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Atomic Oxygen Exposure of Power System and Other Spacecraft Materials: Results of the EOIM-3 Experiment

Thomas L. Morton
NYMA, Inc.
Brook Park, Ohio

and

Dale C. Ferguson
Lewis Research Center
Cleveland, Ohio

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National Aeronautics and
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Thomas L. Morton
NYMA, Inc.
Brook Park, Ohio 44142

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Dale C. Ferguson
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

INTRODUCTION

Atomic oxygen degradation has been recognized as a possible Low Earth Orbit (LEO) spacecraft problem since the flight of STS-3, the first Shuttle mission to last longer than a few days. When camera thermal coverings composed of Kapton returned from orbit with an unexpected matte finish, contamination was at first suspected. However, rather than gaining mass as in a contamination event, this material was found to have lost mass. Examination under a Scanning Electron Microscope showed that the Kapton had taken on a carpetlike texture with a thickness of about a micron, except where it was shadowed from direct impingement of the ram flow. It is believed that atomic oxygen (AO) was responsible for the degradation.

Later, it was shown that Kapton and other materials lose mass through loss of volatile oxidation products, such as carbon monoxide, carbon dioxide, nitrogen dioxide, etc. Further, the rates of mass-loss were seen to be increased by the impact energy of the incoming AO in LEO, about 4.5 eV at LEO circular orbital speeds (ref. 1). Ferguson and others (refs. 2 and 3), using ground and flight test data, showed that for Kapton, the mass-loss rate is approximately proportional to the 0.6 power of the impact energy. Eck and his students (ref. 4) found a mass-loss rate for carbon which is approximately proportional to the 0.3 power of the impact energy. For other materials, only limited ranges of impact energy have been tested, so it is not known how universal such empirical mass-loss rate laws may be.

Some super-oxidized materials, such as magnesium fluoride solar array coatings, show no further oxidation in LEO. Other materials, such as silicon and aluminum, form protective oxide layers that resist further oxidation. In general, these materials gain some mass upon LEO AO attack. Still others, such as silver, form oxide layers that spall off and are lost, leaving unprotected surfaces to undergo further oxidation.

The effect of surface temperature on the mass-loss rate of materials is not known in many cases. It might be argued that the 4.5 eV atomic oxygen impinging on LEO spacecraft is effectively similar to a 52 000 K temperature thermal source, making comparatively small changes of surface temperature (on the order of a few hundred K) seem unlikely to be important. If it is important that the oxygen reside on or in the material for a time before the oxidation can take place, surface temperatures might be important in regulating the rate of desorption from the surface, or the rate of diffusion into the surface, or both. Ground tests have confirmed that surface temperature can be important for some materials (ref. 5).

Future spacecraft designs employing high temperature, high strength materials for solar or nuclear heat sources in combination with high temperature energy conversion systems have not been evaluated with respect to AO degradation. Of particular interest are candidate structural refractory metal alloys and superalloys, candidate thin film refractory metal coatings for space radiators, multi-layer insulation materials, and candidate seal materials. Although some of these candidate materials have been or will be exposed to AO in ground test facilities, it is important to correlate these results with those achieved on orbit where there is a collimated flux of ground state AO.

For these reasons, it was decided to test in orbit a number of material samples of interest to spacecraft power system designers. Furthermore, the samples were flown on two trays held at different elevated temperatures to evaluate the effect of surface temperature on the reaction rates. We also flight-tested other materials of general interest to spacecraft designers, such as space-suit materials, diamond-like films, etc., that were known to react with AO (refs. 6 and 7), to obtain better data and to provide calibration with ground-test results.

The EOIM-3 (Evaluation of Oxygen Interactions with Materials) flight experiment was performed on STS-46 in August of 1992. This experiment was designed to determine the effect of AO on a variety of spacecraft power system materials. The materials selected for this test included refractory metals, superalloys, radiator coatings, and a range of polymers (see Table I).

Before flight, the materials were characterized by mass, visual inspection, and surface reflectance (both in visible and infrared regions). Postflight analysis repeated the preflight tests. In addition, 16 material samples underwent scanning electron miscroscopy (SEM), and 19 metal samples were tested by Auger spectroscopy.

This paper documents the results of the evaluation of these samples. Because of the large quantity of samples, the paper is organized as follows. In the body of the paper is a summary of the results, listing some of the highlights of each of the types of samples flown. Then, the description of each sample, with it's individual test results is listed in the tables. In the appendices are the reports separated by test, i.e. mass, visual inspection, Auger analysis, and SEM results.

Description of Experiment

EOIM-3 was activated at the end of the STS-46 mission. During that portion of the flight, the Shuttle was flown in a bay-to-ram configuration at 124 nautical miles (229 km) for 42 hr. Preliminary mass spectrometer readings indicate a fluence of 2.3×10^{20} AO atoms per square centimeter, a value that is consistent with the predictions of MSIS-86 neutral atmosphere model for the flight conditions (ref. 8).

This portion of the experiment consisted of two sets of 22 samples. The first set (designated A) was exposed to the ambient atmosphere while heated from behind to 60 °C. The second set (designated B) was heated to 200 °C. The two temperatures allow us to examine the temperature dependence of the reaction rates. Each sample consisted of a disk approximatelyinch in diameter. When placed in the sample holder, a 3/8 in. diameter region was exposed, resulting in an area of approximately 0.713 cm^2, or a fluence of 1.64×10^{20} oxygen atoms per sample.

SUMMARY OF RESULTS

Atomic oxygen exposure test results are shown in Table II. In general, the metals showed no change in mass, but each one showed a different color change. There was a measurable difference in the surface reflectance between pre- and post-test samples that is presented qualitatively. Auger analysis indicated that most of the metals showed an increase in surface oxygen, and a decrease in surface carbon. However, the Tungsten-Niobium composite and PWC-11 showed a slight loss of surface oxygen. SEM showed no physical evidence of oxidation products, although at high magnifications there was often a hint that the surface had changed.

The two superalloy materials (Inconel 718 and Udimet 720) both showed a loss of the chromium Auger signal in the post-flight samples. This is surmised to be an example of chemisorption-induced surface segregation (ref. 9). That is, in nickel chromium alloys undergoing high temperature oxidation, the two metals separate into two layers, with the NiO forming about a 500-nm layer on top of the surface, thus hiding the Cr_2O_3 layer underneath.

None of the radiator coatings showed any detectable change after the flight. However, two of the higher temperature samples separated from their base, making mass change estimates difficult to quantify. Three of the four types of coatings showed a smaller change in surface reflectance than their controls did.

The polymer samples showed more dramatic changes between pre- and post-flight. Most of them showed stunning visual changes, both in shading and texture. The large mass losses listed in Appendix A indicate projected lifetimes ranging from a few weeks to about a year at the exposure rate of the experiment. The SEM images showed distinct patterns in the surfaces, and clearly demonstrated major surface changes. Where both A and B samples were the same, the control is designated by C. If A and B were different, then the control for A was D, and the control for B was C.

Metals do not react significantly to 2.3×10^{20} atoms of AO exposure. However, most of the metals showed a color change, which was more pronounced for the higher temperature samples. The polymers generally showed behavior similar to other tests, and show a markedly shorter lifetime in the AO environment than metals.

Description of Appendices

The Mass Loss Table in Appendix A shows pre- and post-flight mass, as well as the difference, in grams. Additionally, there is a fractional change column, and a list of the mass loss per incident atom (in atomic mass units).

Appendix B documents the visual inspection observations of Ferguson and Morton, performed shortly after the samples were returned to Lewis Research Center. Any anomalies that had been seen in the pre-flight observation are so noted.

Appendix C presents the Auger analysis carried out by Zorman, *et. al.* at Case Western Reserve University. Tables II and III in that Appendix list the oxygen and carbon signals detected for the control and two flight specimens.

Appendix D presents the SEM examination carried out by Smith, a Resident Research Associate at NASA Lewis Research Center. This appendix summarizes the findings of his analysis of 16 samples.

The spectra referred to in Appendix C, and photographs in Appendix D, can be obtained from the authors. They were too numerous to include in this paper.

ACKNOWLEDGMENTS

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REFERENCES

1. D. C. Ferguson, "Laboratory Degradation of Kapton in a Low Energy Oxygen Ion Beam" (NASA TM–83530) 1983.
2. D. C. Ferguson, "The Energy Dependence and Surface Morphology of Kapton Degradation Under Atomic Oxygen Bombardment" (NASA CP-2340) 1984, pp. 205–221.
3. C. C. Horton, T. G. Eck, and R. W. Hoffman, *J. Vac. Sci. Technol. A*, 4, (1986), 1236.
4. C. C. Horton, "Pulsed Ion Beam Investigation of Surface Reactions" (Ph.D. thesis, Case Western Reserve University) 1988.
5. D. C. Ferguson, "Atomic Oxygen Effects on Refractory Materials" in *Material Degradation in Low Earth Orbit*, ed. V. Srivinasan and B. Banks, Minerals, Metals, and Materials Society, 1990, pp 97–105.
6. W. L. Miller, "Mass Loss of Shuttle Space Suit Orthofabric under Simulated Ionospheric Atomic Oxygen Bombardment" (NASA TM–87149) 1985.
7. C. C. Horton, T. G. Eck, and R. W. Hoffman, "Pulsed ion Beam Investigation of the Kinetics of Surface Reactions", *J. Vac. Sci. Technol. A*, 7, (1989) pp. 2143–2146.
8. S. L. Koontz, L. J. Leger, J. T. Visentine, D. E. Hunton, J. B. Cross, C. L. Hakes, "An Overview of the Evaluation of Oxygen Interactions with Materials III Experiment: Space Shuttle Mission 46; July-August, 1992" Third LDEF Post-Retrieval Symposium, Williamsburg, VA, Nov. 1993.
9. M. Thompson, M.D. Baker, A. Christie, & J.F. Tyson, "Auger Electron Spectroscopy", Vol 74 in Chemical Analysis Series, pp 297–299, 311.

TABLE I.—NASA LEWIS EOIM-3 TEST SAMPLES

Candidate high temperature system construction materials	Candidate multi-layer insulation materials	Candidate radiator coating materials	Miscellaneous materials
Niobium 1 percent Zirconium Tungsten-Niobium composite Molybdenum 13 percent rhenium PWC-11[1] 6061-T6 Aluminum 304 Stainless steel Inconel 718[2] Udimet 720[3]	Tungsten Molybdenum	GE black on Nb–1 Zr Rokide C/Ti/Al[4] BN paint on Al type A Eu_2O_3/Al	Space suit material Space suit material upside-down Brass Diamond-like film Polypropelene mesh 1/16 in. FEP Teflon disk[5] Polycarbonate Titanium Viton fabric seal material[5] 1/2 mil FEP Teflon on 1 mil Kapton[5] 1 mil Kapton[5]

[1] Trademark of Pratt Whitney Corporation.
[2] Trademark of Inco Alloys International.
[3] Trademark of Special Metals Corporation.
[4] Trademark of Norton Company.
[5] Trademark of E.I. DuPont de Nemours, Inc.

TABLE II.—NASA LEWIS EOIM-3 TEST SAMPLES
[Summary of results.]

Sample	Mass change	Visual change	AES results	SEM results	Surface reflectance
Nb 1 % Zr	No detectable change (<0.1 mg).	Brown crescent discoloration on center of B. Dark discoloration on outside rim of A.	Oxygen signal +33 percent on A, +12 percent on B Carbon signal –39 percent on A, –49 percent on B	No significant surface chemical differences detected.	Gain of some blue reflectance on sample B.
Inconel 718	No detectable change (<0.1 mg).	Faint yellowish discoloration, stronger on B than A. Detectable shadow on both.	Oxygen signal +15 percent on A, +23 percent on B Carbon signal –49 percent on A, –50 percent on B	Signs of oxygen enrichment, but no physical evidence of oxides at 20 000x.	Gain of reflectance across spectrum, stronger on A then B.
Tungsten-Niobium composite	No detectable change (<0.1 mg).	A dull. Both have black rim discoloration. B shows strong brown discoloration in center, with shadow. Some pattern I B – possibly scratched?	Oxygen signal –8 percent on A, –12 percent on B Carbon signal –47 percent on A, –52 percent on B	All samples show fine irregular patches of carbonaceous material, with sample B showing more. No evidence of micro-structural changes.	Sample B shows an increase, stronger at blue than red.
Mo 13 % Re	No detectable change (<0.1 mg)	Mottled gray discoloration, stronger on B, spotted on A. Strong shadow, stain looks washed in, under shadow?	Oxygen signal +25 percent on A, +17 percent on B Carbon signal –76 percent on A, –84 percent on B	Both flight samples show this incrustations of oxide, both at the rim and across the surface. Sample B is more strongly affected. High magnification reveals particulates, even in areas which appear clean.	Sample B shows a stronger reflectance increase than A, with the in-crease happening more at the blue end of the spectrum.
PWC–11	No detectable change (<0.1 mg)	A looks just like C. B has nicotine color with sharp shadow.	Oxygen signal –14 percent on A, –14 percent on B Carbon signal –58 percent on A, –74 percent on B	Oxidation evident, but no obvious oxidation products visible, event at 20 000x.	Strong increase in blue reflectance in sample B, sample A shows little change.
GE–Black	No detectable change (<0.1 mg), however, sample B fell apart, indicating adhesive failure.	Faint white crescent on B– all the way to edge, so not space related (also seen pre-flight).			Flight samples more similar than the control, but little change seen.
Rokide C	No detectable change (<0.1 mg), except on sample B, which fell apart, indicating adhesive failure.	B detached from substrate. No visible differences.			Flight samples more similar than the control, but little change seen.
BN paint–Type A	No detectable change (<0.1 mg)	More specular reflectance under rim of holder. A and B appear smoother.			Flight sample showed a loss in reflectance, mostly at the blue end of the spectrum.

TABLE II.—Continued

Sample	Mass change	Visual change	AES results	SEM results	Surface reflectance
Europium oxide	No detectable change (<0, 1 mg).	All three look the same specularly. Pit on B half-way from center. A shows faint stain.	Oxygen signal –25 percent on A, –25 percent on B Carbon signal –51 percent on A, –51 percent on B	No significant surface chemical differences detected.	Difficult to judge, due to interference caused by thin film.
Brass	No detectable change (<0.1 mg).	B looks browner, both dirty. Dark discoloration at rim. Sharp shadow. C may have fingerprint.	Oxygen signal +43 percent on B Carbon signal –67 percent on B		Flight sample showed gain of reflectance in red region.
Tungsten	No detectable change (<0.1 mg) on B and C. Sample A showed increase of 0.9 mg.	A has central diffuse blob. B has circle. A and B both still shiny.	Oxygen signal +51 percent on A, –40 percent on B Carbon signal –77 percent on A, –85 percent on B	Post-flight samples indicate rim delineations and oxidation product in surface defects. Micrographs indicate higher oxidation of sample B. Some evidence of contamination on sample A.	Sample A shows substantial loss of total reflectance, but diffuse reflectance unchanged in all samples.
Molybdenum	No detectable change (<0.1 mg).	Dirty bluish discoloration in center of A and B. Sharp shadow, dark discoloration on edge.	Oxygen signal +51 percent on A, –40 percent on B Carbon signal –77 percent on A, –85 percent on B	Some evidence of mottling, which may be post-flight oxide. Cannot be confirmed due to small size. Several spots observed on sample B, which appear oxygen rich.	Flight samples showed loss of total and diffuse reflectance, somewhat less at blue end of spectrum.
6061–T6 Aluminum	No detectable change (<0.1 mg) on A and C. Sample B showed loss of 0.1 mg.	A has discoloration, visible shadow. Both A and B appear dirty.	Oxygen signal +30 percent on A, +40 percent on B Carbon signal –61 percent on A, –46 percent on B	Exposed regions of post-flight samples show no significant mircostructural change. Sample B shows slight increase in surface oxygen, but appears to be concentrated in surface recesses.	No detectable changes.
304–Stainless steel	No detectable change (<0.1 mg).	B has yellowish stain, sharp shadow. A has shiny ring around center (also seen pre-flight).	Oxygen signal –4 percent on A, +12 percent on B Carbon signal –49 percent on A, –55 percent on B	EDX comparisons of shadow region do not reveal any apparent fluctuations in surface chemistry.	Some loss of diffuse reflectance, mostly at blue end of spectrum.
Diamond-like film	No detectable change (<0.1 mg) on A and C. Sample B lost 0.2 mg.	A and B have lost blueness, still shiny.		Sample A shows film degradation in isolated regions. There is hardly any film left on Sample B.	Flight samples lost mostly red reflectance, both specular and diffuse.
Udimet 720	No detectable change (<0.1 mg).	A has black ring at edge. C a little shinier.	Oxygen signal +85 percent on A, +92 percent on B Carbon signal –58 percent on A, –60 percent on B	No evidence of oxidation at 20 000x. Evidence of contamination, possibly skin contact beneath retainer rim.	Slight decrease of diffuse reflectance in flight samples.
Polypropylene mesh	Flight sample lost 0.357 mg (1.8 percent), control mass did not change. Approximately 0.0359 propylene units per incident oxygen atom.	A looks duller, strands are more uniform. Has lost some of its roughness.			

TABLE II.—Conclusion

Sample	Mass change	Visual change	AES results	SEM results	Surface reflectance
FEP Teflon	Both flight and control samples lost 0.1 mg.	B is shinier, smoother. May have a shadow.		Sample B shows heavy surface abrasion. All three samples show evidence of barium sulfate particles, probably from mold release agent. Evidence of regions of unreactive regions on surface, surrounded by heavy abrasions.	Flight sample shows ~10 percent loss of diffuse reflectance.
Polycarbonate	Flight sample lost 0.5 mg, control change undetectable. Approximate 0.2 carbons per incident oxygen.	Surface of A is milky, clear shadow. Very thin layer of milkiness. Has a short scratch.			Flight sample shows a 50 percent loss of specular reflectance.
Titanium	No detectable change (<0.1 mg).	Noticeable yellowish discoloration in center of B, loss of specularity.	Oxygen signal No change on B Carbon signal –67 percent on B	Oxide on flight sample present at about the same level as control. No change in surface morphology.	Flight sample showed loss of diffuse reflectance in blue region of spectrum.
Viton fabric	Sample A lost 0.163 mg, Sample B lost 0.764 mg, Control gained 0.008 mg.	B has color difference in center, visible shadow.		Only subtle changes apparent in Sample A. On Sample B, majority of polymer granules on surface have been eroded, leaving shadows on surface. Across exposed surface, there is a fine scale surface degeneration, conspicuous as pores and cavities generally less than 1 μm in diameter.	Sample B showed about a 20 percent increase in specular reflectance.
FEP on Kapton	Flight samples lost no more than control (~0.02 mg).	B has visible shadow. No other visible differences.		Sample A shows signs of scattered debris. A filamentary surface texture becomes apparent in the exposed regions above 500x. Sample B shows a distinct ring shadow. Also apparent are numerous abrasions to the exposed surface. Above 5000x, a highly directional etch pattern is evident.	Flight samples showed ~20 percent increase in blue region of specular reflectance.
Kapton	Sample A lost 0.625 mg, Sample B lost 1.00 mg, Control gained 0.01 mg.	A and B very milky, clear shadow. Loss of specular reflectance.		Typical carpet-like erosion pattern seen on both flight samples. In addition, there is a strong grooved pattern that appears at 200x. The grooves are wider and deeper on Sample B, indicating stronger erosion. Both flight samples show evidence of a spider-web structure on surface, which protects the underlying layers	Both flight samples showed a gain in specular reflectance in the red region of the spectrum, and a larger gain in diffuse reflectance.

APPENDIX A

Mass Measurements

(All masses listed in grams)

		Pre-flight Mass	Post-flight Mass	Change in Mass	Fractional Change	AMU per inc. O
Nb–1 Zr	A	2.04467	2.04468	0.00001	6.52×10^{-6}	
	B	2.03408	2.03401	–0.00006	-3.11×10^{-5}	
	C	2.02840	2.02832	–0.00008	-4.11×10^{-5}	
Inconel 718	A	1.23051	1.23048	–0.00002	-1.90×10^{-5}	
	B	1.24462	1.24457	–0.00005	-4.02×10^{-5}	
	C	1.22293	1.22286	–0.00007	-5.45×10^{-5}	
W/Nb Composite	A	3.87358	3.87349	–0.00009	-2.32×10^{-5}	
	B	3.81699	3.81690	–0.00009	-2.36×10^{-5}	
	C	3.90764	3.90755	–0.00009	-2.22×10^{-5}	
Mo 13 Re	A	1.51099	1.51095	–0.00004	-2.65×10^{-5}	
	B	1.50629	1.50635	0.00006	3.76×10^{-5}	
	C	1.50473	1.50469	–0.00004	-2.66×10^{-5}	
PWC-11	A	1.09083	1.09080	–0.00003	-3.06×10^{-5}	
	B	1.08322	1.08320	–0.00002	-1.54×10^{-5}	
	C	1.06060	1.06056	–0.00004	-3.77×10^{-5}	
GE Black	A	1.59603	1.59595	–0.00007	-4.59×10^{-5}	
	B	1.56101	1.56095	–0.00006	-3.84×10^{-5}	
	C	1.57536	1.57540	0.00004	2.54×10^{-5}	
Rokide C	A	1.17536	1.17538	0.00002	1.42×10^{-5}	
	B	1.17814	1.17696	–0.00118	-9.99×10^{-4}	3.979513
	C	1.17789	1.17797	0.00008	6.79×10^{-5}	
BN Paint	A	1.03337	1.03332	–0.00005	-5.16×10^{-5}	
	B	1.02935	1.02927	–0.00007	-7.12×10^{-5}	
	C	1.03793	1.03798	0.00005	4.82×10^{-5}	
Eu_20_3	A	1.13129	1.13130	0.00001	8.84×10^{-6}	
	B	1.13147	1.13147	0.00000	0.00	
	C	1.13243	1.13244	0.00002	1.47×10^{-5}	
Spacesuit Upside-down	A	0.057723	0.057091	-0.000632	-1.10×10^{-2}	2.138566
	C	0.055634	0.055498	-0.000137	-2.46×10^{-3}	0.46221
Brass	B	1.55924	1.55928	0.00004	2.35×10^{-5}	
	C	1.54873	1.54878	0.00005	3.01×10^{-5}	
Spacesuit	A	0.053478	0.052699	–0.000779	-1.46×10^{-2}	2.633468
	C	0.057495	0.057473	–0.000021	-3.71×10^{-4}	
Tungsten	A	2.18819	2.18912	0.00094	4.28×10^{-4}	
	B	2.19872	2.19870	–0.00003	-1.21×10^{-5}	
	C	1.97138	1.97132	–0.00006	-2.87×10^{-5}	

A– 60 °C B– 200 °C C– Control

APPENDIX A
Mass Measurements
(Page 2 of 2)

		Pre-flight Mass	Post-flight Mass	Change in Mass	Fractional Change	AMU per inc. O
Molybdenum	A	1.28027	1.28024	–0.00003	-2.60×10^{-5}	
	B	1.28064	1.28065	0.00000	2.60×10^{-6}	
	C	1.27351	1.27350	–0.00001	-1.05×10^{-5}	
6061-T6Al	A	0.49921	0.49920	–0.00001	-2.67×10^{-5}	
	B	0.49564	0.49554	–0.00010	-2.02×10^{-4}	
	C	0.49315	0.49315	–0.00000	-6.76×10^{-6}	
304Stainless	A	1.45802	1.45803	0.00001	6.86×10^{-6}	
	B	1.44548	1.44545	–0.00003	-2.31×10^{-5}	
	C	1.45029	1.45027	–0.00002	-1.61×10^{-5}	
Diamond-like film	A	0.51090	0.51093	0.00003	5.87×10^{-5}	
	B	0.51838	0.51817	–0.00020	-3.92×10^{-4}	0.687678
	C	0.52488	0.52482	–0.00006	-1.08×10^{-4}	
Udiment720	A	2.96472	2.96470	–0.00002	-5.62×10^{-6}	
	B	2.98427	2.98421	–0.00007	-2.23×10^{-5}	
	C	2.96745	2.96743	–0.00002	-6.74×10^{-6}	
Polypropylene mesh	A	0.019376	0.019019	–0.000357	-1.84×10^{-2}	1.207382
	C	0.019111	0.019111	0.000000	0.00	
FEP Teflon	B	0.62488	0.62473	–0.00015	-2.35×10^{-4}	0.49603
	C	0.61461	0.61451	–0.00010	-1.63×10^{-4}	
Polycarbonate	A	0.45620	0.45570	–0.00050	-1.09×10^{-3}	1.679738
	C	0.45088	0.45095	0.00007	1.55×10^{-4}	
Titanium	B	0.27529	0.27536	0.00007	2.54×10^{-4}	
	C	0.27778	0.27784	0.00006	2.28×10^{-4}	
Vitonfabric	A	0.032504	0.032341	–0.000163	-5.00×10^{-3}	0.550142
	B	0.033347	0.032583	–0.000764	-2.29×10^{-2}	2.583865
	C	0.032436	0.032445	0.000009	2.67×10^{-4}	
FEP on Kapton	A	0.010991	0.010970	–0.000021	-1.94×10^{-3}	0.07215
	B	0.010787	0.010787	–0.000001	-6.18×10^{-5}	
	C	0.011052	0.011069	0.000017	1.54×10^{-3}	
Kapton	A	0.004829	0.004204	–0.000625	-1.29×10^{-1}	2.113764
	B	0.004897	0.003893	–0.001004	-2.05×10^{-1}	3.396678
	C	0.004902	0.004914	0.000012	2.45×10^{-3}	

A– 60 °C B– 200 °C C– Control

APPENDIX B
EOIM—3 Post—Flight Results
Lewis Research Center High Temp Samples Visual Analysis

T. Morton and D. Ferguson-October 14, 1992

1A, 1B	Nb-1 Zr	Brown crescent discolorationon center of B. Dark discoloration on outside rim of A.
2A, 2B	Inconel 718	Faint yellowish discoloration, stronger on B than A. Detectable shadow on both.
3A, 3B	Tungsten-Niobium Composite	3Adull. Both have black rim discoloration. B shows strong brown discolorationin center, with shadow. Some pattern in B-possibly scratched?
4A, 4B	Mo 13 Re	Mottled gray discoloration, stronger on B, spotted on A. Strong shadow, stain looks washed in, under shadow?
5A, 5B	PWC-11	A looks just like C. B has nicotine color with sharp shadow.
6A, 6B	GE Black/Nb-1 Zr	Faint white crescent on B-all the way to edge, so not space related (also seen pre-flight).
7A, 7B	Rokide C/Ti/Al	B detached from substrate. No visible differences.
8A, 8B	BN Paint/Al Type A	More specular reflectance under rim of holder. A and B appear smoother.
9A, 9B	Eu_2O_3/Al	All three look the same specularly. Pit on B half-way from center. A shows faint stain.
10A, 11A	Space Suit Material	Outside looks frizzy. Smoother in center. One brown stripe appears less clear. 10A shows some discoloration in center of white side. 11A and D look the same on stripe side.
10B	Brass	B looks browner, both dirty. Dark discoloration at rim. Sharp shadow. C may have finger print.
12A, 12B	Tungsten	A has central diffuse blob. B has circle. A and B both still shiny.
13A, 13B	Molybdenum	Dirty bluish discoloration in center of A and B. Sharp shadow, dark discoloration on edge.
14A, 14B	6061-T6 Al	A has discoloration, visible shadow. Both A and B appear dirty.
15A, 15B	304 Stainless Steel	B has yellowish stain, sharp shadow. A has shiny ring around center (also seen pre-flight).
16A, 16B	Diamond-Like Film	A and B have lost blueness, still shiny.

17A, 17B	Udimet720	A has black ring at edge. C a little shinier.
18A	Polypropylene Mesh	A looks duller, strands are more uniform. Has lost some of its roughness.
18B	1/16 in. Teflon Disk	B is shinier, smoother. May have a shadow.
19A	Polycarbonate	A is milky, clear shadow. Very thin layer of milkiness. Has a short scratch.
19B	Titanium	Noticeable yellowish discoloration in center of B, loss of specularity.
20A, 20B	Viton Fabric	B has color difference in center, visible shadow.
21A, 21B	1/2 mil FEP on 1 mil Kapton	B has visible shadow. No other visible differences.
22A, 22B	1 mil Kapton	A and B very milky, clear shadow. Loss of specular reflectance.

APPENDIX C

Auger Electron Spectroscopy and Rutherford Backscattering Spectroscopy Measurements on Conducting Samples from EOIM-3

C.A. Zorman, T.G.Eck, and R.W. Hoffman

I. Introduction

Auger electron spectroscopy (AES) measurements were made on 13 sets of samples from EOIM-3. Table I on page 13 of this report gives the identification numbers and names of these sets and information on the composition of some of the samples. Eleven of the thirteen sets had 3 samples in each set: A, which was exposed in space while held at a temperature of approximately 60 °C; B, exposed at a temperature of approximately 200 °C; and C, the control, which was not carried into space. Two of the sets (numbers 10 and 19) had only B and C samples.

All of the AES data were taken with a Physical Electronics single pass cylindrical mirror analyzer. The energy of the incident electron beam was 3 keV and the beam current was approximately 5μA. The diameter of the electron beam at the sample surface was less than 0.5mm. At least two sets of data were obtained for each of the 37 samples, one for AES signals in the 0 to 1keV range and one at lower resolution for signals in the 0 to 2keV range. For several of the samples more sets of data were taken for the 0 to 1keV range to check on the reproducibility of the data or to compare data from different regions of the sample surface.

The AES technique examines the chemical composition of the surface to a depth of approximately 10Å. The samples were not depth profiled to obtain information about the bulk composition, since this would have disturbed the surface structure, which is to be examined with a scanning electron microscope.

Rutherford backscattering spectroscopy (RBS) data were taken for four of the sets of samples (numbers 1, 5, 13, and 19) to look for diffusion of oxygen in to these samples or the formation of thick surface oxide layers. The probe particles for these measurements were 2 MeV He^+ particles. The incident currents were 10 to 20 nA and the exposure times were either 500 or 1500 sec. For these measurements the beam spot on the sample surface was approximately 2mm in diameter.

II. AES Data

Appendix I (available from authors) of this report shows a recorder trace of the AES signals in the 0 to 1keV range for each of the 37 samples. Examples of the lower resolution data (0 to 2keV) are not included, since the AES signals above 1 keV are usually quite weak and, where present above the noise level, serve mainly to confirm the identification of signals below 1keV.

A. Oxygen

Table II on page 14 gives the strength of the oxygen signal (labeled O on the traces in Appendix I) for each of the samples in mm/μA. This strength was determined by measuring the vertical distance between the minimum negative and maximum positive peaks of the signal and dividing this distance by the incident electron current. Table II shows no appreciable difference in the strength of the oxygen signal from the 3 (or 2) samples of a given set. There is no indication for any significant accumulation or depletion of surface oxygen due to exposure to the space environment.

B. Carbon

Table III on page 14 gives the strength of the carbon signal (labeled C on the traces in Appendix I) for each of the samples. Here, in contrast to the results for the oxygen signal, the signal for the exposed samples is weaker than that for the relevant control by at least a factor of 2 and as much as a factor of 5. This removal of carbon from the surface of the exposed samples is not surprising, since the residual atmosphere at the altitude at which these samples were exposed is approximately 90 percent atomic oxygen. The carbon signal for all of the samples of set 9 (Eu_2O_3 film on an aluminum substrate) was very weak, indicating that these samples were prepared in a clean environment, and that the carbon we see on the surfaces of the rest of the 37 samples was not introduced by the storage and handling of these samples before the AES data were taken.

C. SiO_2

An Si or SiO_2 signal was observed for each of the exposed samples. Except for set 10, the corresponding signal on the control sample was substantially weaker. This indicates that the exposed samples were contaminated by SiO_2 during the time they were exposed. The source of this SiO_2 was probably other samples in the sample holder.

We suspect that the silicon on all the 37 samples is present as SiO_2 and that where we observe the Si signal shape it is the result of dissociation of SiO_2 by the incident electron beam. The evidence for this conclusion is the data for sample 19B, where both the Si and SiO_2 signals are seen. A second recorder trace for this sample taken immediately after the trace shown in Appendix I showed a large increase in the strength of the Si signal with a corresponding decrease in the strength of the SiO_2 signal.

D. Further Comments

The remaining signals on the traces in Appendix I are those anticipated from the known bulk compositions of the samples or are the signatures of common surface contaminants (P, S, Cl, N, F, and Na).

The shape of the carbon signal for all of the control samples and most of the exposed samples is that associated with carbon bound to carbon, indicating that it arises from aggregates of carbon atoms on the sample surface which are only weakly bound to the bulk material. For samples 3B, 15A, and 15B the structure on the low energy side of the carbon signal indicates an appreciable contribution to the signal from carbon atoms more tightly bound to the bulk material.

For the control samples of sets 2 and 17 there is a pronounced chromium signal. This signal is absent in the recorder traces for the exposed samples of these sets. We conclude that exposure to the space environment has removed the chromium from the surfaces of these samples.

The 0 to 2 keV recorder traces for set 14, which are not included in this report, show the expected Al_2O_3 signal at 1380 eV. There is also a MgO signal at 1175 eV. While the Al_2O_3 signal has the same strength for all three samples of the set, the MgO signal is just above noise for samples A and C and nearly as strong as the Al_2O_3 signal for sample B. It would appear that the higher temperature at which sample B was exposed resulted in diffusion of magnesium from the bulk to the surface.

III. RBS Data

The RBS technique involves bombarding a target with low mass, monoenergetic ions and measuring the kinetic energy distribution of the projectiles which are scattered into a small solid angle centered about a direction which is at a large angle with the incident beam direction. For the data reported here, this angle was 170°. A monatomic target, such as pure Nb, Mo or Ti, would give an energy distribution which, with decreasing energy, rises steeply from a high energy cutoff to a plateau whose height increases slowly with decreasing energy (See any of the figures in Appendix II.). The high energy edge of the distribution is produced by incident particles which are backscattered in binary collisions with target particles at the surface of the target. The rest of the distribution is produced by incident particles which are backscattered from target particles below the surface. These projectiles lose energy on their way into and out of the target due to multiple small angle scatterings. The deeper the penetration into the target before backscattering, the greater the energy loss. For 2 MeV He^+ particles incident on Nb, Mo, or Ti targets, this energy loss mechanism limits the depth of the target sampled by the RBS technique to approximately 10^4 Å.

Oxygen atoms dispersed throughout the volume of a Nb, Mo, or Ti target would produce a plateau atop the host atom distribution with a high energy cutoff at the position indicated in the figures in Appendix II. If, instead of being dispersed, the oxygen is concentrated in a thin layer at the surface of the target, the plateau is replaced by a narrow bump.

The RBS technique is not very sensitive for detecting a low mass impurity in a high mass host, since the impurity signal sits atop the host signal and tends to be buried in the statistical noise of the host signal. For a high mass impurity in a low mass host, the cutoff of the impurity signal is at a higher energy than that of the host, so that over a substantial energy range the impurity signal is unobscured by the noise in the host signal.

The RBS data for all of the samples investigated using this technique are shown in Appendix II. The first two pages of Appendix II show the data for sets 1 and 5. These are Xerox copies of the originals, which display the data for samples A, B, and C in three different colors. There is no evidence of an oxygen signal, either a plateau cutoff of a bump at the indicated energy of 0.72 MeV, for any of the six samples of these two sets. The data for the three samples of set 13 are shown on the next three pages of Appendix II. Again, there is no oxygen signal apparent in the data for any of these samples.

For set 19, the titanium samples, there is the hint of an oxygen signal (a bump), which is more pronounced for the exposed sample (B) than for the control (C). However, the evidence for this conclusion is certainly not compelling. Comparison of the data for sample 19B with computer simulations suggests the presence of a surface layer of TiO_2 which is diffuse, i.e., the concentration decreases with increasing depth into the sample, and has a thickness between 300 and 500 Å.

The limiting factor in our ability to detect an oxygen signal is the statistical noise in the RBS signal from the host. The fractional noise can be decreased by increasing the fluence of the incident beam of He^+ particles, but this increase in fluence could damage the surface of the target. To limit this damage, only the molybdenum samples were investigated with the longer exposure time (1500 sec) .

Our final conclusions from the RBS study are:

1. For the samples of sets 1, 5, 13, and 19 the ratio of the number of O atoms per unit volume to the number of host atoms per unit volume is less than ~1/10.

2. We see no evidence for an oxide layer on the surface of any of the samples in sets 1, 5, and 13. Oxide layers thinner than ~ 100 Å would not have produced a detectable signal and, thus, cannot be ruled out.

3. There appears to be a diffuse oxide layer on the surface of sample 19(b) with a thickness of 300 to 500 Å.

TABLE I.—EOIM-3 SAMPLES

Number	Name	Composition
1	Nb–1 Zr	Nb, 1 percent Zr
2	Inconel 718	19 percent Cr, 3 percent Mo, 5.1 percent Nb, 18.5 percent Fe, 52.8 percent Ni, 0.9 percent Ti, 0.5 percent Al, 0.08 percent C, and 0.15 percent other
3	Tungsten-Niobium Composite	
4	Mo 13 Re	Mo, 13 percent Re
5	PWC-1 1	Nb, 1 percent Zr, 0.1 percent C
9	Eu_2O_3/Al	Eu_2O_3 film on aluminum substrate
10	Brass	
12	W Thin Film/Al	Tungsten disk (not a film), no aluminum substrate
13	Mo Thin Film/Al	Molybdenum disk (not a film), no aluminum substrate
14	6061-T6 Al	
15	304 Stainless	
17	Udimet 720	18 percent Cr, 14.7 percent Co, 3 percent Mo, 1.25 percent W, 5 percent Ti, 2.5 percent Al, 55.6 percent Ni, and traces of C, B, and Zr
19	Titanium	

TABLE II.—OXYGEN AES SIGNAL STRENGTH IN mm/μA

Set	Sample		
	C, control	A, 60 °C	B, 200 °C
1	15	20	17
2	13	15	16
3	26	24	23
4	12	15	14
5	29	25	25
9	20	15	15
10	9.1		13
12	8.6	13	12
13	8.5	12	12
14	20	26	28
15	26	25	29
17	13	24	25
19	31		31

TABLE III.—CARBON AES SIGNAL STRENGTH IN mm/μA

Set	Sample		
	C, control	A, 60 °C	B, 200 °C
1	8.7	5.3	4.4
2	8.4	4.3	4.2
3	9.3	4.9	4.5
4	5.8	1.4	0.91
5	9.6	4.0	2.5
9	0.81	0.40	0.40
10	9.7		3.2
12	6.6	1.5	1.0
13	6.8	1.1	1.2
14	15	5.8	8.1
15	13	6.6	5.8
17	14	5.9	5.6
19	10		3.3

APPENDIX D

SEM Examination of EOIM-3 Post-Flight Samples

Lewis Research Center High Temp Samples

James W. Smith

PROCEDURE

A group of 24 test samples consisting of three coupons of eight different materials were received for SEM surface examination. Individual coupons of each material were identified as follows; (A) exposed in space at 60 °C, (B) exposed in space at 200 °C, and (C) a control sample.Individual samples were 12.5 mm in diameter and l~mm in thickness. The support surface of each coupon was inscribed to indicate the proper orientation.

Samples were individually affixed to 12.5 aluminum mounts by carbon conductive tape. The coupons were examined uncoated in a JEOL 84OA Scanning Electron Microscope at 15Kv operating voltage. Both SEI (secondary electron images) and BEI (backscatter electron- images) were obtained from each sample. A minimum of five micrographs were obtained from the control sample at the following magnifications; SEI and BEI at 1000x, SEI at 5kx, 10kx, and 20kx. A minimum of seven micrographs were obtained from the post-flight coupons: SEI and BEI at 25x of the coupon edge where the retainer rim overlapped to coupon surface, SEI and BEI at 1000x on the coupon center, SEI at 5kx, 10kx, and 20kx of the coupon center. Additional micrographs were obtained from some coupons at the covered edge region for comparison purposes and at specific surface details.

Appropriate elemental analyses were performed by EDX (energy dispersive x-ray spectrometry) or WDX (wavelength dispersive x-ray spectrometry) for light element analyses (oxygen). X-ray distribution dot maps are included for several samples, all of which were collected by WDX. The distribution of dots on these maps is proportional to the relative concentration of the element scanned.

All micrographs referred to in this Appendix are with the authors.

RESULTS

1a.1b.1c Niobium - 1 percent Zirconium

The most prominent features on all samples are polishing striations and other pre-flight mechanical damage (pitting and material pullout). Both post-flight specimens illustrate rim delineations (shadows) at low magnification in the SEI mode with higher oxygen levels on the exposed surfaces, but there is very little discrimination in the BEI images. There appears to be slightly more particulate on the sample exposed at 200° as compared with the control, particularly at magnifications of 10 and 20k. No chemical differences could be detected by EDX.

2a.2b.2c Inconel 718

Pre-flight mechanical damage, primarily in the form of polishing striations are prominent on all three coupons and show no evidence of alteration. Distinct rim bands are present in the SEI mode and to some extent, detectable in the BEI mode. Both post-flight samples illustrate oxygen enrichment in the exposed areas but no physical evidence of oxide formation at 20k.

3a.3b.3c Tungsten—Niobium Composite

Polishing striations and other forms of pre-flight surface damage such as pits and gouges are prominent and there is a progression in the roughness and definition of surface features through the sample series. Sample 3(b) appears to have more fine particulate on the surface. In addition, all samples illustrate fine irregular patches of carbonaceous material. It is unclear if this is part of the microstructure or not, as these patches are absent from the surface of sample 3(a). Rim delineations are observed on post-flight samples indicating oxygen enrichment of the exposed surfaces. Composite microstructure is not obvious on the planer surface of any sample.

4a.4b.4c Molybdenum -13 Percent Rhenium Alloy

Post-flight samples illustrate thin incrustations of oxide material around the rim delineations and in spotted areas across the exposed surface. Some areas of the incrustation appear distinctly crystalline and other areas appear amorphous with included crystallites. A defined delineation is present at the retainer ring boundary as well as relatively large particulate particles (2-3 µm). Large areas of the exposed surface of sample 4(a) appear unaffected. Approximately 85 to 90 percent of sample surface 4(b) has been affected, but the oxidation products are different in form than those observed on the 60 °C sample. High magnification illustrates fine particulate and acicular crystal formation even in areas which appear clean. Chemical characterization of the oxidation products was not possible during this examination due to equipment malfunction. If deemed necessary, this can be accomplished at a later date.

5a.5b.5c PWC-11 Niobium - 1 Percent Zirconium - 0.1 Percent Carbon

Surface etch pits and 1-3 µm particles of what are likely Nb-Zr oxy-carbides are characteristic of all surfaces, even the control. Oxidation of the exposed surfaces is evident, particularly in sample 5(b), but there is no obvious oxidation product, even at 20k. Backscatter images of the 200 °C sample appear "dirty" which probably reflects increased oxidation.

12a.12b.12c Tungsten

Characteristic pre-flight surface microstructure includes polishing striations, pits, and processing porosity. Post-flight samples illustrate distinct rim delineations and oxidation product, most predominantly in the polishing striations. Some spotting is apparent, which is typically a thin encrustation of amorphous material. Sample 12(a) illustrates notably more debris on the surface which suggests it may have been contaminated. BEI micrographs indicate higher oxidation of the 200 °C sample.

13a.13b.13c Molybdenum

Pre-flight surface microstructure includes directional grinding marks, processing porosity, and machine-smear marks. Extremely fine particulates which average 50nm in diameter are also present. This might be pre-flight oxide. Post-flight surfaces illustrate rim delineation, a concentration of oxide product at the retainer rim ID, and spotting and mottling of the exposed surfaces. Fine acicular needles are observed over the surface which resemble molybdenum-trioxide, but this cannot be confirmed by SEM techniques due to extremely small size. An amorphous product is frequently observed in striations and crevices. Several spots are observed on sample 13(b) which are very oxygen rich.

17a.17b.17c Udimet 720

Circular grinding striations are prominent on all three coupons. Post-flight samples illustrate rim delineations in both SEI and BEI modes. Striations which were beneath the retainer rim are characterized by contamination build-up. Any oxidation product present is not evident at 20k. The visual analysis refers to a black ring at the edge of sample 17(a). This was not obvious during this examination. Dark spots are observed in the SEM around the edge and EDX indicates they are rich in O,N,C,Na,Cl, and K (see EDX-17a). The sodium and chlorine suggest skin contact.

Reported 12/10/93 J.W.S

14a.14b.14c 6061-T6 Anodized Aluminum

Concentric grinding striations are prominent on all three coupons. Characteristic microstructures on the pre-flight coupon include smear ridges frequently associated with irregular 0.5-3.0 µm inclusions containing Al, Si, Fe, and Cr (see EDX 03a). Some porosity has been introduced on the surface by mechanical debonding of the hard inclusions during the grinding process. Oxygen is not detected by EDX on the anodized surface (EDX 03b).

The exposed regions of the post-flight coupons illustrate no significant microstructural alterations. Secondary SEM images illustrate retainer ring shadows at the coupon edges, most prominent on the 200° sample. As observed in the visual examination, the surfaces are dirty and appear to be contaminated with a carbonaceous residue, most apparent at the coupon edges as spots and blotches and on a finer scale, as a thin film residue in the recesses of grinding marks.

The retainer ring shadow in sample 14(b) illustrates a binary secondary image most prominently characterized by a 500 μm wide dark discoloration extending back toward the edge from the retainer ring i.d. Wavelength dispersive line-scan-profiles indicate a slight increase in oxygen across this band. The oxidation product appears to be concentrated in the surface recesses. Very small amounts of oxygen and silicon (an alloy component as well) are detected on exposed regions of sample 14(b) which are free of the organic-like residues. No changes in the pre-flight morphology of the Al, Si, Fe, and Cr inclusions was observed.

15a.15b.15c No.304 Stainless Steel

Pre-flight grinding marks are the prominent microstructural features on all surfaces. Retainer ring shadows are observed on the post flight coupons. EDX comparisons of the shadow and exposed region do not reveal any apparent fluctuations in surface chemistry. A low-level oxidation of the surface is suspected, as has been illustrated in other test group samples. The lustrous ring visually observed on sample 15a likely resulted from a momentary lag in the tool-feed during the preflight lathe operation.

16a.16b.16c Diamond-like Film on Silicon

The diamond-like film is apparently a polymeric type material consisting of carbon and hydrogen. The initial thickness of the film was approximately 200 nm on a polished silicon substrate (personal correspondence). The preflight specimen is heavily contaminated with an amorphous substance consisting primarily of oxygen (compare EDX spectra 06a and 06b). This is most likely the adhesive used to secure the silicon substrate to the metal platen.

The presence of the film is indicated by the higher carbon level detected on the silicon substrate (micrograph 0006 and associated carbon x-ray map). In order to image the film the sample was examined on-edge at a 50° tilt. The backscatter image obtained at 10 000 x illustrates an atomic number contrast consistent with the film composition. There is some sacrifice in atomic number contrast in order to maintain the spatial resolution.

The post-flight coupons also illustrate adhesive contamination on the exposed surfaces. It is interesting that the adhesive surface shows no evident morphological alterations, aside from what appears to be thermal crazing. Edge-on examinations of sample 16(b) (60°exposure) suggest film degradation in isolated regions. As illustrated in the high magnification photomicrographs (number 0007 0008), the film is initially corrupted by sub-micron dimples in the film. Initial defects in the film, such as a blemish or fine surface scratches appear to be susceptible to deterioration.

There is only a suggestion of the film remaining on the 200° sample. A few isolated areas of film remain which are heavily etched (micrographs number 0008 to 0010).

19b.19c Titanium

A grit-blasted surface with occasional embedded grit is characteristic of the preflight sample. Sub-micron particulate, suspected to be oxide, is lightly dispersed on the surface, primarily along projections.

The post-flight 200° sample illustrates a retainer ring shadow at the sample edge. The grit-blasted morphology is prominent but there appears to be etch pits associated with this microstructure, even under the retainer ring (preflight acid cleaning). The sub-micron oxide is present at about the same level observed in the pre-flight specimen. No suspected silicon was detected on the titanium surface by EDS analysis.

20a.20b.20c Viton Fabric

The prominent surface feature is the vestige +45/-45 ply layup of the interwoven glass fibers, encapsulated by the Viton fluor-elastomer. Arbitrarily dispersed across this surface are irregular granules of what appears to be polymer debris and broken glass fibers. The polymer granules are typically 10-30 µm in diameter and are fused to the sample surface, as are the pieces of glass fiber. A number of micrographs are enclosed which illustrate this. Backscatter electron images illustrate (i.e. number 0005) high atomic number spheroids dispersed in the polymer which range in size from 0.2-1.0 µm in diameter. EDS scans of the largest particles indicate that they are lead rich (EDX-06a and 06b). These may be a catalyst to aid in the vulcanization. The fluorine detected is contributed by the surrounding Viton elastomer. The source of the magnesium in the Viton was not investigated, but is likely a filler or additive to the system.

Only subtle changes are apparent in the 60° post-flight sample. There is evidence of cone texturing on some of the polymer debris particles (micrograph number 0013) and in some instances, the partial removal on debris granules (micrographs number 0002 and 0004). On the 200 °C coupon (20(b)) the majority of polymer granules have been eliminated from the exposed surface but vestige marks remain indicating their previous location (the debris remains in the region protected by the retainer ring). At such locations, the microstructure suggests an erosive influence, presumably from atomic oxygen attack. Across the exposed surface, there is a fine scale surface degeneration, conspicuous as pores and cavities generally less than 1.0 µm in diameter (see micrographs number 0006 0008).

18b.18c 1/16" Teflon Disk

The primary microstructural features of the pre-flight coupon are abrasions and process imperfections. Barium sulfate particles, possibly from the mold release agent, are randomly impressed in the surface of the Teflon.

A shadow of the retainer ring is present on the 200° sample, which appears to have been heavily abraded following the experiment. Uncorrupted regions are present which illustrate a fine-textured, atomic oxygen etch. Micrograph numbers 0021 to 0023 illustrate the etched surface at a sample tilt of 45°, which accentuates the texture.

As in the pre-flight sample, barium sulphate particles are randomly embedded in the surface (micrographs number 0011 0015). Several spots of post-flight contamination were observed on the surface (polymeric in nature).

21a.21b.21c 1/2mil FEP on 1 mil Kapton

At low magnifications, the pre-flight coupon illustrates surface abrasions and a perceptible linear texture, possibly from the production process. At magnifications above 10 000x, the surface is essentially featureless.

There is a trace of retainer ring shadow on the post-flight 60° coupon, most obvious in the secondary imaging mode (SEI). The overall sample surface is somewhat soiled with scattered debris. A filamentary surface texture becomes apparent in the exposed regions at magnifications above 5000x which can develop into free standing fibrils (micrographs number 0014 0015).

Sample 21(b) illustrates a distinct retainer ring shadow in both secondary and backscatter imaging. Also apparent are numerous post-flight abrasions to the exposed surface. At magnifications above 5000x, a highly directional etch pattern results from atomic oxygen attack to the surface (micrographs number 0006-0012). Undoubtedly, the FEP polymer microstructure influences the texture observed.

22a.22b.22c 1 mil Kapton

Other than a few surface abrasions, the pre-flight Kapton film is featureless.

Each of the post flight samples was examined both at tilts of 0° and 20° to better characterize the effects of atomic oxygen etching.

A distinct retainer ring shadow is present on sample 22(a) 60° exposure. The exposed area of the specimen illustrates the classic atomic oxygen texturing consisting of circular voids generally less than 0.5 µm in diameter. Spires develop between the voids. The chronological development of this microstructure can be observed to some extent by examining the retainer ring contact point (micrographs 0016-0019 and 0007-0013-20°tilt). The incipient etch appears to be relatively homogeneous. At a later stage in the attack, preferentially etched linear features develop, which appear to have some geometric orientation. These might be related to residual stresses in the film and/or localized orientation in the polymer chains. The regions defined by these linear grooves have also been preferentially etched resulting in a perceptible radial pattern. One influence on the preferential etching of the surface is the presence of a secondary residue which is observed as a gossamer canopy over some areas of the atomic oxygen texturing (micrographs number 0021 0022). It is clear that gaps in the film result in a greater degree of etch in the Kapton (this is particularly well illustrated in the 20° sample tilt series, micrographs number 0016 0018). The source of this film is unknown. It has been suggested that is may be an anti-stick film on the tape or a residue product.

Foreign particles on the surface of the sample which are not eroded by atomic oxygen shield the underlying Kapton and remain supported on a pedestal of polymer. Micrograph number 0057 illustrates several particles of boron nitride (a common mold release agent) supported by Kapton pedestals. Several other examples are illustrated.

The surface texture of sample 22(b) (200 °C) is an escalation of the morphology described above. Continued erosion results in a more obtuse angles along the linear furrows and other regions of preferential etch. The gossamer residue remains as a canopy over some areas of the sample.

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In order to test their reactivity with Atomic Oxygen, twenty five materials were flown on the EOIM-3 (Evaluation of Oxygen Interactions with Materials) portion of the STS-46 Mission. These materials include refractory metals, candidate insulation materials, candidate radiator coatings, and a selection of miscellaneous materials. This report documents the results of the pre- and post-flight analysis of these materials.

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