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International Test Program for Synergistic Atomic Oxygen and VUV Exposure of Spacecraft Materials

Sharon Rutledge, Bruce Banks, and Joyce Dever
Glenn Research Center, Cleveland, Ohio

William Savage
Cleveland State University, Cleveland, Ohio

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Sharon Rutledge, Bruce Banks, and Joyce Dever
Glenn Research Center, Cleveland, Ohio

William Savage
Cleveland State University, Cleveland, Ohio

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INTERNATIONAL TEST PROGRAM FOR SYNERGISTIC ATOMIC OXYGEN AND VUV EXPOSURE OF SPACECRAFT MATERIALS

Sharon Rutledge, Bruce Banks, and Joyce Dever
National Aeronautics and Space Administration
Glenn Research Center at Lewis Field
21000 Brookpark Road, MS 309-2
Cleveland, Ohio 44135

William Savage
Cleveland State University
Cleveland, Ohio 44115

***ABSTRACT** - Spacecraft in low Earth orbit (LEO) are subject to degradation in thermal and optical performance of components and materials through interaction with atomic oxygen and vacuum ultraviolet radiation which are predominant in LEO. Due to the importance of LEO durability and performance to manufacturers and users, an international test program for assessing the durability of spacecraft materials and components was initiated. Initial tests consisted of exposure of samples representing a variety of thermal control paints and multilayer insulation materials that have been used in space. Materials donated from various international sources were tested alongside a material whose performance is well known such as Teflon FEP or Kapton H for multilayer insulation, or Z-93-P for white thermal control paints. The optical, thermal or mass loss data generated during the test was then provided to the participating material supplier. Data was not published unless the participant donating the material consented to publication. This paper presents a description of the types of tests and facilities that have been used for the test program as well as some examples of data that have been generated. The test program is intended to give spacecraft builders and users a better understanding of degradation processes and effects to enable improved prediction of spacecraft performance.*

1 – INTRODUCTION

Spacecraft in Low Earth Orbit (LEO) are subjected to many components of the environment such as atomic oxygen and vacuum ultraviolet (VUV) radiation which can cause them to degrade, thereby compromising performance or shortening their functional life. Sensitive surfaces such as thermal control paints, multilayer insulation (MLI), and optical surfaces are especially susceptible because small changes in their surface properties can have large effects on their functional ability. The Low Earth Orbit Spacecraft Materials Test Program (LEO-SMT) was initiated to assess the effects of simulated LEO exposure on current spacecraft materials in order to understand LEO degradation processes and to enable predictions of in-space durability. It is important not only to be able to select materials for future spacecraft which will survive the environment, but to be able to determine if currently flying spacecraft may fail due to interactions with the environment. By durability testing materials currently flying in LEO using ground based simulation facilities, degradation evidenced in these facilities can be compared with that observed on orbit. This will allow refinement of ground laboratory test systems and the development of algorithms to enable the prediction of performance of new materials in LEO based on ground laboratory testing. More

accurate predictions based on ground test data can lead to lower development costs and greater reliability.

A directed atomic oxygen beam system with synergistic VUV radiation exposure and in-situ reflectance measurement capability has been used to test thermal control paints and MLI materials supplied by a variety of international participants in the LEO-SMT program. This paper presents a description of the facilities used for the test program as well as some examples of the data that have been generated and provided to the participants.

2 – TEST FACILITIES

2.1 – Atomic Oxygen and VUV Exposure

Atomic oxygen was generated using an Electron Cyclotron Resonance (ECR) plasma source manufactured by Applied Science and Technology Inc. (ASTeX). The source was operated on pure oxygen gas. Microwaves from the source at 2.45 GHz in combination with the field from two electromagnets dissociate the molecular oxygen into atomic oxygen by energetic electron collisions. The atomic oxygen is directed into a beam by gas expansion from the higher pressure plasma formation region to the lower pressure sample region. This pressure difference can be 2-3 orders of magnitude. The vacuum chamber region where the samples are located is 71 cm in diameter by 1.71 m long. Pumping is provided by a diffusion pump, mechanical pump and roots-type blower that all operate on Fomblin (perfluorinated polyether) oil. The base pressure of the vacuum chamber is 2.7×10^{-4} Pa (2×10^{-6} Torr), but during operation can range from 0.027 Pa (2×10^{-4} Torr) to 0.107 Pa (8×10^{-4} Torr) depending on the oxygen gas flow rate.

For exposure of the thermal control paints, a realistic proportion of VUV radiation and atomic oxygen was desired which would simulate a radiator exposure for approximately five years on orbit at a 400 km altitude with 28.5 degree inclination. In order to achieve this, the atomic oxygen flux from the ECR had to be adjusted in proportion to the VUV radiation. This was accomplished by reducing the operating energy of the ECR to 700 W and adding a reducing plate at the entrance to the vacuum chamber from the plasma formation region. The reducing plate was made of 0.15 mm thick aluminum foil with a 3.175 cm diameter hole in the center for the beam to pass through. The VUV radiation from the ECR itself had to be eliminated as well. This was accomplished by using a series of fused silica panels to scatter the atomic oxygen and focus it onto the sample region while also hiding the samples from the source VUV with 0.127 mm thick aluminum foil that lines the primary scattering panels. The vacuum chamber and scattering apparatus is shown in Figures 1 and 2. The atomic oxygen flux during exposure was $1-2 \times 10^{15}$ atoms/cm²-sec. VUV radiation was provided by Hammamatsu deuterium lamps with magnesium fluoride windows to achieve a level of approximately 2.0-4.5 VUV suns (115-200 nm) at the sample location. The thermal control paints were applied by the manufacturers to 2.54 cm diameter aluminum substrates, 1.6 mm thick in order to fit into the sample holder. Figure 1 also shows the sample holder that is attached to a 3-axis positioning system. This system allows the samples to be moved while still under vacuum to an integrating sphere internal to the vacuum chamber where reflectance as a function of wavelength can be measured from 300 to 2500 nm without breaking vacuum. A multitude of safety interlocks and monitors allow the system to shut down in a safe manner should there be a power failure, microwave leak, loss of vacuum or other failure condition. Further details of the system can be found in previous publications. [Stid 93] [Rutl 96]

The MLI samples did not require in-situ reflectance measurements. They were also larger (approximately 5.08 x 5.08 cm) and covered a broader area, so the fused silica panels were

removed, the sample holder on the multiaxis positioner was retracted farther back into the chamber, and a flat sample holder plate installed in its place. The atomic oxygen flux during exposure was

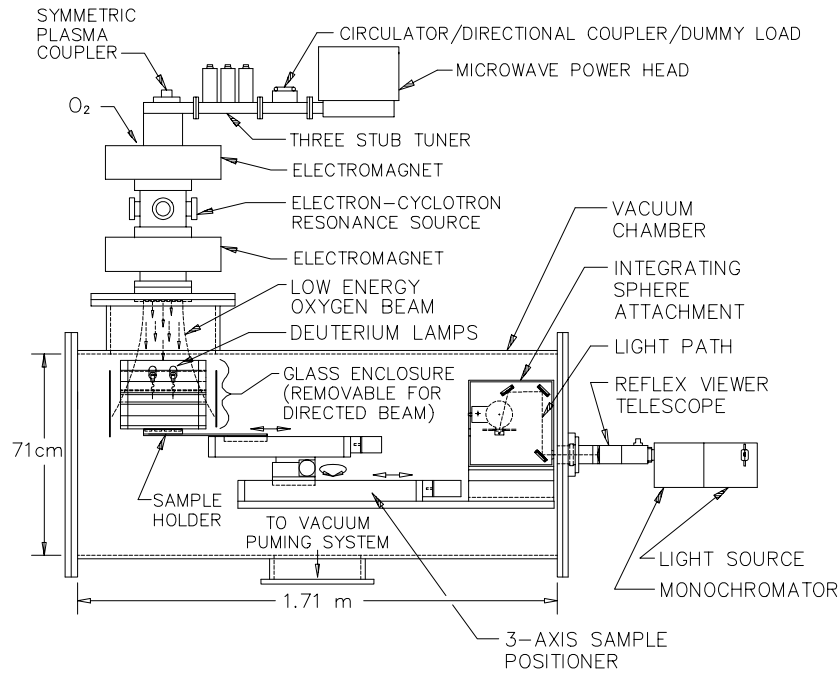


Figure 1. Side view schematic of vacuum chamber showing ECR source, reflectance measurement system, motion arm, and VUV blocking apparatus (From [Stid 93])

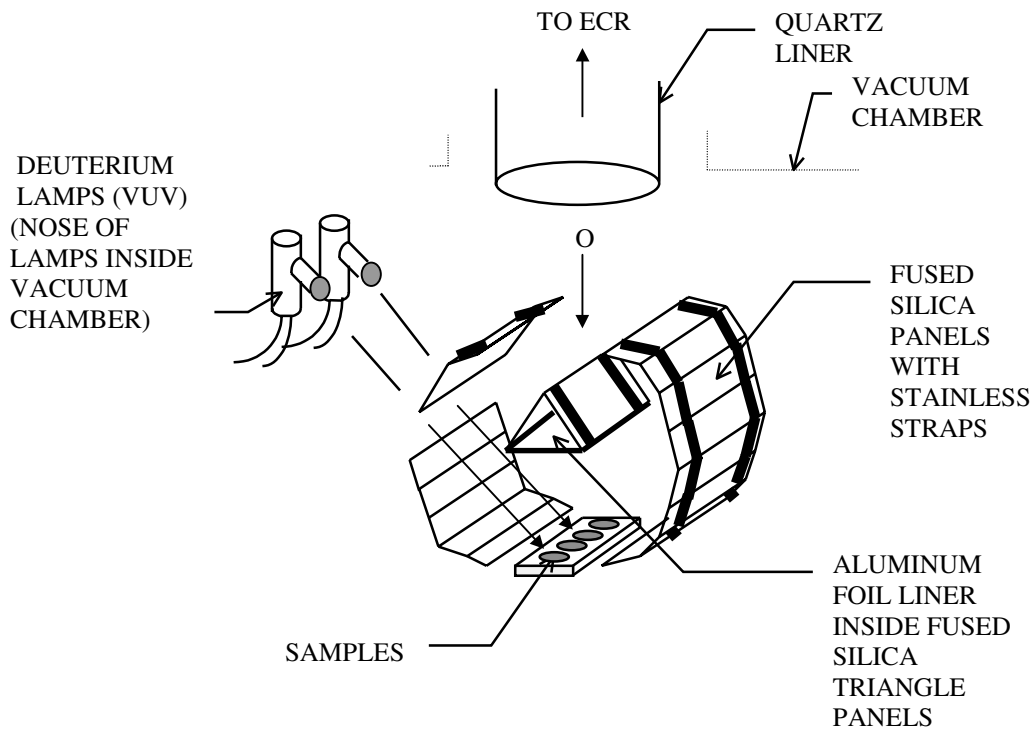


Figure 2. Atomic oxygen scattering and VUV blocking apparatus inside vacuum chamber

approximately 4.3×10^{15} atoms/cm²-sec with the ECR source operating at 700 W of microwave power. Because the VUV from the source was not blocked during exposure, VUV at an intensity of approximately 150 suns (predominantly at 130 nm) was provided to the samples. This was measured using an Acton Research photomultiplier and filter wheel calibrated to a deuterium lamp that was calibrated by the National Institute of Standards and Technology (NIST).

The size of the vacuum chamber also allows exposure of spacecraft components to atomic oxygen. The facility has been used to expose battens under tension [Stid 95], and a section of a solar array panel under load and rotating to simulate sweeping ram exposure [Fork 96]. The configuration of the facility allows a great flexibility in the materials tested and the manner in which they are exposed.

2.2 - Sample Characterization

The reflectance for the thermal control paint samples was measured in-situ using a reflectance measurement system (RMS) manufactured by Optronic Laboratories Inc. It was designed specifically for this vacuum chamber. It measures the hemispherical spectral reflectance of opaque samples over a wavelength range from 300 to 2500 nm. The reflectance in air was measured using a Lambda-9 UV-VIS-NIR spectrophotometer manufactured by Perkin Elmer. It measures the hemispherical spectral reflectance from 250 to 2500 nm. This instrument was used to determine the in-air solar absorptance of both the thermal control paints and the MLI. The solar absorptance was calculated by integrating the spectral absorptance (1.0-spectral reflectance) with respect to the air mass zero solar curve [Raus 80]. The change in mass of the exposed MLI samples was measured using a Sartorius Balance R-150-P. Thermal emittance was also measured for the MLI samples using a Gier Dunkle DB-100 Reflectometer. This instrument measures the reflectance at 355 K, which can be subtracted from unity to obtain the emittance at 355 K.

2.3 – Sample Preparation and Atomic Oxygen Effective Fluence Measurement

Aluminum substrates, 2.54 cm diameter by 1.6 mm thick, were supplied to participating thermal control paint manufacturers for paint application. MLI material was supplied by the participants as sheets from which 5.08 cm x 5.08 cm samples were cut for testing. The MLI was fully dehydrated in vacuum for the pre and post exposure mass measurements in order to minimize errors in mass measurement due to water absorption. [Rutl 86][Rutl 96] A vacuum dessicator operating at pressures between 8-13 Pa (60-100 mTorr) was used to dehydrate the samples for a period of 48 hrs prior to mass measurement. Samples were quickly removed from vacuum and weighed to reduce errors in mass measurement due to water reabsorption.

The effective fluence of atomic oxygen onto the samples was determined using a 2.54 cm diameter sample either punched from a 0.0127 cm thick sheet of DuPont Kapton H or a 0.0051 cm thick sheet of DuPont Teflon FEP. The witness coupon was selected based on the type of material being exposed. Kapton was used for the thermal control paints and polyimide-based MLI, and Teflon FEP was included in tests where there was FEP-based MLI. During exposure, the witness coupon was placed next to the samples being exposed. Knowing the density of the material, the measured mass loss per unit area during the exposure, and the erosion yield of the material in space, an effective fluence can be calculated. [Rutl 96] The effective fluence is not an absolute measure of atoms per unit area arriving at the surface. It is the calculation of the equivalent atoms per unit area in LEO that would produce the same damage as observed in the ground based facility and is strictly used as a means of comparison.

3 – TESTING OF THERMAL CONTROL PAINTS

As part of the test program, the reflectance of the thermal control paints exposed to atomic oxygen and VUV radiation was measured prior to exposure in air, prior to exposure in vacuum, during exposure at selected intervals in vacuum, and again at the end both in vacuum and in air. Spectral reflectance data was then plotted as a function of wavelength and sent to the participant that donated the samples. Figure 3 contains a typical spectral plot as a function of wavelength. This particular sample experienced a darkening upon atomic oxygen and VUV radiation exposure. The reflectance of this paint was found to remain fairly stable after an approximately 7.5×10^{20} atoms/cm² atomic oxygen effective fluence and 816 ESH VUV exposure. Data was not available in this particular exposure beyond these levels due to difficulties with the RMS system. The reflectance recovered slightly when the sample was exposed to air. Although not all paint samples experience a bleaching effect with air, it is important to make measurements in vacuum prior to venting the exposure chamber. This can provide verification of whether or not bleaching will occur, thereby giving a better indication of what the change in optical properties can be in space.

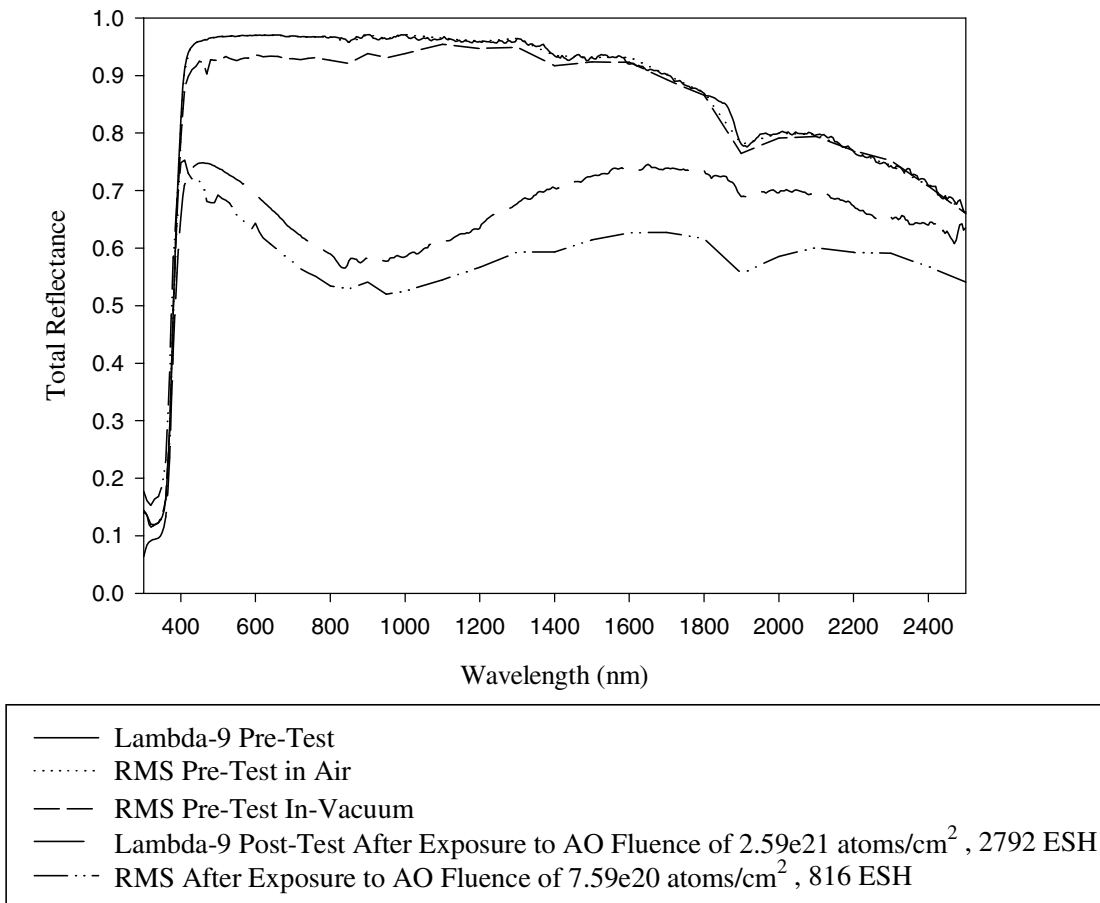


Figure 3. Hemispherical total spectral reflectance data for PSB Silicate both in air (before and after exposure with Lambda-9) and in vacuum (before and during with RMS)

Measured reflectance data was then used to calculate the absorptance spectral data that was integrated with respect to the air mass zero solar spectra to obtain the solar absorptance of each sample. These values were tabulated and also supplied to the participant along with an estimate of the VUV radiation and effective atomic oxygen dose experienced by each sample. These were estimated by calibration using a positional photodiode, and witness coupons respectively. The variation of the VUV dose is due to the sample position with respect to the pair of deuterium lamps. There is some overlap of the lamp output for the two samples in the central sample slots and more of a single lamp exposure for those on the edge.

Table I contains examples of the integrated solar absorptance data gathered for selected white thermal control paints. The paints listed that are from MAP are older formulations and do not represent their current paint products. These and other formulations were tested because they have been used on spacecraft in the past and can give the participant a basis for comparison and understanding of their spacecraft's performance.

Table I. White thermal control paint optical and exposure level data

Material and Manufacturer	Atomic Oxygen Effective Fluence (atoms/cm ²)	VUV Equivalent Sun Hours (115-200 nm)	Solar Absorptance (In-vacuum before exposure)	Solar Absorptance (In-vacuum after exposure)
Z-93-P (ITTRI)	4.1x10 ²¹	1130	0.09	0.11
PSB (MAP)	4.1x10 ²¹	1640	0.08	0.18
S13GPLO-1 (ITTRI)	4.1x10 ²¹	2200	0.18	0.18
SG120FD (MAP)	4.1x10 ²¹	1590	0.14	0.13

4 – TESTING OF MULTILAYER INSULATION

Solar absorptance data prior to and after atomic oxygen and VUV radiation exposure was obtained for multilayer insulation samples. An example of the data generated is shown in Table II. Thermal emittance was also measured prior to and after exposure and is included in the table. The variation in fluence dose noted in the table is due to the placement of the sample with respect to the atomic oxygen beam. With the scattering apparatus removed, the beam arriving at the samples follows a Gaussian distribution pattern.

In general, for the MLI material types tested to date, the solar absorptance increased when the metal reflector surface was oxidized. Where a stable metal reflector surface existed, the absorptance remained fairly stable even with a thinning of the first surface material. The samples were not completely shielded so some atomic oxygen was able to reach the back of the sample. This allowed some oxidation of the second surface reflector to take place. The emittance, however, seems to be much more dependent on the first surface. Thinning of the primary surface due to oxidation resulted in a decrease in thermal emittance. Materials that exhibited the least change had a coating on the exposed surface. However, if the coating is disturbed by processing, such as occurred with the UTC-025R-AAEN embossed aluminum coated polyimide, then atomic oxygen can enter defects in the coating at the site of the processing and attack the underlying material. In this case, there were embossed dimples that actually fell out after the MLI was exposed.

Figure 4 shows the mass loss experienced by selected MLI materials with atomic oxygen exposure. As would be expected, MLI with a first surface of either polyimide or Teflon FEP loses mass with increased atomic oxygen exposure. The polyimide loss rate is higher than that for FEP as a function of the effective fluence. Samples with a surface coating exhibited a much lower mass loss as a

function of effective fluence due to the coating acting as a barrier to attack by atomic oxygen. Where there were defects in the coating, such as with the embossed sample, the mass loss experienced was in between that of the coated and the uncoated samples.

Table II. Multilayer insulation optical, thermal and exposure level data

Material Description	Manufacturer	Approximate Atomic Oxygen Effective Fluence (atoms/cm ²)	In-Air Solar Absorptance Prior to Exposure	In-Air Solar Absorptance After Exposure	Emittance Prior to Exposure (355 K)	Emittance After Exposure (355 K)
UTC-025R-TANN (ITO/25 micron UPILEX Polyimide/Al)	UBE	5.0x10 ²¹	0.364	0.357	0.536	0.547
UTC-025R-AAEN (Al/25 micron UPILEX Polyimide/Al (Embossed))	UBE	2.47x10 ²¹	0.127	0.128	0.045	0.115
UTC-025R-AANN (Al/25 micron UPILEX Polyimide/Al)	UBE	5.0x10 ²¹	0.089	0.123	0.021	0.027
UTC-050R-AANN (Al/50 micron UPILEX Polyimide/Al)	UBE	5.0 x10 ²¹	0.089	0.157	0.02	0.047
UTC-050R-NANN (50 micron UPILEX Polyimide/Al)	UBE	7.29x10 ²⁰	0.397	0.494	0.769	0.723
Flex OSR FST-8403	Sumitomo Bakelite	5.0x10 ²¹	0.142	0.137	0.808	0.804
IP 139481 (50 micron Teflon/Silver-Inconel)	Sheldahl	2.07x10 ²²	0.053	0.092	0.638	0.416
IP 139480 (127 micron Teflon/Silver-Inconel)	Sheldahl	2.07x10 ²²	0.073	0.15	0.794	0.753
IP 207413 (50 micron Teflon/Al)	Sheldahl	2.07x10 ²²	0.116	0.113	0.647	0.453
IP 502303 (127 micron Teflon/Al)	Sheldahl	2.07x10 ²²	0.121	0.118	0.78	0.725

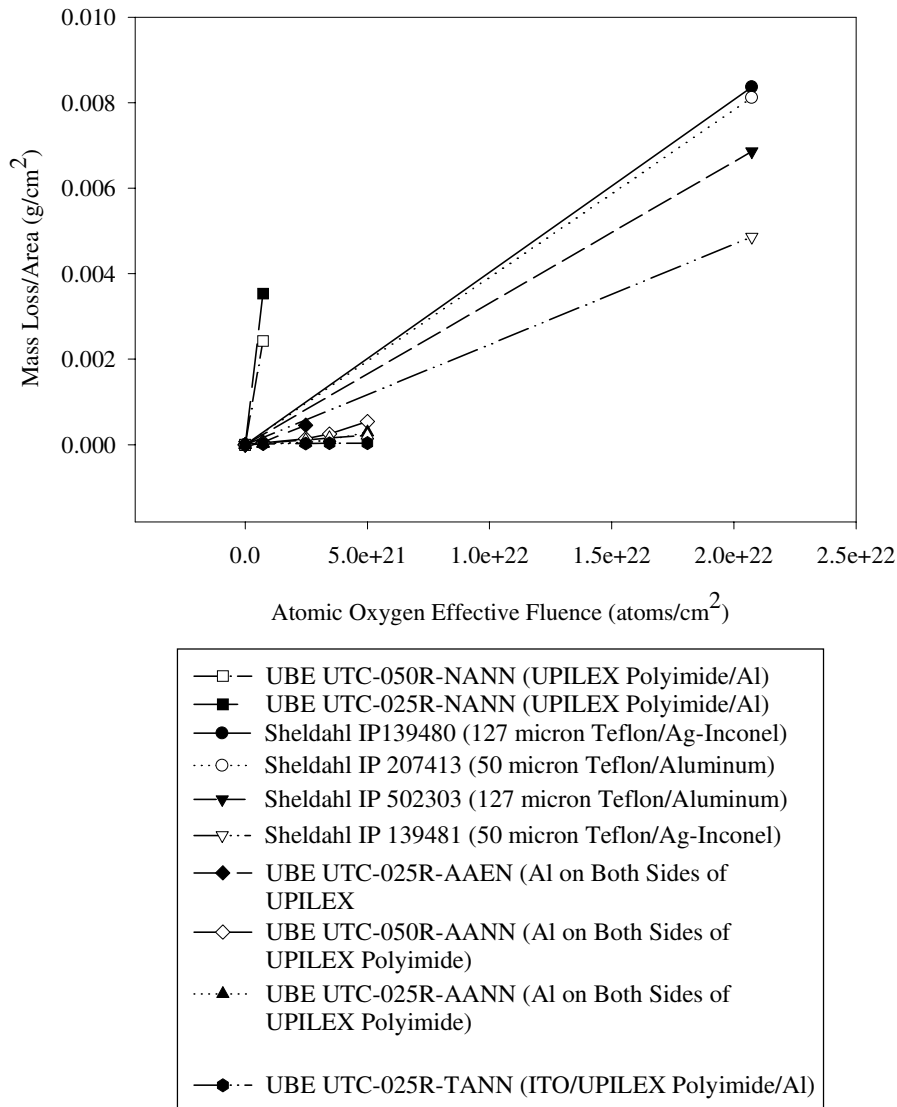


Figure 4. Mass loss per unit area for selected MLI materials as a function of atomic oxygen effective fluence

5 – LEO-SMT REPORT

Participants in the LEO Spacecraft Materials Test Program have received a brief description of the tests conducted as well as tables and plots of their data as presented in this paper. Each participant only receives the information on the materials they have donated and that of a well known baseline material such as Z-93-P. The results of the tests are only shared with others with the agreement of the participant donating material.

6 – CONCLUSIONS

The LEO-SMT program was initiated in order to give manufacturers and users information about the durability and performance of spacecraft materials and components that can be used to understand spacecraft performance and aid in selection of materials for future missions. Initial testing has focused on thermal control paints and multilayer insulation (MLI). The initial testing demonstrated that in-situ measurement of reflectance for white paints is important because it points

out the extent to which some paints darkened by exposure recover some of their original reflectance when exposed to air. It was also shown that second surface reflector integrity is important for maintaining the solar absorptance of MLI, while the integrity of the front polymer is important for maintaining the thermal emittance. Through this testing, general characteristics of classes of materials can be obtained as well as specific material performance information. Data is provided to each participant, for their own use, on the materials that they donate. Information from these tests along with available space data can also be used to develop and validate performance and durability prediction models. NASA welcomes the opportunity to consider additional international participants in this program that should greatly aid spacecraft designers of the future in material selection for LEO missions.

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