percent of the ram fluence (Ref. 11). So over 2.0 years in orbit in the wake orientation, the AO fluence was about a third of the EOIM-III ram fluence that accumulated over 42 h. It must be noted, however, that the wake flux of AO is reported to be about 10^{-18} that of the ram flux (Ref. 11), so essentially all of the wake-side AO exposure came as the result of ISS reorientations such that, for short periods of time, the wake-side ORMatE-III samples faced the ram direction.

Figure 1.—On-orbit photo of ORMatE-III with the P-100 and P-100/Br epoxy composite samples circled in red.

Methods and Materials

As related earlier, the fabrication of the composite samples was detailed in an earlier publication (Ref. 12). Each sample was cut into a circle about 25 mm in diameter. A circumferential area about 2 mm wide around each sample was pressed tight under the lip of the sample holder during the flight, protecting it from direct exposure to the LEO environment, leaving an exposed diameter of about 22.7 mm, and an exposed area of 4.0 cm^2. This portion of the samples beneath the lip will be referred to as the protected area. The characteristics of the samples are summarized in Table 1.

Since 25 years passed between the characterization of the EOIM-III and ORMATe-III samples, analysis by the same instruments was not feasible, and the EOIM-III data were no longer accessible. All four samples were imaged with survey photography and optical microscopy at magnifications of 7.1×, 10×, 25×, 50×, and 100×. Four areas of the W7 sample (protected, protected/SiO_2 coated, exposed, exposed/SiO_2 coated) were imaged with a Hitachi S-4700 Field Emission Scanning Electron Microscopy (FESEM) at 250×, 500×, 1000×, and 5000× magnifications. Half of the sample was coated prior to FESEM examination with a few tens of nm of Pt to decrease the amount of charging in the electron beam. Energy dispersive x-ray spectroscopy (EDS) was also used on W7 to examine the samples for contamination. This was done in conjunction with the FESEM such that the elemental composition of specific microscopic areas of the samples could be determined.

Optical spectroscopy was performed on the W4, W5, and W6 samples to look for signs of degradation. Of particular concern was whether the thermal properties would change such as to impose an additional heat load on the structure. Total reflectivity ($\rho(\lambda)$), was measured with a Cary 5000 (Varian) spectrophotometer equipped with an integrating sphere over wavelengths (λ) from 250 to 2500 nm in increments of 1 nm, at a scan rate of 600 nm/min. A deuterium lamp was used to illuminate the samples to measure the 250 to 350 nm data, and a halogen lamp to illuminate the samples to measure the 350 to 2500 nm data. Immediately prior to measuring each sample, a spectrum of the Spectralon® reference was collected as a sample, to determine whether the baseline was still valid. In all instances the deviations in the baseline were less than 1 percent.

Since all of the incident energy must be transmitted, reflected, or absorbed, assuming the samples were opaque, their wavelength-dependent absorptivities ($\alpha(\lambda)$) can be calculated using Equation (1).

$$\alpha(\lambda) = (1 - \rho(\lambda)) \tag{1}$$

The total solar absorptance (α) was calculated by integrating the $\alpha(\lambda)$ over all values of λ, and convolving it with the ASTM air mass zero solar spectral irradiance table E–490–00 expressed as a fraction of the solar spectrum ($S(\lambda)$). In practice nearly all of the solar energy is emitted between 250 and 2500 nm, so the approximation shown in Equation (2) can be made with little loss of accuracy.

$$\alpha \approx \sum_{250\ \mathrm{nm}}^{2500\ \mathrm{nm}} \alpha(\lambda) S(\lambda) \tag{2}$$

TABLE 1.—CHARACTERISTICS OF GRAPHITE FIBER EPOXY SAMPLES FLOWN ON OREMaTE-III

Sample identifier	Fiber type	Coating	Cu screen	Analysis
M8 – W4	P-100	None	No	Spectroscopy/FESEM
M8 – W5	P-100/Br	None	No	Spectroscopy
M8 – W6	P-100/Br	SiO_2	No	Spectroscopy
M8 – W7	P-100/Br	Half SiO_2	Yes	FESEM

Results and Discussion

Survey Photography

Upon return of the flight samples to the NASA Glenn Research Center, the samples were removed from the Kapron® packaging and photographed. Figure 2 shows the visual comparison of the composite samples on exposure to the space environment. The protected areas of the samples were clearly visible as rings around their perimeters. A comparison of W4 to W5 shows that there was no difference to the eye between the exposed P-100 fiber composites and their Br_2 intercalated counterparts. A comparison of the W5 to the W6 samples showed that, to the eye, the SiO_2 coating was effective at preventing degradation in the LEO environment. This was consistent with the results of the EOIM-III study. There was, however, an apparent color change with the SiO_2 exposed area taking on a violet tint, as was true in the EOIM-III samples.

The effectiveness of the SiO_2 protective coating was shown dramatically in the W7 sample. The figure clearly shows that the coating covered the lower half of the sample. The pattern of the copper screen showed clearly on the upper half of the sample but was not seen at all on the lower half. The copper blocked AO erosion, but since the SiO_2 did as well, the pattern was not visible.

Optical Microscopy

Figure 3 shows optical microscopy of the W7 sample centered on the uncoated region. The pattern of the copper screen dominated the images. The exposed areas were much darker than those protected by the copper screen. Figure 4 shows the same sample but centered on the SiO_2 coated region. Even at 100× magnification there was no evidence of the copper screen pattern. It appeared that the SiO_2 coating was at least as effective at blocking AO erosion as the copper screen.

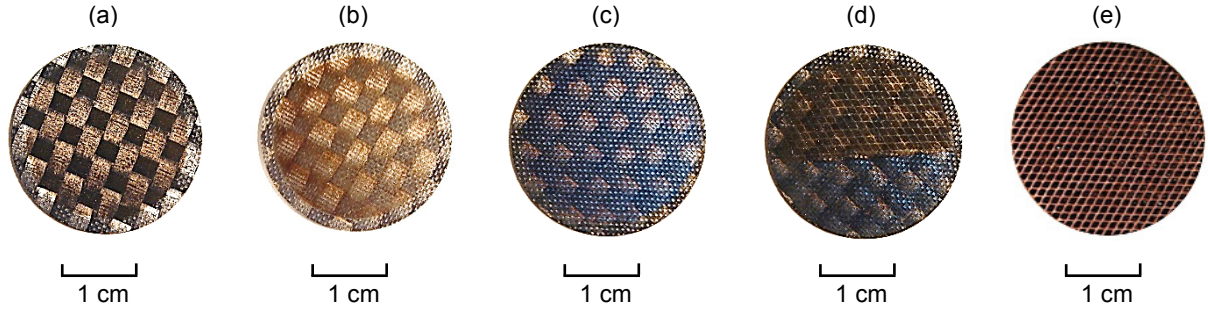

Figure 2.—Post-flight photographs of the ORMatE-III composite samples (a) M8-W4, (b) M8-W5, (c) M8-W6, and (d) M8-W7. The protected areas of the sample are clearly visible as a ring around the outside of each sample. A pre-flight photograph of (e) M8-W7 is shown with the copper screen.

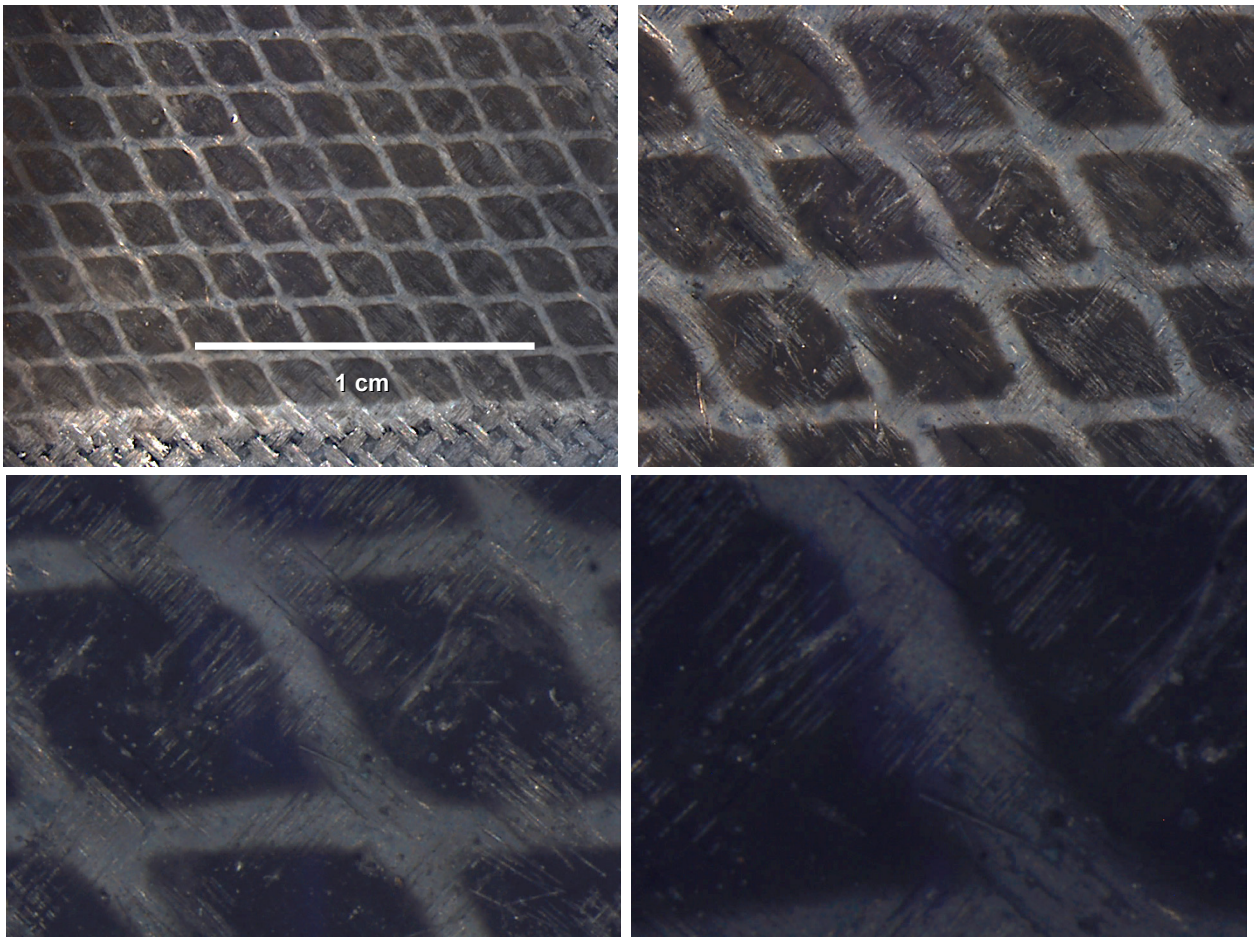

Figure 3.—Optical micrographs of the unprotected side of W7 at 10×, 25×, 50×, and 100× magnification.

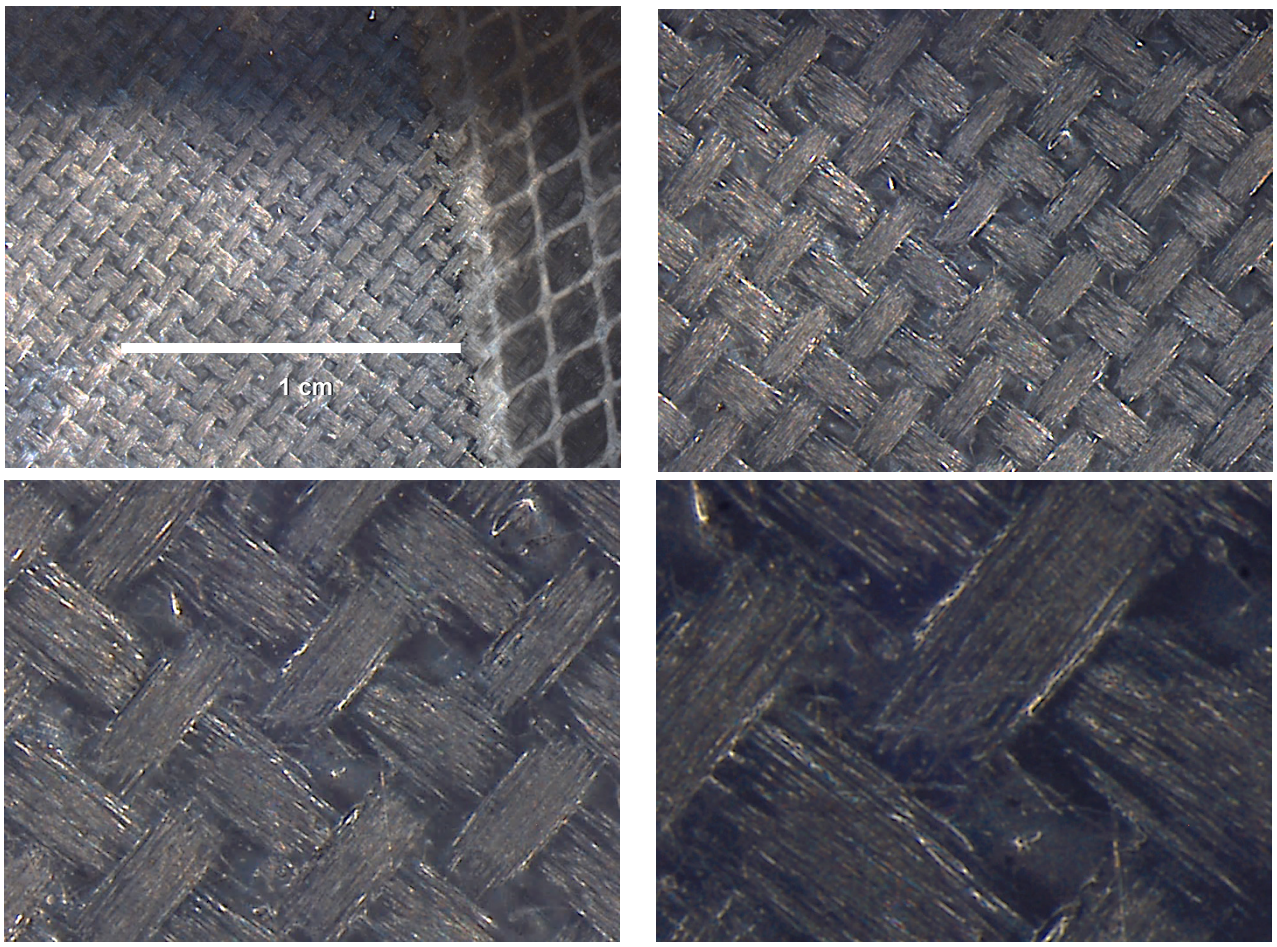

Figure 4.—Optical micrographs of the SiO_2 protected side of W7 at 10×, 25×, 50×, and 100× magnification.

Electron Microscopy

FESEM images of the protected region of the W4 sample are shown in Figure 5. The thickness of the epoxy layer varied across the surface of the woven P100 fabric. At the top of the weave the layer was quite thin and in fact the bare fiber appeared to be exposed. Such was the case for the fibers shown. Even at 20,000× magnification, no erosion or other degradation of the fiber surface were observed.

Figure 6 shows comparable photomicrographs for the space-exposed regions. There was obvious AO etching of both the fibers (Figure 6(a) and (c)) and the epoxy (Figure 6(b) and (d)), but the AO degradation was much less than was seen in the EOIM-III samples (Figure 7). This is expected because of the lower AO fluence in the ORMatE-III sample.

Both Figure 6 and Figure 7 illustrate that the epoxy eroded in the AO at a much faster rate than the fibers. This is consistent with accepted erosion yields for the two materials (Ref. 7). The cone shaped micro-texture is confirmation that the AO degradation of the ORMatE-III samples was dominated by those times when the ISS reorientation caused it to be oriented in the AO ram direction (Figure 6(d)).

The EDS spectrum of the region on the fiber indicated by the box in Figure 6(a) is shown in Figure 6(e). The spectrum was very clean showing only the carbon of the fiber and a small platinum peak from a coating applied to enable the measurement. Figure 6(f) shows the EDS spectrum from a region dominated by the epoxy. Unsurprisingly, in addition to carbon and platinum seen on the fiber, components of the epoxy, oxygen and sulfur, were also detected. There were no indications of contamination in these spectra. Perhaps the most common contaminant would be outgassing from silicones, which would be visible as a silicon peak. Not only was there no silicon peak in these samples, but there was also none found in space suit fabrics that were exposed at the same time on ORMatE-III (Ref. 13).

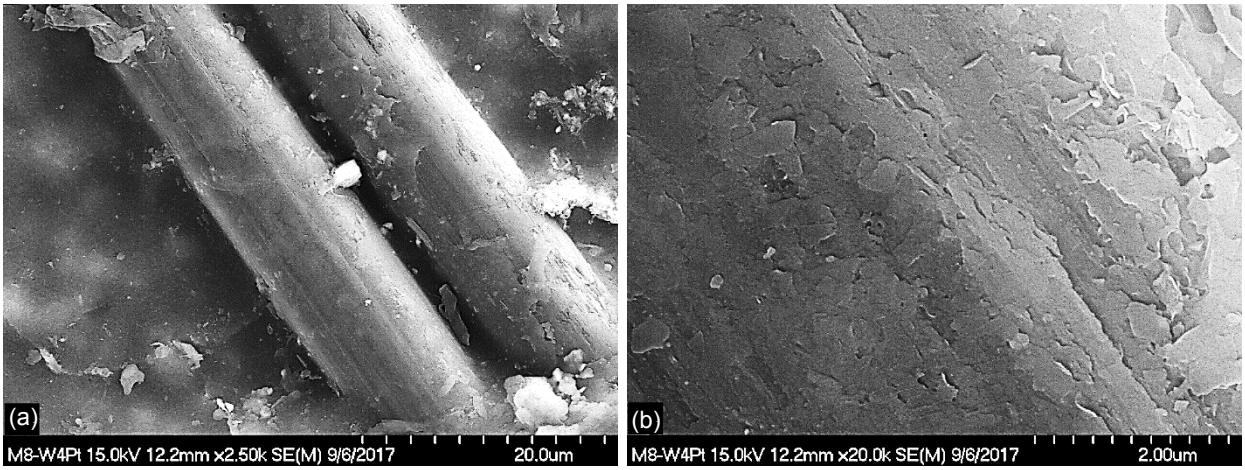

Figure 5.—FESEM images of the protected region of the M8-W4 P100/Epoxy sample at (a) 2500×, and (b) 20000×, showed no signs of degradation.

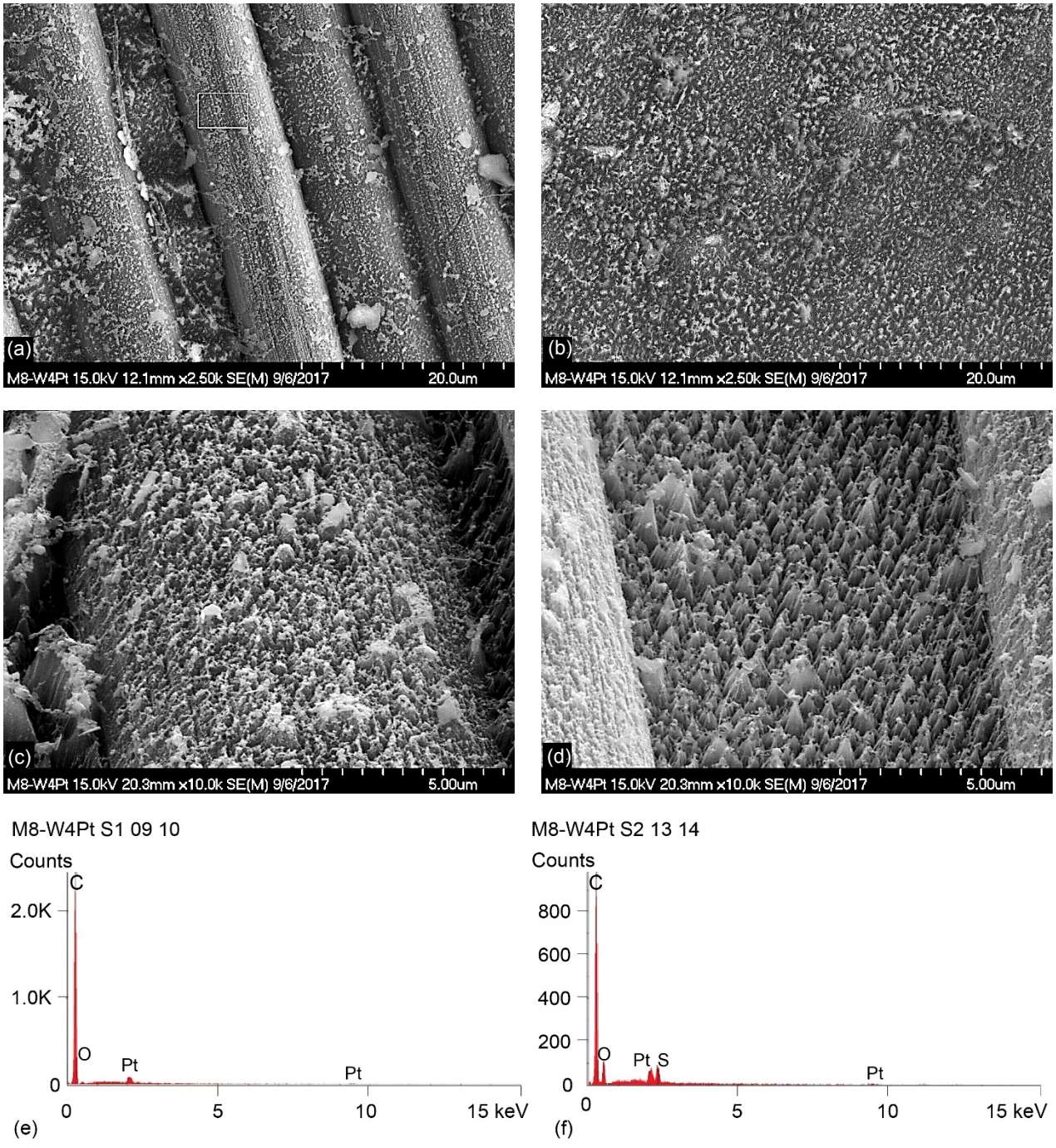

Figure 6.—FESEM images of the exposed region of the M8-W4 P100/Epoxy sample showing the P100 fiber at (a) 2500×, the epoxy at (b) 2500×, the P100 fiber at (c) 10000×, the epoxy at (d) 10000×, and EDS of the (e) P100 fiber and the (f) epoxy showing unmistakable signs of degradation.

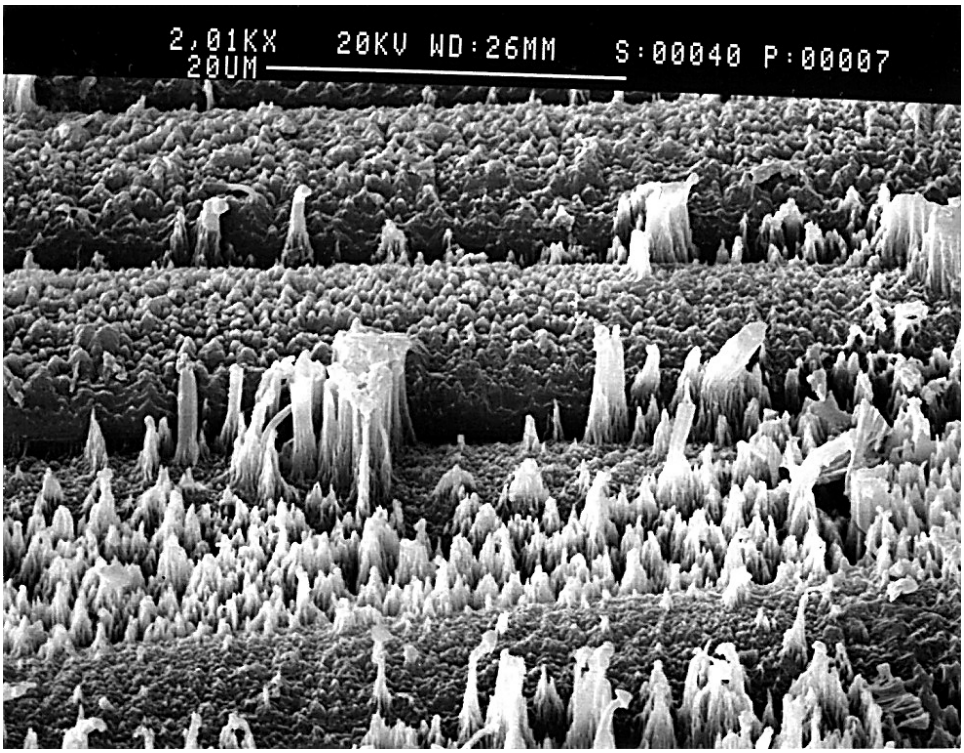

Figure 7.—P100/Epoxy sample flown on EOIM-III and exposed to 42 h of ram AO.

Figure 8(a) shows the erosion of the unprotected side of the W7 bromine intercalated graphite fiber composite sample. Comparison with Figure 6(a) reveals that the erosion rate was not qualitatively different from that of the pristine graphite fiber composite. This confirmed the conclusion of the EOIM-III study. In contrast, the SiO_2 protected composite (Figure 8(b)) showed no etching of the fiber or epoxy. This confirmed another conclusion of the EOIM-III study, that intercalated graphite fiber composites can be protected from AO degradation by a thin coating of SiO_2.

Figure 9(a) shows the EDS spectrum of the P100-Br fiber in the unprotected area showing bromine was present in the fiber in significant quantities and did not diffuse away during AO erosion. Figure 9(b) shows the EDS spectrum of the copper screen trap that was placed on top of M8-W7. Since there was no detectable bromine on the copper, if free bromine was outgassed during intercalated fiber erosion, it was not in sufficient amount to damage even very reactive copper in physical contact with it. This confirmed another of the conclusions of the EOIM-III study.

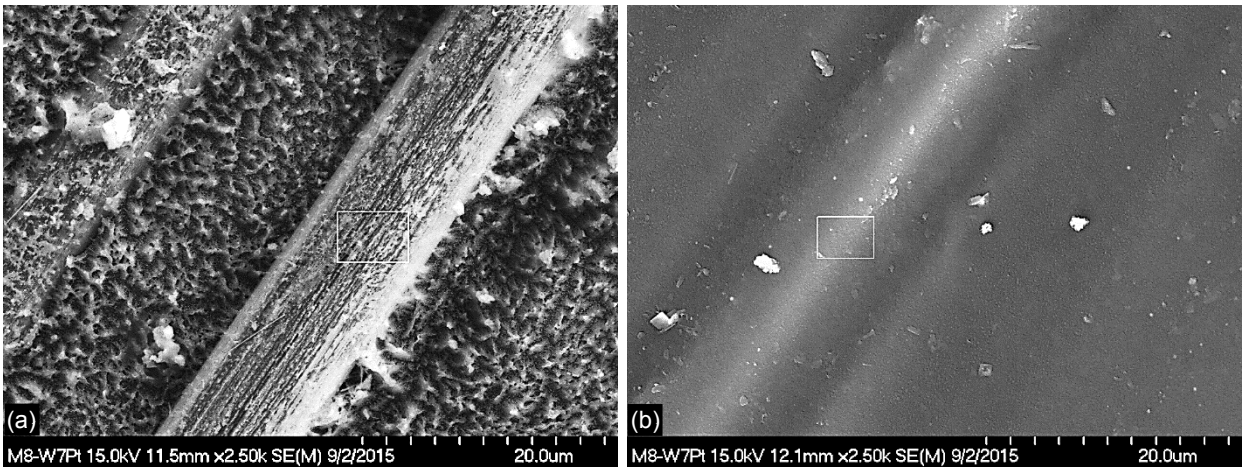

Figure 8.—FESEM images of the exposed region of the M8-W7 P100-Br/Epoxy sample showing (a) AO erosion of the P100-Br fiber and epoxy matrix at 2500×, (b) the lack of AO erosion in the SiO_2 protected regions.

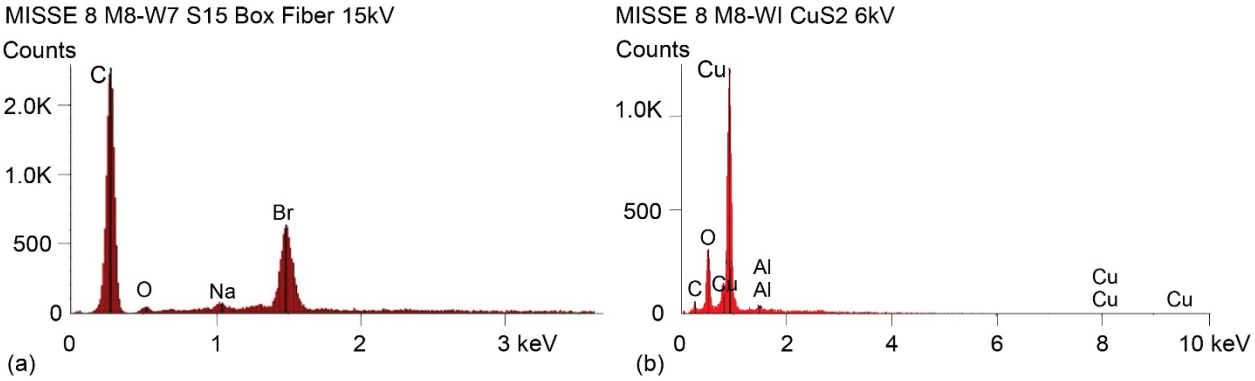

Figure 9.—The EDS spectrum of (a) P100-Br fiber showing the unmistakable presence of bromine in the eroded fibers and, (b) the absence of bromine on the copper screen that was over top of it demonstrates that insignificant amounts of bromine gas were released as the fibers eroded in the AO environment.

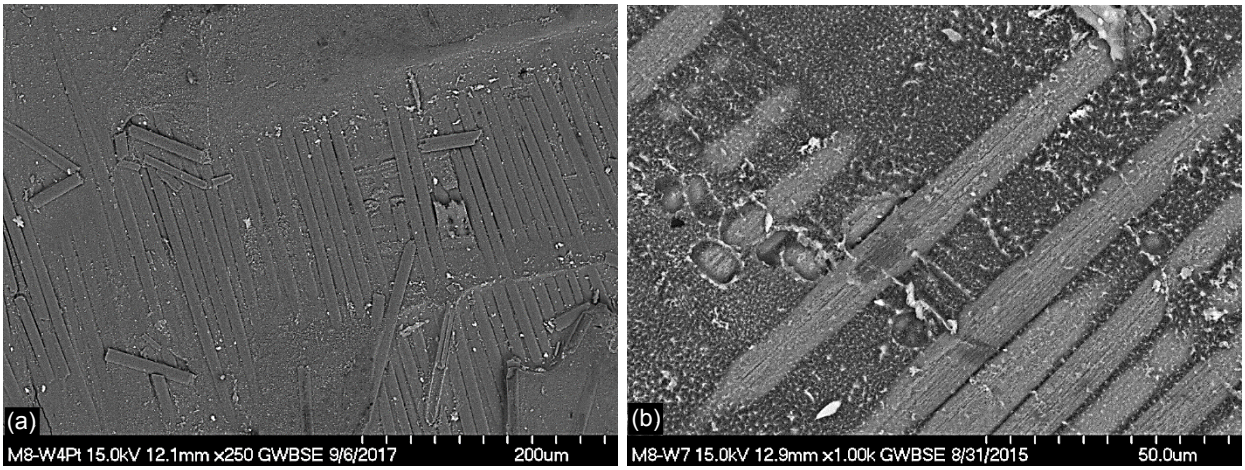

Figure 10.—FESEM backscatter images of the exposed region of the (a) M8-W4 P100/Epoxy sample and the (b) M8-W7 P100-Br/Epoxy sample. Whereas there is almost no contrast in the M8-W4 sample there is considerable contrast in the M8-W7 sample between the P100-Br fibers and the epoxy due to the presence of bromine in the fibers.

The backscatter images of the pristine fiber composite, W4, and the bromine intercalated fiber composite, W7, are compared in Figure 10. As expected, there was almost no contrast in the pristine fiber composite (Figure 10(a)) because carbon was the dominant element in both the fiber and the epoxy. However, there was considerable contrast in the intercalated fiber composite (Figure 10(b)) because bromine is a more potent backscatterer of electrons. Note the clear demarcation between the fibers and the epoxy, demonstrating that there was no significant diffusion of bromine into the epoxy even in the eroded fibers. This again confirms the findings of the EOIM-III study.

Spectroscopy

The reflectance spectrum in the 250 to 2500 nm wavelength range is important because that is the region where the sun emits over 99 percent of its intensity. Figure 11(a), and (b), show the reflectance spectrum of unprotected P100/epoxy composite (W4) and unprotected P100-Br/epoxy composite (W5) and respectively. Shown for comparison for W4 and W5 is the spectrum from the back side of the sample, which was protected from the AO and solar UV radiation environment. The analyses above showed that the elemental composition of the composites was unchanged, but a texture developed with features on the order of 0.5 μm. No new spectral features were seen in this spectral region, but both samples had increased absorption in the UV region. The pristine composite had a crossover with decrease IR absorption above about 1900 nm, whereas the bromine intercalated fiber composite had essentially the same IR absorption above 2200 nm.

Surprisingly, the data from the corresponding composites flown EOIM-III looked very different (Figure 12). The spectra that were measured before the EOIM-III flight look similar to the spectra measured of the back side of the ORMatE-III composites. But the post-flight EOIM-III spectra exhibited a decrease in the total reflection decreased by about a factor of 3 across the entire spectrum. At no wavelength did the two curves approach each other (Ref. 8).

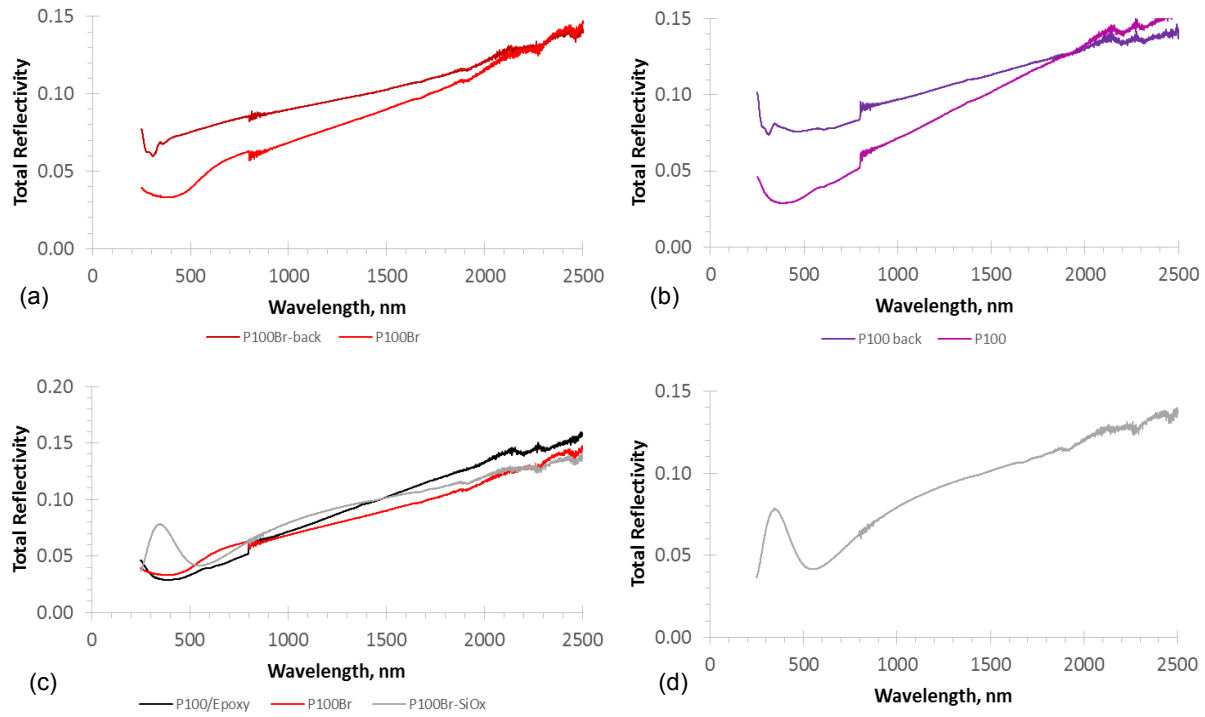

Figure 11.—The 250 to 2500 nm spectra of the composite samples flown on ORMatE-III (a) W4 P100/epoxy composite, (b) W5 P100-Br/epoxy composite, (c) W6 SiO_2-coated P100-Br/epoxy composite, and (d) comparison of the three.

Figure 11(c) shows the spectrum of the SiO_2-coated P100-Br/epoxy composite (W6). Note the strong reflection feature that peaks around 350 nm. This UV-centered peak tailed into the visible part of the spectrum and was responsible for the violet color noted in the survey photography. Figure 11(d) shows spectra of the three composites exposed to the LEO environment superimposed. It is interesting to note that, despite the texturing of the unprotected composites, the reflection spectra are nearly the same, except for the region around the 350 nm SiO_2 peak.

Contrary to the unprotected P100/epoxy and P100-Br/epoxy spectra above, the spectrum of the SiO_2-coated P100-Br/Epoxy composite in the EOIM-III looked similar. This raised the question of why were the spectra of the unprotected composites so different, and the protected composites similar. It seems unlikely that instrument or procedural differences between the experiments conducted 20 years apart would account for the differences since the pre-flight EOIM-III and the back-side ORMatE-III were similar and the post-flight SiO_2-protected composite spectra were similar. Thus it seems likely that there were real spectral differences between unprotected composites exposed to 42 h of ram AO and to one-third that in the ORMatE-III samples decreased the solar reflection significantly, particularly in the infrared.

The α calculated from the ORMatE-III composites spectra were nearly identical. Both pristine and intercalated uncoated samples had an α of 0.94 and the SiO_2 coated sample had an α of 0.93. Regarding the thermal properties, utilizing bromine intercalated fiber composites and/or coating the composite with SiO_2 had no practical effect. The α of the EOIM-III samples was not calculated, but it is clear from the spectrum that the α increased slightly, probably to about 0.96 to 0.98.

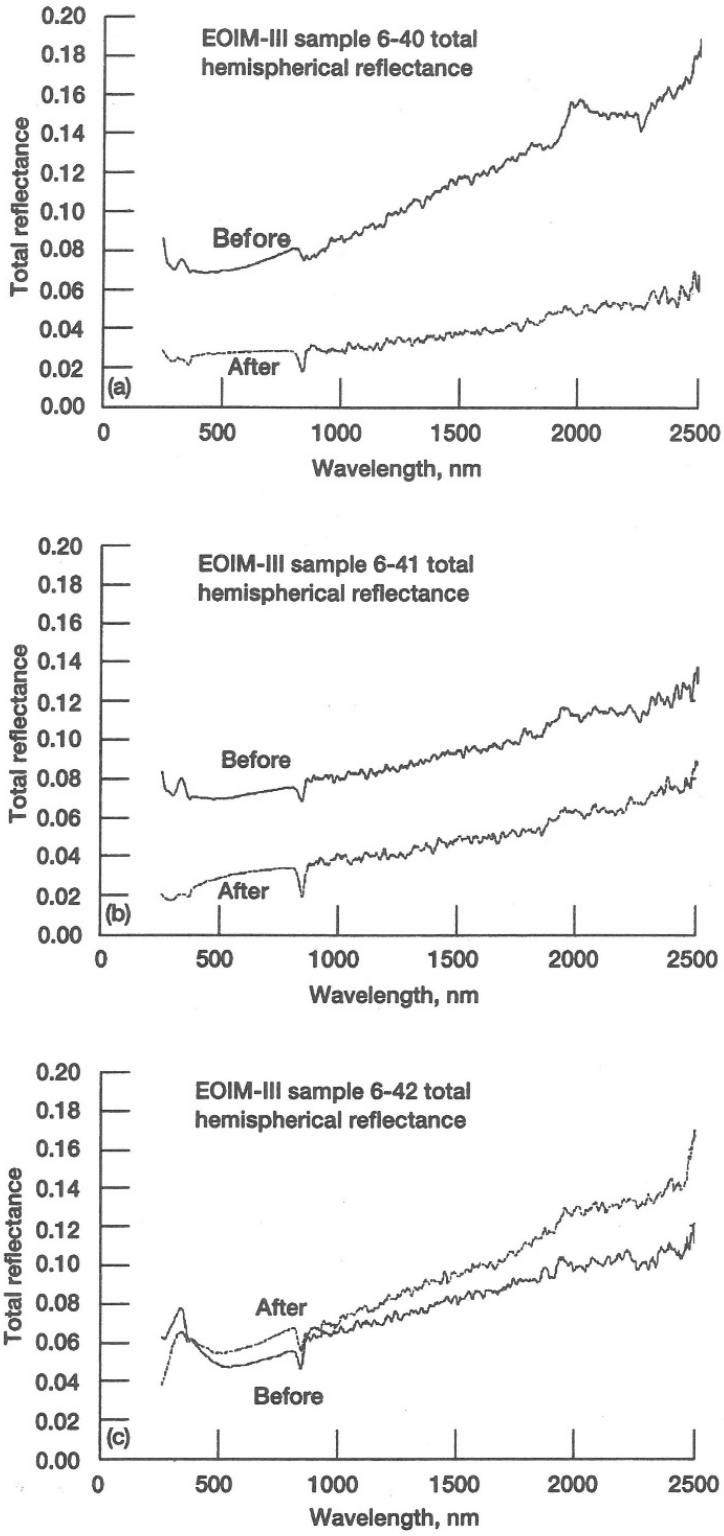

Figure 12.—The 250 to 2500 nm spectra of the composite samples flown on EOIM-III (a) P100/epoxy composite corresponding to W4, (b) P100-Br/epoxy composite corresponding to W5, (c) SiO_2-coated P100-Br/epoxy composite corresponding to W6. Reproduced from Reference 8.

Conclusions

Four different graphite fiber/epoxy composites were exposed to the wake-side LEO environment on the ORMatE-III platform mounted on the exterior of the ISS for two years in order to determine their long term durability in the space environment. Three of the samples used bromine intercalated P100 fibers and one used pristine P100 fibers. One of the P100-Br samples was coated with a few tens of nm of a protective SiO_2 layer, and half of another was coated with SiO_2. The half coated sample had a copper screen over the top of it to react with and capture any corrosive bromine that might outgas.

Results were compared with the EOIM-III experiment which exposed the same materials in the LEO ram direction for 42 h on the Space Shuttle. Although the AO fluence of the ORMatE-III samples was one-third of the EOIM-III exposure, the resulting effects were qualitatively the same. Both experiments found that SiO_2 coated intercalated graphite composites showed no AO erosion, verifying that conventional protection strategies are applicable to bromine intercalated composites. Both found that bromine intercalation does not significantly alter the AO erosion rates of graphite fiber/epoxy composites. Both found no bromine was detected to have migrated into the surrounding epoxy, even for highly eroded fibers and epoxy, allaying fears that bromination could compromise the properties of the epoxy. And both found no corrosive bromine was detected to have been released by the fibers, allaying fears that outgassing could be disruptive to the sensitive electronics.

The principal difference was that the ram AO exposure of unprotected samples showed a somewhat deeper erosion which manifested as a difference in the infrared spectrum out to 2500 nm. Although the α of the exposed pristine and intercalated composites of the EOIM-III samples was not measured, it was estimated to be about 0.96 to 0.98 compared with that measured to be 0.94 for the corresponding ORMatE-III samples.

The general conclusion is that ram AO exposure had very similar effects on graphite fiber epoxy composites in both the EOIM-III and ORMatE-III experiments, and in neither case did bromine intercalation of the fibers substantially influence that degradation.

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