Evaluation of an ionic liquid-based epoxy after exposure on the MISSE-8 Carrier

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\textbf{Abstract}

An ionic liquid-based epoxy was evaluated after more than two years of continual nadir space exposure on the MISSE-8 sample rack outside of the International Space Station. In addition to space radiation, atomic oxygen and vacuum space exposure the samples also experienced approximately 12,500 thermal cycles between \(-40^\circ\text{C}\) and \(+40^\circ\text{C}\). The returned samples exhibited no cracking or de-bonding from the aluminum discs to which the epoxy was initially applied; there was a slight change in color, and a minuscule variance in before-and-after weight was measured. Microscopic examination revealed some slight deformities, dimpling, and deposits on the exposed surfaces. These are put into the context of an on-going effort to develop viable carbon-fiber based composite tanks for, but not inclusively, cryogenic liquid containment.

\textbf{Introduction}

The Materials International Space Station Experiment-8 (MISSE-8) was deployed on May 20th, 2011 on the Shuttle Transportation System-134 (STS-134) and was retrieved July 9th, 2013. It was located on the Express Logistics Carrier-2 (ELC-2). Included on MISSE-8 were two trays with 92 sites, each \(25\text{ mm}\) in diameter, where samples could be attached with the goal of determining how the space environment affected the exposed material, Fig. 1. These trays experienced 2 years and 2 months of nadir exposure (facing the earth) and were subjected to an oxygen fluence of \(3.6 \pm 0.1 \times 10^{19}\text{ atoms/cm}^2\), 12,500 cycles between approximately \(-40^\circ\text{C}\) and \(+40^\circ\text{C}\), a high vacuum environment, and radiation\textsuperscript{[1]}. Four of the tray sites contained a novel ionic-liquid based epoxy\textsuperscript{[2]} spread on an aluminum disc; their positions, pre- and post-flight, are outlined in Fig. 1. Results for both sets are essentially the same but focus is directed to the reddish samples on the left hand side as that particular epoxy is part of an on-going investigation.

\textbf{Results and discussion}

ISS sample ILEP15 is shown in Fig. 2. There is a difference in color between the center of the sample, which was exposed to the space environment, and the unexposed sample edge that was covered by a retaining ring. Energy dispersive spectroscopy (EDS) along the exposed/unexposed boundary showed negligible variations in composition. Because of the similar compositions, the color change is attributed to ultraviolet (UV) radiation. Visual observations showed no cracking or deformation of the epoxies, and there is a continued strong adherence of the epoxy to the aluminum base. Pre and post flight weight measurements of ILEP15 (initial = \(2.3549\text{ g}\)) and ILEP17 (2.3294 g) showed a weight loss of, respectively, 0.0002 and 0.0004 g.

Scanning electron microscopy (SEM), showed some nano-scale surface dimpling on the flight samples (Fig. 3a) but not on the ground ones (Fig. 3b). Electron Spectroscopy Chemical Analysis (ESCA) showed a comparable chemistry of the flight samples to the ground samples, though there were some breaking of nitrogen (N) bonds (Fig. 4) on the surface, when compared to the ground sample. Cl, F, O, and S are inherent to the epoxy. Na peaks are attributed to NaCl crystals that are randomly observed by SEM on the sample. Some small areas of surface (likely space) contamination were also observed in which Si was detected. Bond breaking, and the dimpling, could be due to impingement by atomic...
oxygen (AO), though surface effects attributed to AO in the other MISSE samples had a streaked appearance.

Summary and conclusions

Initial observations of the flight samples revealed a miniscule weight change and no mechanical-type deformation; strong adherence to the aluminum base was also maintained. Overall, the ionic liquid epoxy appeared to well tolerate the space environment in which the experiment was conducted, a desired attribute for potential cryogenic fluid storage tanks [3]. It is noted, however, that this was for a nadir orientation and different results might be expected for “ram” and/or “zenith” exposures.

Fig. 1. MISSE-8 sample trays pre-flight and post-flight, with the ionic epoxy samples highlighted.

Fig. 2. Sample “ILEP15” showing a darker exposed region, denoted by the dashed blue line, and the lighter area which was covered by the sample lockdown ring.

Fig. 3. SEM image of the surface of (a) flight and (b) ground/control samples.

Fig. 4. (left) ESCA analysis for the flight and ground samples and (right) magnified view of N peak separation. ILEP13 is a ground control sample; ILEP17 is an ISS flight sample. Inflections in the ILEP17 peak correspond to the two ILEP13 peaks. “Smooth” and “Cut” also refer to ground samples, but not specific to this investigation.
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References

