10-18-2013

Atomic Oxygen Modification of the Nanodielectric Surface Composition of Carbon-Loaded Polyimide Composites

Kelby T. Peterson
Utah State University

JR Dennison
Utah State Univesity

Recommended Citation

Peterson, K., & Dennison, J. R. (2013, October 18). Atomic Oxygen Modification of the Nanodielectric Surface Composition of Carbon-Loaded Polyimide Composites. Presented at the Fall 2013 Meeting of the Four Corner Section of the American Physical Society, Denver, CO.

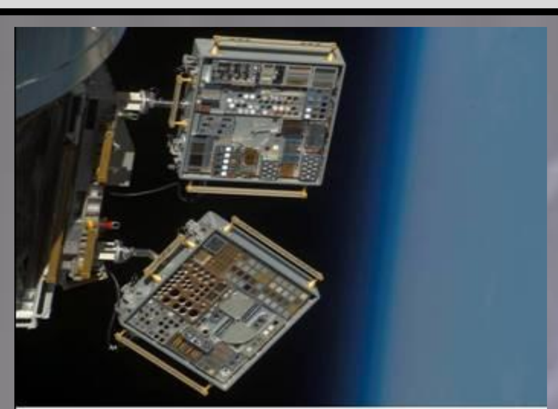
Atomic Oxygen Modification of the Nanodielectric Surface Composition of Carbon-Loaded Polyimide Composites

Kelby T. Peterson and J.R. Dennison

Utah State University, Logan, UT 84332-4414
Materials Physics Group, Physics Department
E-mail: kelby.peterson@aggiemail.usu.edu

MISSE-6 Sample Exposure

MISSE-6 is part of the MISSE project, that subjects various materials to the space environment to understand the effects in a controlled setting (1). MISSE-6 samples were compiled, launched into space, suspended off of the International Space Station for 18 months, and then returned to Earth in pristine condition for analysis (2).



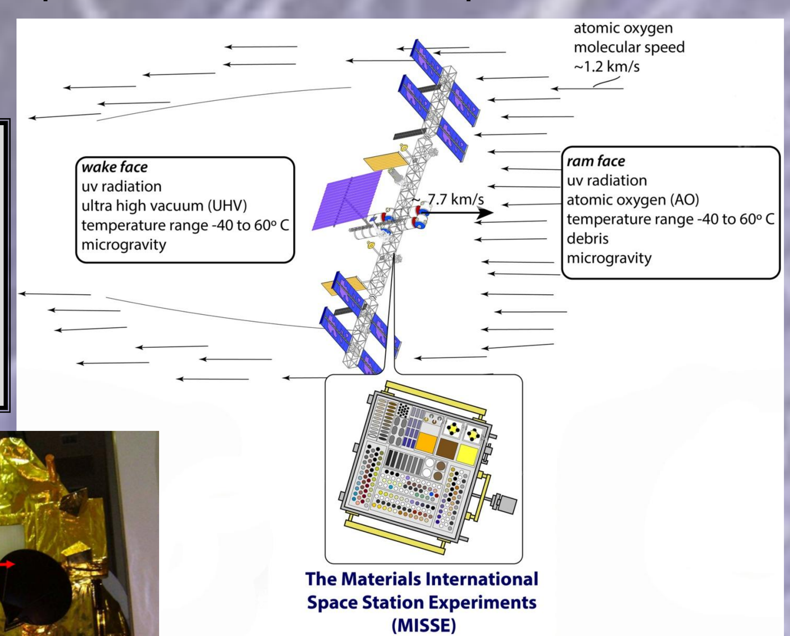
SUSpECS Objective:

The Utah State University SUSpECS project was a unique student experiment on MISSE-6 (3). The purpose of SUSpECS is to characterize the performance of prospective spacecraft materials when subjected to the synergistic effects of the space environment (4).

Figure 1: MISSE-6 Sample Containers suspended from the International Space Station

Figure 2: Space environment exposure of MISSE-6 samples in various locations

The location of the sample on the tray varied the exposure of SUSpECS(5):
- **UV radiation, known to discolor the surface of polymers.**
- **Atomic oxygen fluence determined by polyimide mass loss.**



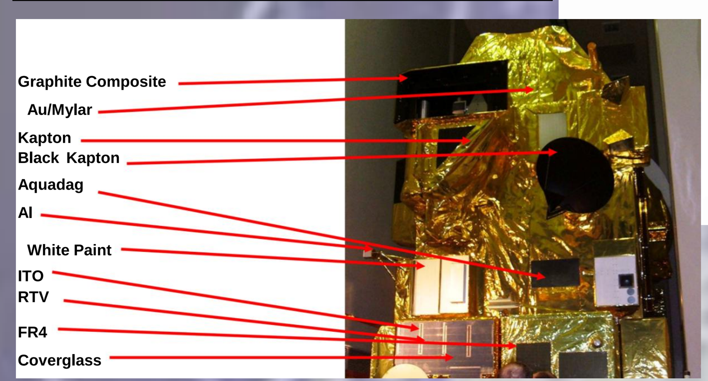


Figure 3: Note use of Carbon-Loaded Polyimide (Black Kapton) here

MISSE-6 Carbon-Loaded Polyimide Samples

The location of the samples on the bottom layer eliminates the UV radiation variable and decreases the atomic oxygen exposure to only the reflected portions, giving a reduced exposure as well. The bias voltages were designed to test the effects of charge enhanced contamination. The RAM samples allow for analysis of the maximum exposure to UV Radiation and Atomic Oxygen erosion. The wake samples give a decreased exposure to UV radiation as well as atomic oxygen erosion. Utilizing this variety of exposures and sample locations there is ample opportunity for study and comparison of the affects of the space environment on Carbon-Loaded Polyimide.

Material	Location	Position Number	Exposure	Comments
Black Kapton 100XC	Wake, grounded	W-12-A	Med UV, Med AO	Test for effects of UV & low AO
Black Kapton 100XC	Wake, +5 VDC	W-14-A	Med UV, Med AO, +5 VDC	Test for charge enhance contamination
Black Kapton 100XC	Wake, -5 VDC	W-21-A	Med UV, Med AO,-5 VDC	Test for charge enhance contamination
Black Kapton 100XC	Wake, -15 VDC	W-22-A	Med UV, Med AO,-15 VDC	Test for charge enhance contamination
Black Kapton 100XC	Ram, Top Tier Double Stack	R-2-A	High UV, High AO	Test for effects of UV & high AO
Black Kapton 100XC	Ram, Underside Top Tier Double Stack	R-24-C	Low UV, Low AO	Test for effects of low UV & high AO

Figure 4: Carbon-Loaded Polyimide Samples (Black Kapton) flown on MISSE-6

Carbon-Loaded Polyimide Properties

Macro-Scale Properties:

Nanodielectric composite material consisting of an insulating polyimide matrix (~100-5000 nm depth) loaded with conductive carbon particles (~100-500 nm). Insulating regions build up charge that ultimately leads to cathodoluminscence and arcing of the material surface, particularly at low temperature vacuum environments like those used for space-based observations (6).

Nano-Scale Properties:

High conductivity material ranging from 10^{-7} to 10^{-3} $(\Omega\text{-cm})^{-1}$ dependent on the carbon concentrations. Designed to withstand high voltages and extreme temperatures. Polyimide is utilized to determine the flux of Atomic Oxygen based on the rate of the degradation of the polymer (5).

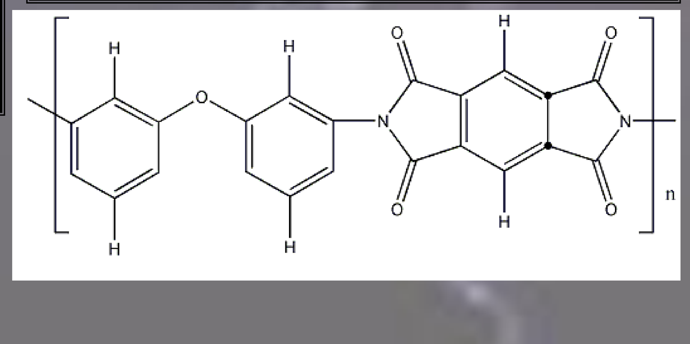


Figure 5: Structure of typical Polymer $(C_{22}H_{8}N_{2}O_{5})_n$

Abstract

Carbon-Loaded Polyimide is a nanodielectric composite of carbon particles (~100-500 nm) embedded in an insulating polyimide polymer matrix (~100-5000 nm depth). Analysis of this nanodielectric composite has been done via optical imaging, scanning electron microscopy, and energy-dispersive x-ray analysis in order to gain insight into its nanodielectric properties. The insulating polyimide is known to be inert and impervious to strong bases and acids, but is affected by atomic oxygen exposure. We have observed changes in the surface structure and relative carbon-polymer concentrations in MISSE-6 samples that were exposed to the low earth orbit environment for 18 months outside the International Space Station. The MISSE-6 sample tray arrangement permitted studies of the effects due to varied atomic oxygen exposure. MISSE samples received maximum atomic oxygen exposure on the ram side with decreased exposure on the wake and shielded sides, respectively. Early observations suggest that the atomic oxygen modifications reduce the polymer matrix on the surface, whilst the carbon-loaded regions remain largely unaffected by the exposure. Affects of the surface modifications on spacecraft charging and cathodoluminescence will be discussed.

Optical Analysis

Microscope Imaging:

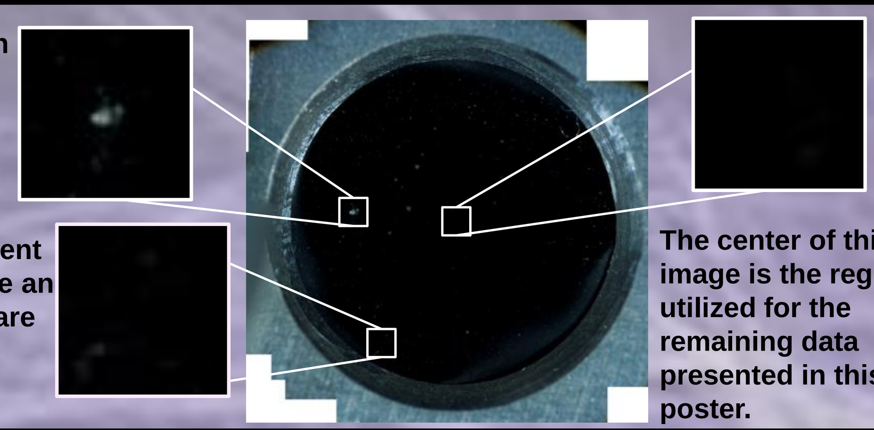
Detailed microscope images of each sample were complied to give a high resolution view of each sample. Look for visible surface defects and change in surface color .

Before

After

Abnormalities in the samples warrant further analysis.

Regions of apparent continuity provide an average to compare with pre-flight samples.



The center of this image is the region utilized for the remaining data presented in this poster.

Scanning Electron Microscopy Analysis

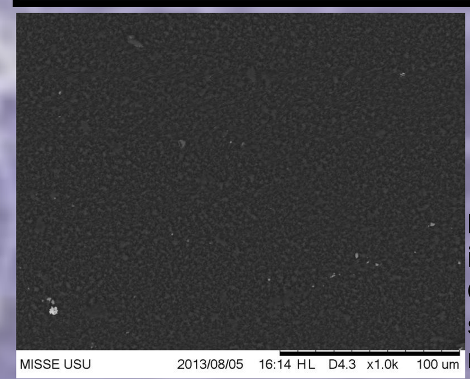
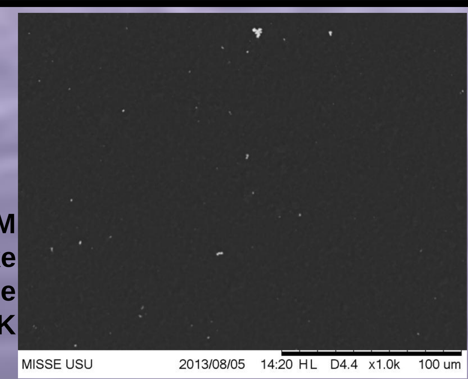


Figure 6 (Right): SEM image of stock Dupont Carbon-Loaded Polyimide XC sample at x1.0K magnification

Figure 7 (Left): SEM image of MISSE-6 wake Carbon-Loaded Polyimide sample at x1.0K magnification



Scanning Electron Microscopy (SEM) allows for surface analysis of materials. Light regions correspond to charged regions of the sample; in this case the polyimide matrix. Dark regions represent the conductive regions; in this case the carbon regions of the samples.

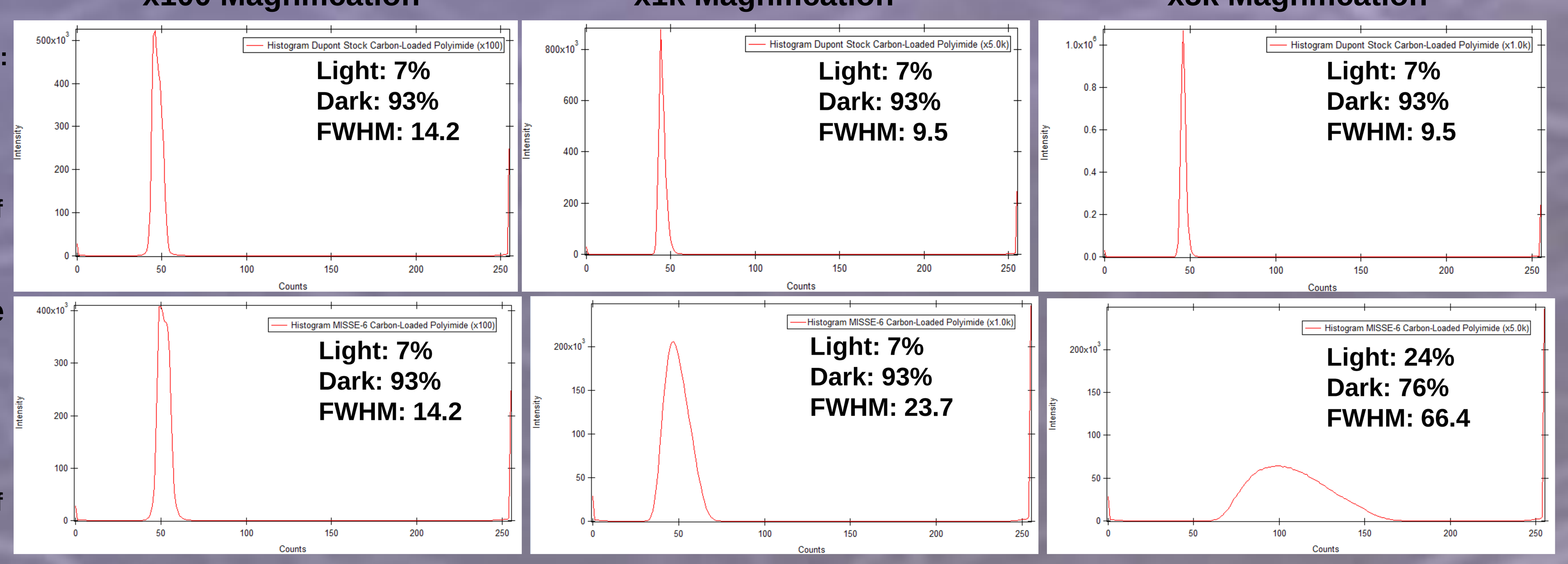
Stock Sample

Figures 8-10 (Right): Histograms of SEM image. Gray scale intensity at increasing magnification. All of stock Carbon-Loaded Polyimide.

MISSE-6 Sample

Figures 11-13 (Right): Histograms of SEM image. Gray scale intensity at increasing magnification. All of MISSE-6 sample.

x100 Magnification | x1k Magnification | x5k Magnification



Energy Dispersive X-Ray Analysis

Figure 14 (Right): EDAX image of MISSE-6 wake Carbon-Loaded Polyimide sample at x5.0k magnification (Below) EDAX analysis of percent concentrations and graph of the elemental composition.

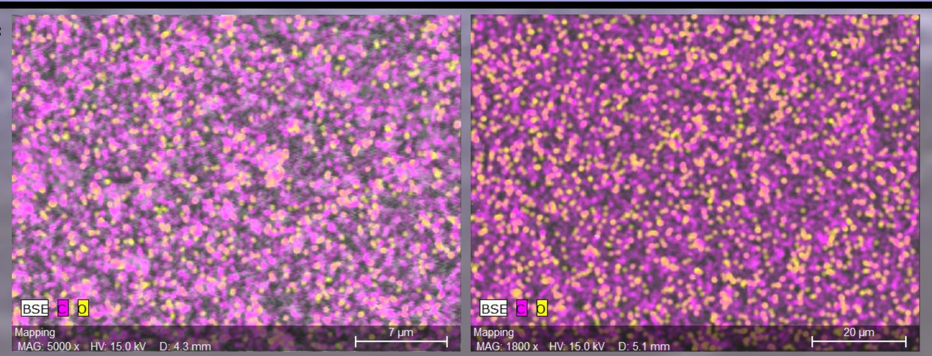
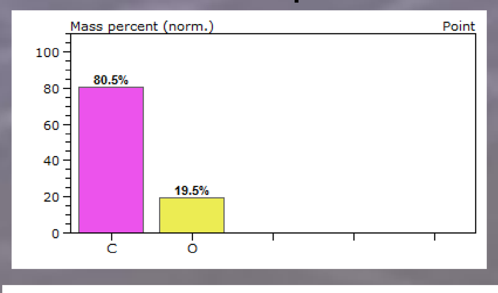
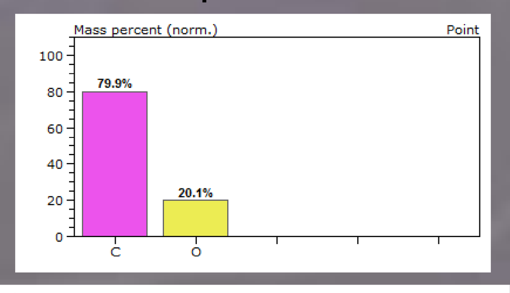
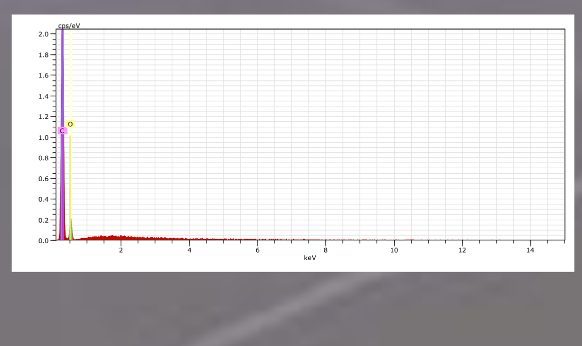
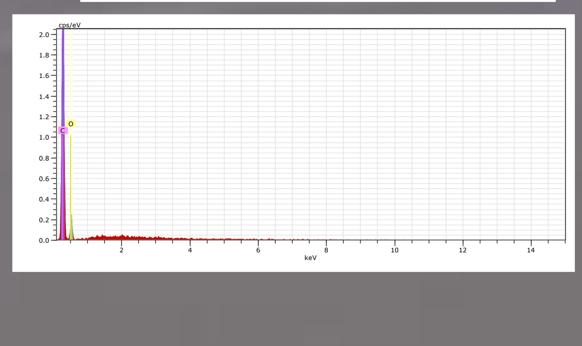


Figure 15 (Left): EDAX image of stock Dupont Carbon-Loaded Polyimide XC sample at x5.0k magnification (Below) EDAX analysis of percent concentrations and graph of the elemental composition.









EDAX analysis provides information about the chemical characterization of a material. EDAX has low surface sensitivity due to the nature of the electron penetration in the material. Surface variations due to atomic oxygen and UV exposure occur in the top few nm but EDAX probes 1-10μm deep. This prevents light elements, such as Hydrogen to be viewed. Due to this high energy there is little apparent variation between the stock and MISSE-6 flight Carbon-Loaded Polyimide samples.

Relative Surface Concentrations

The relative surface concentrations of the stock and MISSE-6 samples give insight into the erosion due to atomic oxygen exposure and UV radiation whilst suspended from the international Space Station. Via optical analysis it is evident that erosion occurred. Low surface sensitive EDAX analysis shows little variation in the elemental concentrations of the samples. Regardless the SEM's high surface sensitivity shows decreased polymer (dark) regions and increased full width at half max (FWHM) with increased magnification.

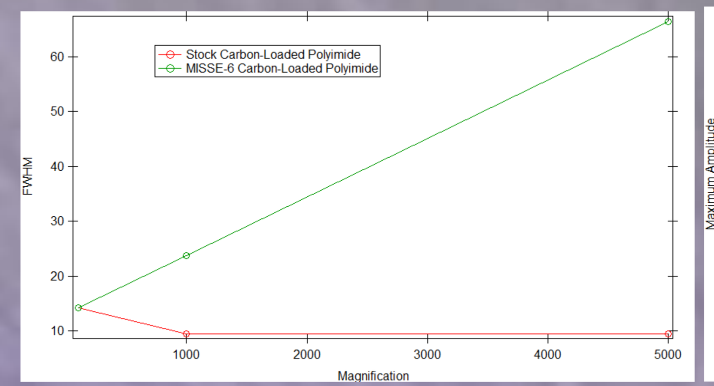


Figure 16: Graph of the full width at half maximum of both samples to emphasis the change in width for the MISSE-6 Carbon-Loaded Polyimide Sample.

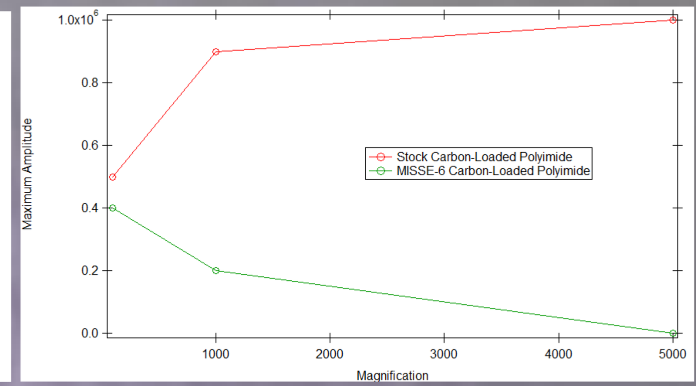


Figure 17: Graph of the maximum amplitude of both samples to emphasis the significant decrease in the MISSE-6 Carbon-Loaded Polyimide Sample.

Conclusions

EDAX analysis is done at a higher energy and is thus able to penetrate deeper into the sample (~0.1-50 μm) therefore focusing less on the surface variation. SEM analysis on the other hand is done at lower energies, penetrating less deep (~6-40 nm) and therefore more accurately representing the surface variations of the carbon-Loaded polyimide.

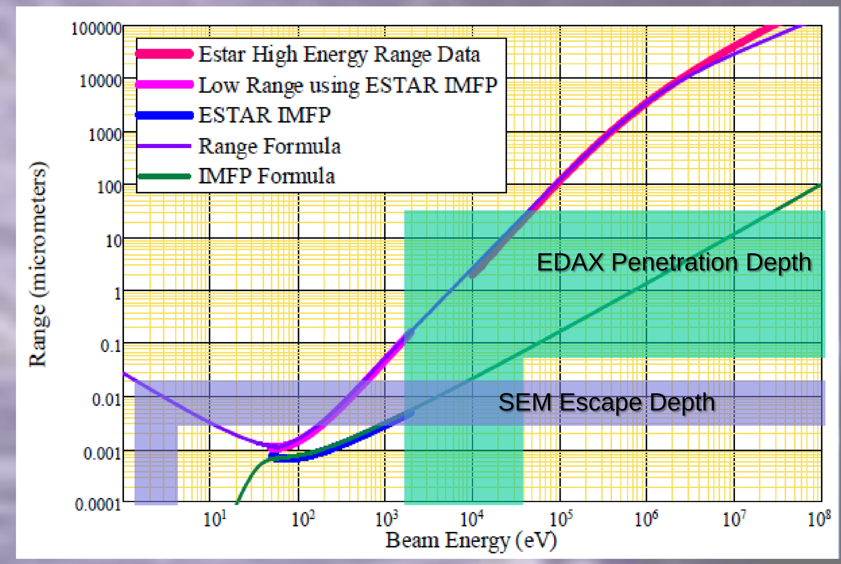


Figure 18: Graph showing the penetration depth of Kapton (7)

Analysis of the full width at half maximum of the histograms of the SEM data shows that there is a significant increase in the range of intensities of the charged regions. SEM analysis of the conducting and charging regions shows a correlation between magnification and percent of light regions, charging regions. This correlation implies an increased variety in the light and dark regions present on the material surface. This implication reaffirms the expected atomic oxygen erosion by eliminating the repetitive pattern of the stock material's surface features.

Future Work

Current analysis has been done on a MISSE-6 wake sample; further analysis will be done on ram samples to determine if the effects are more prominent. Beyond that there will be analysis of the remaining MISSE-6 samples to find a correspondence between exposure and the noted effects. There will also be analysis of the changes in reflectivity and electron emission of the samples.

Acknowledgements/References

Research on SUSpECS was supported by funding from USU Space Dynamics Laboratory, the NASA Solar Probe Mission Program through Johns Hopkins Applied Physics Laboratory, Salt Lake Community College and a Utah State University Undergraduate Research Fellowship from the Office of Research and Graduate Studies.

1) (1) JR Dennison, Amberly Evans, Danielle Fullmer, and Joshua L. Hodges, "Charge Enhanced Contamination and Environmental Degradation of MISSE-6 SUSpECS Materials," accepted for publication in IEEE Trans. on Plasma Sci., 40(2), 254-261 (2012). DOI: 10.1109/TPS.2011.2178104
2) (2) JR Dennison, John Prebola, Amberly Evans, Danielle Fullmer, Joshua L. Hodges, Dustin H. Crider and Daniel S. Crews, "Comparison of Flight and Ground Tests of Environmental Degradation of MISSE-6 SUSpECS Materials," Proceedings of the 11th Spacecraft Charging Technology Conference, (Albuquerque, NM, September 20-24, 2010), 12 pp.
3) (3) J.R. Dennison, Joshua L. Hodges, J. Duce, and Amberly Evans, "Flight Experiments on the Effects of Contamination on Electron Emission of Materials," Paper Number: AIAA-2009-3641, Proceedings of the 1st AIAA Atmospheric and Space Environments Conference, 2009.
4) (4) Material International Space Station Experiment (MISSE), Mar. 24, 2005. [Online]. Available: http://misse1.larc.nasa.gov/
5) (5) D. Hastings and H. Garrett, Spacecraft–Environment Interactions. Cambridge, U.K.: Cambridge Univ. Press, 1996.
6) (6) JR Dennison, Amberly Evans Jensen, Justin Dekany, Gregory Wilson, Charles W. Bowers and Robert Meloy, "Diverse Electron-induced Optical Emissions from Space Observatory Materials at Low Temperatures," Proceedings of the Society of Photo-Optical Instrumentation Engineers Cryogenic Optical Systems and Instruments Conference, Paper No. 8863-12, (San Diego, CA, August 25-29, 2013).

(7) Gregory Wilson and J.R.Dennison, "Approximation of Range in Materials as a Function of Incident Electron Energy," Proceedings of the 11th Spacecraft Charging Technology Conference, (Albuquerque, NM, September 20-24, 2010)

(8) Amberly E. Jensen, JR Dennison, Justin Dekany, and Gregory Wilson, "Nanodielectric Properties of High Conductivity Carbon-Loaded Polyimide Under Electron-Beam Irradiation.," Proceedings of the 2013 IEE International Conference on Solid Dielectrics

QR Code (Right) can be scanned by a smartphone for a link to the Materials Physics Group website containing information on past presentations and published works.