NASA/TM—2002-211479

Techniques for Measuring Low Earth Orbital Atomic Oxygen Erosion of Polymers

Kim K. de Groh and Bruce A. Banks
Glenn Research Center, Cleveland, Ohio

Rikako Demko
Cleveland State University, Cleveland, Ohio

March 2002

The NASA STI Program Office . . . in Profile

Since its founding, NASA has been dedicated to the advancement of aeronautics and space science. The NASA Scientific and Technical Information (STI) Program Office plays a key part in helping NASA maintain this important role.

The NASA STI Program Office is operated by Langley Research Center, the Lead Center for NASA's scientific and technical information. The NASA STI Program Office provides access to the NASA STI Database, the largest collection of aeronautical and space science STI in the world. The Program Office is also NASA's institutional mechanism for disseminating the results of its research and development activities. These results are published by NASA in the NASA STI Report Series, which includes the following report types:

- TECHNICAL PUBLICATION. Reports of completed research or a major significant phase of research that present the results of NASA programs and include extensive data or theoretical analysis. Includes compilations of significant scientific and technical data and information deemed to be of continuing reference value. NASA's counterpart of peer-reviewed formal professional papers but has less stringent limitations on manuscript length and extent of graphic presentations.

- TECHNICAL MEMORANDUM. Scientific and technical findings that are preliminary or of specialized interest, e.g., quick release reports, working papers, and bibliographies that contain minimal annotation. Does not contain extensive analysis.

- CONTRACTOR REPORT. Scientific and technical findings by NASA-sponsored contractors and grantees.

- CONFERENCE PUBLICATION. Collected papers from scientific and technical conferences, symposia, seminars, or other meetings sponsored or cosponsored by NASA.

- SPECIAL PUBLICATION. Scientific, technical, or historical information from NASA programs, projects, and missions, often concerned with subjects having substantial public interest.

- TECHNICAL TRANSLATION. English-language translations of foreign scientific and technical material pertinent to NASA's mission.

Specialized services that complement the STI Program Office's diverse offerings include creating custom thesauri, building customized data bases, organizing and publishing research results . . . even providing videos.

For more information about the NASA STI Program Office, see the following:

- Access the NASA STI Program Home Page at ***http://www.sti.nasa.gov***

- E-mail your question via the Internet to help@sti.nasa.gov

- Fax your question to the NASA Access Help Desk at 301–621–0134

- Telephone the NASA Access Help Desk at 301–621–0390

- Write to:
  NASA Access Help Desk
  NASA Center for AeroSpace Information
  7121 Standard Drive
  Hanover, MD 21076

NASA/TM—2002-211479

Techniques for Measuring Low Earth Orbital Atomic Oxygen Erosion of Polymers

Kim K. de Groh and Bruce A. Banks
Glenn Research Center, Cleveland, Ohio

Rikako Demko
Cleveland State University, Cleveland, Ohio

Prepared for the
2002 Symposium and Exhibition
sponsored by the Society for the Advancement of
Materials and Process Engineering
Long Beach, California, May 12–16, 2002

National Aeronautics and
Space Administration

Glenn Research Center

March 2002

Available from

NASA Center for Aerospace Information
7121 Standard Drive
Hanover, MD 21076

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22100

Available electronically at http://gltrs.grc.nasa.gov/GLTRS

TECHNIQUES FOR MEASURING LOW EARTH ORBITAL ATOMIC OXYGEN EROSION OF POLYMERS

Kim K. de Groh and Bruce A. Banks
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Rikako Demko
Cleveland State University
Cleveland, Ohio 44115

ABSTRACT

Polymers such as polyimide Kapton® and Teflon® FEP (fluorinated ethylene propylene) are commonly used spacecraft materials due to their desirable properties such as flexibility, low density, and in the case of FEP, a low solar absorptance and high thermal emittance. Polymers on the exterior of spacecraft in the low Earth orbit (LEO) environment are exposed to energetic atomic oxygen. Atomic oxygen reaction with polymers causes erosion, which is a threat to spacecraft durability. It is therefore important to understand the atomic oxygen erosion yield (E, the volume loss per incident oxygen atom) of polymers being considered in spacecraft design. The most common technique for determining E is through mass loss measurements. For limited duration exposure experiments, such as shuttle experiments, where the atomic oxygen fluence is often so low that mass loss measurements can not produce acceptable uncertainties, recession measurements based on atomic force microscopy analyses can be used. Equally necessary to knowing the mass loss or recession depth for determining the erosion yield of polymers is the knowledge of the atomic oxygen fluence that the polymers were exposed to in space. This paper discusses the procedures and relevant issues for mass loss and recession depth measurements for passive atomic oxygen erosion yield characterization of polymers, along with techniques for active atomic oxygen fluence and erosion characterization. One active atomic oxygen erosion technique discussed is a new technique based on optical measurements. Details including the use of both semi-transparent and opaque polymers for active erosion measurement are reviewed.

1. INTRODUCTION

Polymers such as polyimide Kapton and Teflon FEP are commonly used spacecraft materials due to their desirable properties such as flexibility, low density, electrical properties and in the case of FEP, a very low solar absorptance and high thermal emittance. A few specific examples of the use of polymers on the exterior of spacecraft include: back surface metallized Teflon FEP thermal control materials on the Hubble Space Telescope, polyimide Kapton solar array blankets (structural member) and Teflon ePTFE (expanded polytetrafluorethylene) cable insulation on the International Space Station.

Polymers on the exterior of spacecraft are exposed to atomic oxygen in the LEO environment. Atomic oxygen is formed when short wavelength ultraviolet radiation (>5.12 eV, <243 nm) from the Sun dissociates molecular oxygen in the upper atmosphere.[1] Although atomic oxygen is the predominant species in LEO (below ≈1,000 km)[2], these neutral oxygen atoms have mean free paths on the order of 10^4 m at 400 km, resulting in extremely low probabilities of re-association. A typical LEO spacecraft orbits the Earth with a velocity on the order of 7.7 km/sec as it rams into the atmospheric oxygen (hence the term ram atomic oxygen). The flux of atomic oxygen at International Space Station (ISS) altitudes is approximately 1.0×10^{14} atoms/cm^2 sec for normal incident ram surfaces, and the average energy of an oxygen atom impacting spacecraft at ram velocities is 4.5 eV.[3] A number of processes can take place when an oxygen atom strikes a spacecraft surface at orbital velocities. These include chemical reaction with surface atoms or adsorbed molecules, elastic scattering, scattering with partial or full thermal accommodation, recombination, or excitation of ram species.[4] Because the oxidation product for most polymers is a gas, erosion results.

Atomic oxygen erosion of polymers in LEO is a serious threat to spacecraft durability. For example, depths of more than 0.0127 cm (0.005") thickness of Kapton and Mylar sheets were eroded away after 5.8 years in LEO on the leading edge, or ram atomic oxygen surface, of the Long Duration Exposure Facility (LDEF).[5] The atomic oxygen fluence for the leading edge of LDEF was 8.99×10^{21} atoms/cm^2.[6] In addition to the erosion of the Kapton and Mylar films, ≈1 ply (0.0127 cm or 0.005") of graphite-epoxy and 0.0025 cm (0.001") of Teflon FEP were also eroded away after 5.8 years on LDEF's leading edge.[5] Figure 1 shows a fluoropolymer (polychlorotrifluoroethylene) that was exposed to near normal incidence ram atomic oxygen on the leading edge of LDEF. A cone-like or carpet type morphology developed, which is characteristic of directed atomic oxygen erosion for materials with gaseous oxidation products. Protective coatings are effective in preventing atomic oxygen erosion, yet oxidation erosion of the underlying polymer can occur at pinhole and scratch defects through undercutting erosion.[7–9]

In addition to the obvious potential degradation to the structural stability of polymers, such as the support of solar cells on Kapton polyimide blankets on the International Space Station solar array, atomic oxygen is a threat to other polymer properties. For example, the thermal emittance of thin polymer thermal control materials is dependent upon the thickness of the polymer. Thus erosion of the polymer by atomic oxygen can result in a reduced thermal emittance capability which would give rise to increases in the spacecraft temperature if the solar absorptance does not change much, which would be the case for very thin films. Therefore, it is essential to understand the atomic oxygen erosion yield (E, the volume loss per incident oxygen atom) of polymers

being considered in spacecraft design. Procedures have been established for ground laboratory atomic oxygen interaction evaluation of materials for space applications.[10] But, although ground laboratory procedures have been established and are used for erosion yield determination, actual in-space data is more reliable and therefore greatly more desired than ground test data.

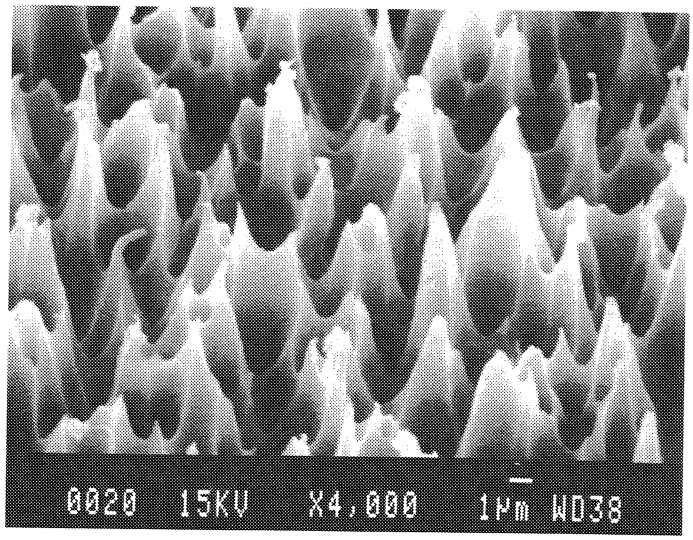


Figure 1. This fluoropolymer was located on the leading edge of LDEF and shows the recession morphology which is typical of directed ram atomic oxygen erosion.

The most common technique for determining E is through passive mass loss measurements. For limited duration exposure experiments, such as shuttle experiments, where the atomic oxygen fluence is often so small that mass loss measurements can not produce acceptable uncertainties, recession measurements based on atomic force microscopy analyses can be used. Such a recession measurement technique, which uses intimate contact selective protection of polymer samples combined with post-flight atomic force microscopy (AFM) analysis, has been developed by NASA Glenn Research Center.[11] Equally necessary to knowing the mass loss or recession depth for determining the erosion yield of polymers is the knowledge of the atomic oxygen fluence that the polymers were exposed to in space. This paper discusses the procedures and relevant issues for mass loss and recession depth measurements for passive atomic oxygen erosion yield characterization of polymers, along with techniques for active atomic oxygen fluence and erosion characterization. One active atomic oxygen erosion technique discussed is a new technique based on optical measurements. Details of this optical technique are discussed.

2. PASSIVE ATOMIC OXYGEN EROSION MEASUREMENT TECHNIQUES

2.1. Mass Loss Measurements The most common technique for determining the E of flight samples is through passive mass loss measurements. These measurements are made by obtaining mass measurements of the flight sample before and after flight. The erosion yield of the sample (E_S) is then calculated through the following equation:

$$E_S = \frac{\Delta M_S}{(A_S \rho_S F)} \tag{1}$$

where

E_s = *erosion yield of the flight sample* ($cm^3/atom$)
ΔM_s = *mass loss of the flight sample* (g)
A_s = *surface area of the flight sample exposed to atomic oxygen attack* (cm^2)
ρ_s = *density of sample* (g/cm^3)
F = *fluence of atomic oxygen* ($atoms/cm^2$)

The atomic oxygen fluence (F) can be characterized also passively by determining the mass loss of a Kapton H witness sample because Kapton has a well characterized erosion yield in the LEO environment, and can be calculated using the following equation:

$$F = \frac{\Delta M_K}{(A_K \rho_K E_K)} \tag{2}$$

where

ΔM_K = *mass loss of Kapton witness sample* (g)
A_K = *surface area of Kapton witness sample exposed to atomic oxygen* (cm^2)
ρ_K = *density of Kapton witness sample* ($1.42\ g/cm^3$)
E_K = *erosion yield of Kapton witness sample* ($3.0\times10^{-24}\ cm^3/atom$)

Thus, for passive flight data

$$E_S = E_K \frac{\Delta M_S A_K \rho_K}{\Delta M_K A_S \rho_S} \tag{3}$$

2.1.1. Rehydration/Dehydration Issues One of the critical issues with obtaining accurate erosion yield data from mass measurements is making sure that dehydrated mass measurements are used. Many polymer materials, such as Kapton, are very hygroscopic and therefore their mass can fluctuate significantly with humidity and temperature. Therefore, for accurate mass loss measurements to be obtained, it is necessary that the samples be fully dehydrated (in a vacuum desiccator, for example) prior to measuring the mass for both the pre-flight and post-flight measurements. Even in doing so, error can be introduced into the data because hygroscopic materials will start absorbing water as soon as they are exposed to ambient atmosphere. Therefore, even with careful techniques, variations in the weight of a sample from day to day can be significant with respect to the very small changes in mass often associated with low fluence exposures. For this reason is it recommended that weight loss of samples be measured using vacuum dehydrated samples as specified in ASTM E 2089-00 (reference 10).

2.2. Recession Techniques Recession measurements have been used for erosion yield determination based on erosion depth step-heights. The erosion or recession depth can be measured from a protected surface using profilometry with a stylus profilometer, or with scanning electron microscopy (SEM), optical interferometry or atomic force microscopy.[12] If the

surface is protected by a mesh placed over the surface or by applying a protective film onto the surface, the erosion yield, E, can be calculated simply through the following equation

$$E = y/F \tag{4}$$

where

y= step height, or erosion depth (cm)

For very small erosion depths, techniques for step-height determination such as stylus profilometry, SEM and even optical interferometry are not sensitive enough or have other faults. For example, a SEM is not typically set up to obtain accurate erosion depth measurements (Z direction) and making stylus measurements on soft polymers can be problematic. The only technique that can provide accurate very small step-height measurements is AFM. An atomic force microscope can have a lateral resolution of 10-20 Å and sub-angstrom vertical resolution.[13]

2.2.1. Mesh Techniques Metal meshes such as stainless steel or nickel etched mesh have been used to protect surfaces from atomic oxygen attack resulting in step-height changes in polymers for atomic oxygen erosion yield measurement.[12] A frequent problem with using a mesh technique is that the mesh needs to be very thin and in intimate contact with the polymer to accurately measure the step-height. However, it is very difficult to place a thin mesh in intimate contact with the polymer. The quantification of the error associated with the thickness and intimacy of the mesh with respect to the polymer (specifically the height of the exposed mesh surface to the protected polymer surface) is similar to that reviewed in the section 2.4. And as will be seen, the height plays a critical role in the error of the erosion yield measurements.

2.2.2. Film Techniques Coatings such as metals, SiO_2, and Al_2O_3 are listed as serving as potential effective masks as long as the coatings are ≥ 20 nm thick.[12] The problem with the mask technique is that the thickness of the coating needs to be very accurately known for low fluence exposures and the coating must end cleanly and sharply. That is to say a gradual thinning of the protective coating at its margin (which commonly occurs) will contribute to step-height errors.

2.3. AFM Recession Technique (Intimate Protection & Post-Flight AFM Analysis) The recession depth technique developed at Glenn involves pre-flight protection of the sample surface using isolated intimate contact of small particles.[11] These particles, such as salt crystals or mica powder are applied either by salt spraying or mica dusting resulting in isolated protective particles. The particles are then removed post-flight (i.e., washing off the salt with distilled water and then nitrogen gas drying) and erosion depth step-height or recession measurements are then obtained using AFM. Information regarding salt spraying and mica dusting techniques, and AFM profiling are discussed in sections 2.3.1, 2.3.2, and 2.3.3, respectively. Figure 2 is an example of how a small protective particle (i.e., salt likely deposited from ocean mist on the launch pad) can protect the underlying polymer from atomic oxygen erosion.

2.3.1. Salt Spraying and Salt Crystal Variations Protective salt particles are applied to the sample substrate by spraying a saturated salt solution using an atomizer. Ideally it is desired to have a uniform distribution of small cubic crystals. Experiments conducted for the development of a shuttle flight experiment called PEACE (Polymer Erosion And Contamination Experiment) have shown that salt spraying can result in a variety of different salt particles being formed.[11]

Examples of these particle types are small or large cubic crystals, oval or asymmetric spherical particles, crystals with salt "rings" around them and ring deposits with a doughnut-shaped appearance (formed when the solution is heated). These examples are shown in Figure 3. Issues related to salt rings and possible condensation build-up effects (while down at the Cape) are provided by de Groh et al. in reference 11.

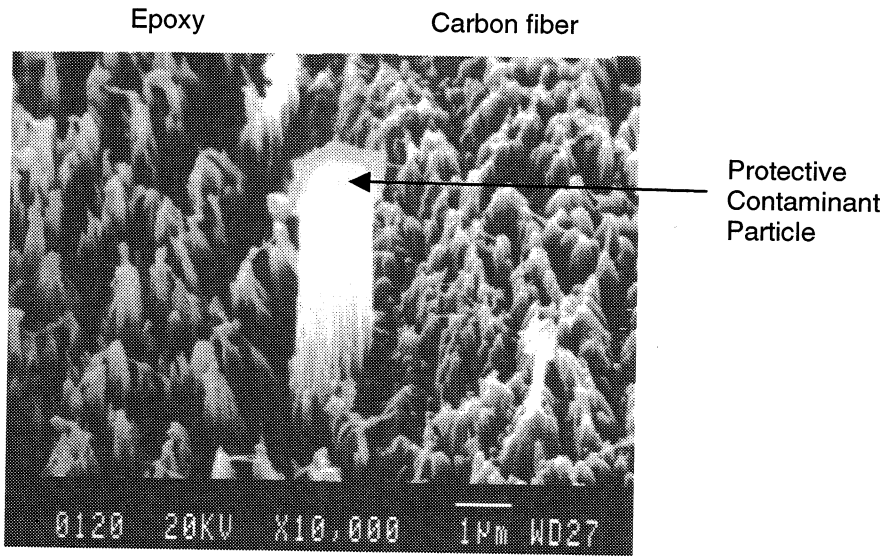


Figure 2. High magnification image of a graphite epoxy composite sample that was flown as part of the Environmental Oxygen Interaction with Materials (EOIM-III) experiment aboard STS-46, exposed to an atomic oxygen fluence of 2.3×10^{20} atoms/cm^2.

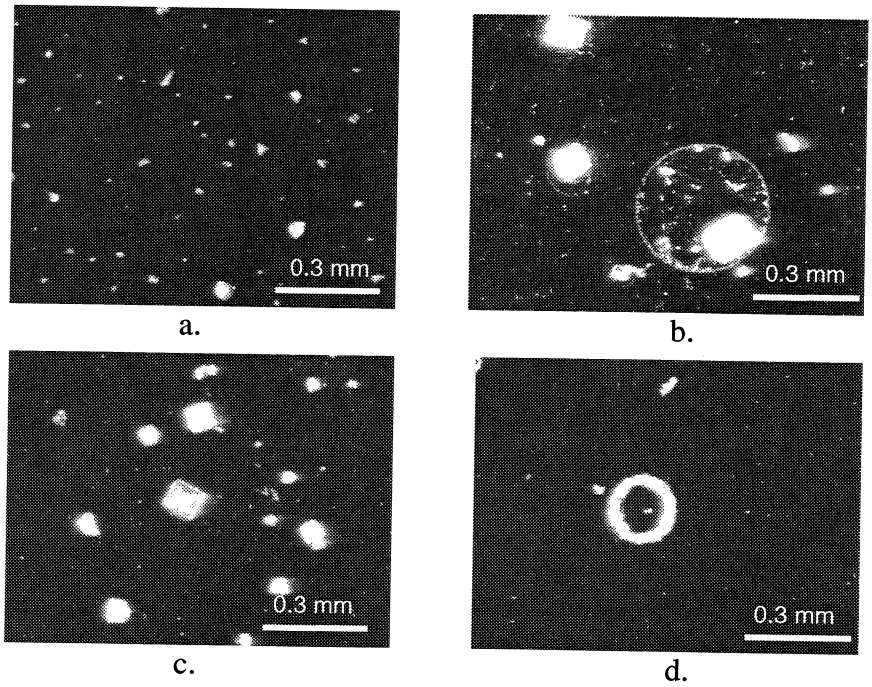
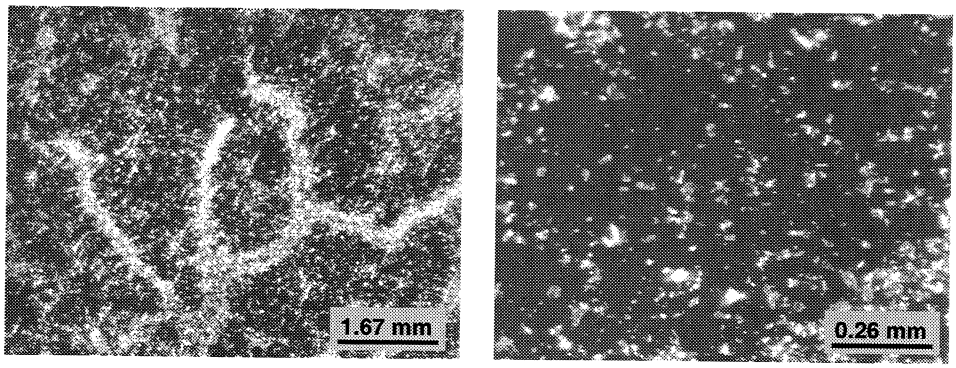


Figure 3. Different types of salt particles formed on a Kapton HN substrate during salt-spraying: a. small irregular shaped particles, b. salt rings around large (roughly cubic) crystals, c. large cubic and irregular shaped crystals and d. ring deposits.

2.3.2. Dusting Mica For samples that can not be protected with salt particles, such as polymers that are water sensitive or those where condensation effects are a problem, other types of protective materials need to be considered. Mica dust is proposed as an alternative protective particle. Mica dust has the advantage of being extremely thin and flat. Experiments have been conducted with mica dusting for the PEACE flight project.[11] Figure 4 is an example of mica dusted Kapton samples. One of the problems encountered with mica dusting was mica clustering due to electrostatic charge build-up on the sample. An example of such clustering is shown in Figure 4a. Figure 4b shows a Kapton sample with more uniformly distributed mica particles.



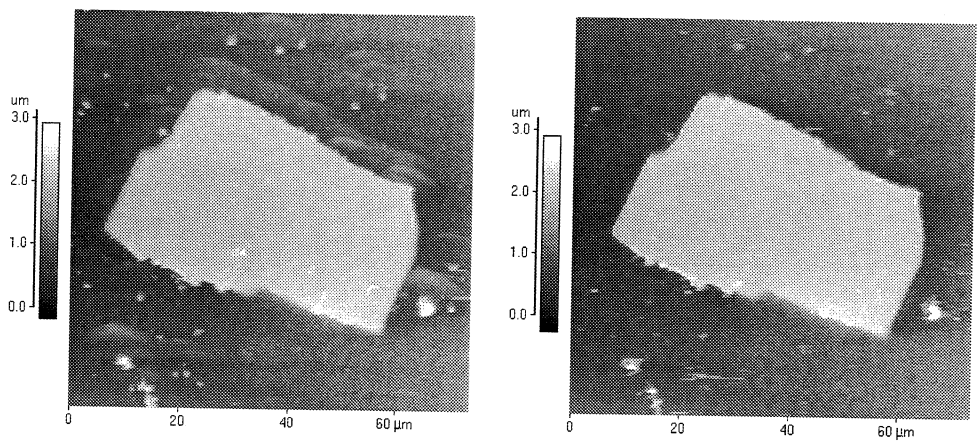
a. b.

Figure 4. Mica dusted Kapton: a. example of mica clustering due to electro-static charge problem, and b. example of good dust distribution.

2.3.3. AFM Procedures and Issues: Contact versus Non-Contact Imaging A study has been conducted by de Groh et al. to determine which method of AFM data collection, contact versus non-contact, yielded the best estimate of erosion depth.[11] The concern was that contact imaging might produce less accurate results because of flattening of the fine carpet like texture of the eroded area. On the other hand, non-contact imaging (where the cantilevered probe "floats" above the surface) is much more difficult to obtain. An actual flight sample that was prepared for erosion depth measurements by salt-protecting the surface and then exposed to a low LEO atomic oxygen fluence while in the shuttle bay was used to obtain contact and non-contact AFM erosion depth data. Figure 5 shows a comparison of images taken first in non-contact mode and then in contact mode of a typical salt-protected "island" on Kapton. The results indicated that there was no statistically significant difference between the measured step heights using the two different measurement methods and atomic oxygen erosion depth can be estimated using either AFM mode. Because contact-mode measurements are much easier to make, one can use the technique for atomic oxygen erosion depth measurement without fear of significantly degrading the sample. Details of the flight sample and the procedures are provided in reference 11.

2.4. Uncertainty Comparisons between Mass Loss and AFM Recession Techniques A comparison of the mass loss and erosion depth techniques with respect to their associated uncertainties has been evaluated by Banks et al. and more recently by de Groh et al., as reported in references 14 and 11, respectively. Figure 6 provides a comparison of the uncertainties, in terms of percent probable error, of the atomic oxygen erosion yields as a function of atomic oxygen fluence for the mass loss and AFM recession techniques for the more recently conducted analysis.[11] As can be seen in Figure 6, the uncertainty in the erosion yield as determined by the AFM recession measurement technique is highly dependent on both the fluence and on the

thickness of the protective particle. This is because thicker particles produce a much fuzzier step height erosion edge, upon which AFM analyses are dependent. Also, as seen in Figure 6, for low atomic oxygen fluences the AFM recession measurement technique, for thin protective particles (<17 μm thick) is more sensitive than the mass loss technique.[11] The analysis indicates that AFM erosion depth measurements for 10 μm thick protective particles, have approximately one half the uncertainty of mass loss measurements for an atomic oxygen fluence exposure of 1×10^{19} atoms/cm^2: 7.74% probable error for recession measurements and 13.1% for mass loss measurements.[11] For a fluence of 2×10^{19} atoms/cm^2, the probable error is 6.72% for the mass loss technique and 4.23% for the AFM recession technique for a 10 μm thick particle.[11]



a. b.

Figure 5. A typical pair of AFM topographic images from a salt-protected region of a Kapton flight sample: a. image taken in NC mode, and b. image obtained in contact mode.

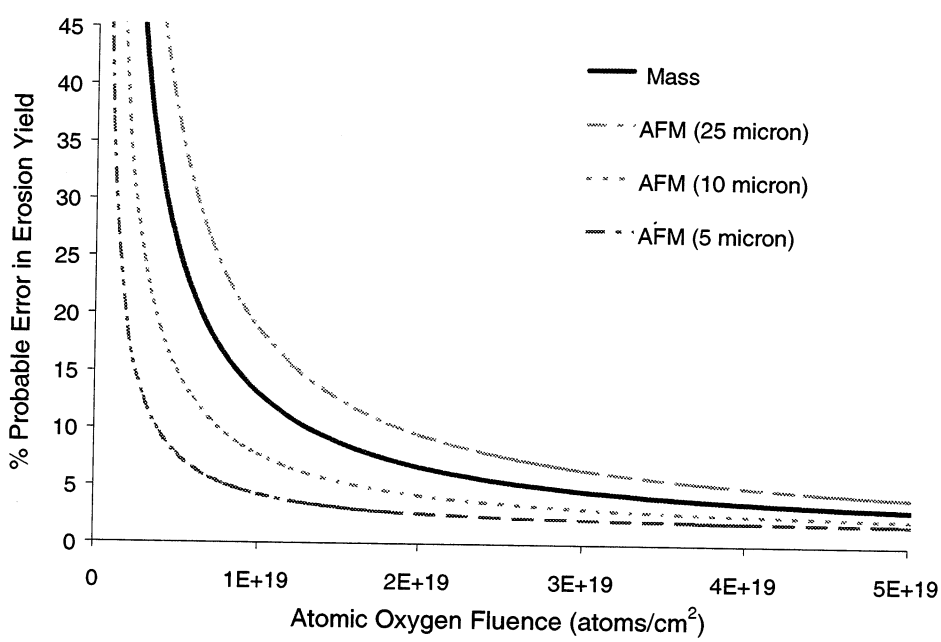


Figure 6. Comparison of the percent probable error in the atomic oxygen erosion yield as a function of atomic oxygen fluence for the mass loss technique and the AFM recession technique for three different protective particle sizes.

For a reasonable flight experiment goal, the error analyses indicate that a 5% probable error in erosion yield requires an atomic oxygen fluence of 2.78×10^{19} atoms/cm^2 for the mass loss technique and 1.64×10^{19} atoms/cm^2 for the AFM recession technique using 10 μm thick particles.

2.5. Additional Comparisons between AFM Recession and Mass Loss Techniques In addition to the fact that the AFM recession technique can be more accurate for low atomic oxygen fluence exposures, this technique has other desirable advantages as compared to the mass loss technique. One advantage of the AFM recession technique is that no pre-flight data acquisition is necessary. This means that less sample handling is necessary, and it eliminates the additional error introduced in taking sample measurements over a potentially extended period of time (samples often need to be characterized up to a year or more a head of a shuttle mission). Another significant advantage is that very small sample areas can be used. Also, multiple types of polymers can be put together as part of one flight sample. This is not possible with mass loss measurements. The disadvantage of the AFM recession technique is that an AFM is necessary, and there is more work involved in the post-flight data analyses.

3. ACTIVE ATOMIC OXYGEN EROSION MEASUREMENT TECHNIQUES

Under ideal circumstances one may have the opportunity to make active measurements of atomic oxygen erosion. This may be desired to measure atomic oxygen fluence or the atomic oxygen erosion yield of a test material as a function of time. Because atomic oxygen erosion yield measurement requires the measurement of both the atomic oxygen fluence and the thickness loss (or mass loss) of the test material, one typically needs to measure a material of known erosion yield for the fluence data at the same time as measuring the erosion of the test material.[15] For fluence measurement purposes, Kapton H polyimide, with a LEO atomic oxygen erosion yield of 3.0×10^{-24} cm^3/atom, is the most widely accepted witness material. Other materials that have been used include carbon, diamond-like carbon and silver. It should be noted that for test materials which have much lower erosion yields than the witness sample, the percent probable error in the erosion yield data will increase due to the greater uncertainty in the test sample measurement. The following sections (3.1 to 3.3) discuss various types of active atomic oxygen fluence measurement techniques or devices, commonly known as dosimeters, along with active atomic oxygen erosion measurement techniques.

3.1. Quartz Crystal Microbalance Measurements Quartz crystal microbalances (QCM's) have been used to measure erosion of polymers and atomic oxygen fluence by coating the exposed face of the crystal with hydrocarbon polymers or carbon films. Two difficulties are present with use of QCM's. First the housing of the QCM's do not allow measurement of atomic oxygen arriving at high angles from normal, because of shadowing effects. The housing may also cause atomic oxygen to be scattered (or reflected) onto the QCM face at a higher concentration along the edge where the atomic oxygen scatters thus giving one a deceptively high erosion measurement as the resulting erosion is an averaged reading. The density and structure of the polymer must be known if one wants to compare results with erosion yield of well-accepted materials. High fluence missions may also require unacceptably thick coating

over the QCM face. Although QCM measurement techniques have many potential calibration problems, they allow very sensitive measurements for low fluence missions.

3.2. Electrically Conductive Coating Sensors Electrically conductive coatings such as carbon, silver or osmium can be used to measure atomic oxygen fluence. However, only those elements or compounds that have volatile oxidation products will have a linear increase of in-the-plane electrical conductivity with atomic oxygen fluence. Silver develops an oxide that is larger than the elemental silver. The silver oxide is self-shielding and over time spalls randomly. Thus the response becomes less sensitive to atomic oxygen in a rather difficult to predict manner. Atomic oxygen oxidizes osmium to a very toxic OsO_4, but the oxide is only volatile if the temperature is sufficiently high which may be difficult to predict thus leading to uncertainty in its response to atomic oxygen. One can layer conductive thin-film carbon coated polymers (back-surface coated) in a multilayer stack to measure atomic oxygen erosion of the polymer by sensing the loss of conductivity of each layer of a stack.

3.3. Optical Erosion Measurement Techniques Sunlight or artificial light can be used to measure the erosion of semitransparent or opaque polymers as a result of atomic oxygen attack. The technique is simple and adaptable to a rather wide range of polymers providing that they have a sufficiently high optical absorption coefficient. If one covers a photodiode with a uniformly thick sheet of semitransparent polymer such as Kapton H polyimide, then as atomic oxygen erodes the polymer the short circuit current from the photodiode will increase in an exponential manner with fluence. This nonlinear response with fluence results in a lack of sensitivity for measurement of low atomic oxygen fluences. An optical technique is useful to either measure atomic oxygen fluence or erosion depending upon the information desired. To measure atomic oxygen erosion yield of a test material, one would need to have two photodiode sensors, one for the test material and one which uses a known erosion yield material (such as Kapton) to measure the atomic oxygen fluence.

However, if one uses a variable thickness polymer or carbon sample as illustrated in Figure 7 then a linear response can be achieved for opaque materials using a parabolic well for a circular geometry detector (see Figure 7a) or a V-shaped well for a rectangular-geometry detector (see Figure 7b). Variable thickness samples can be fabricated using many thin polymer layers. For semitransparent polymers such as Kapton H polyimide, there is an initial short circuit current which is greater than zero. This current has a slightly nonlinear dependence upon atomic oxygen fluence compared to opaque materials such as black Kapton as shown in Figure 8. For Figure 8 the total thickness of Kapton H was assumed to be 0.03 cm. Based on preliminary measurements, Figure 8 was generated based on a total reflectance (ρ) of 0.0424 and an optical absorption coefficient (α) of 146.5 cm^{-1}.

For semitransparent polymers, the short circuit current of the photodiode, I_T, can be approximated by

$$I_T = \frac{I_0 EF}{L} + \frac{I_0(1-\rho)}{\alpha L}\left[1 - e^{-\alpha(L-EF)}\right] \tag{5}$$

where:

I_o = *Short circuit current with no polymer present (mA)*
E = *Atomic oxygen erosion yield of the polymer* ($cm^3/atom$)
F = *Atomic oxygen fluence* ($atoms/cm^2$)
ρ = *Total reflectance*
α = *Optical absorption coefficient of the polymer* (cm^{-1})
L = *Thickness of the polymer layer (cm)*

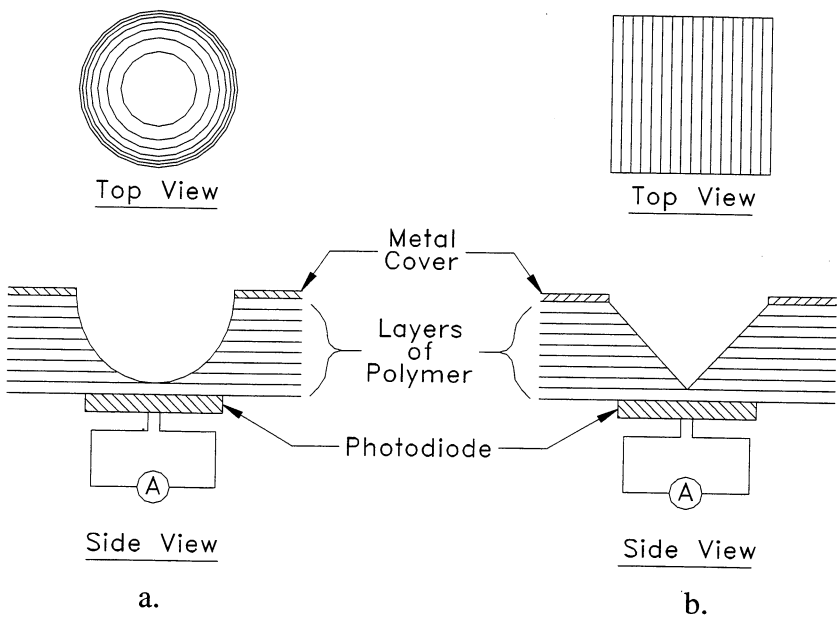


Figure 7. Optical atomic oxygen erosion measurement technique: a. Circular parabolic well polymer layer, and b. Rectangular V-shaped polymer layer.

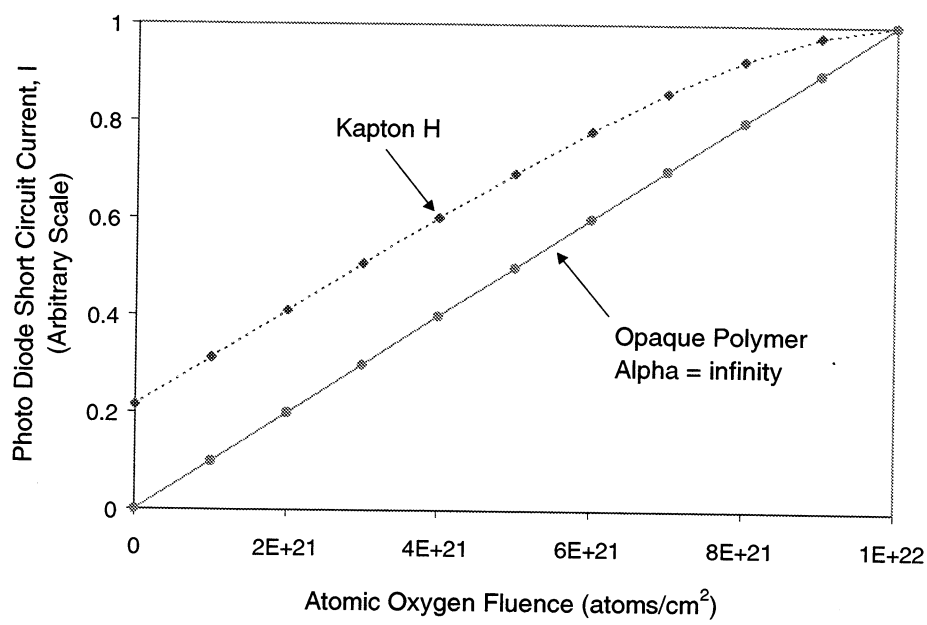


Figure 8. Short circuit current as a function of atomic oxygen fluence for the optical atomic oxygen erosion measurement techniques shown in Figure 7.

As previously stated, measurement of the erosion yield of a sample requires two photodiode measurements: one to measure the fluence of a known erosion yield polymer such as Kapton H polyimide and the second to measure the erosion depth, y, of the test polymer. This is accomplished in this optical erosion technique by a plot of the photodiode short circuit current of the fluence witness (based on the witness material optical absorptance and reflectance) as a function of atomic oxygen fluence, as shown in Figure 9a. And also, as shown in Figure 9b, by a plot of the short circuit current of the test sample (based on the test sample optical absorptance and reflectance) as a function of the erosion depth, y, where y is substituted for EF in equation 5. The atomic oxygen erosion yield is then simply calculated by $E= y/F$, as discussed in section 2.2.

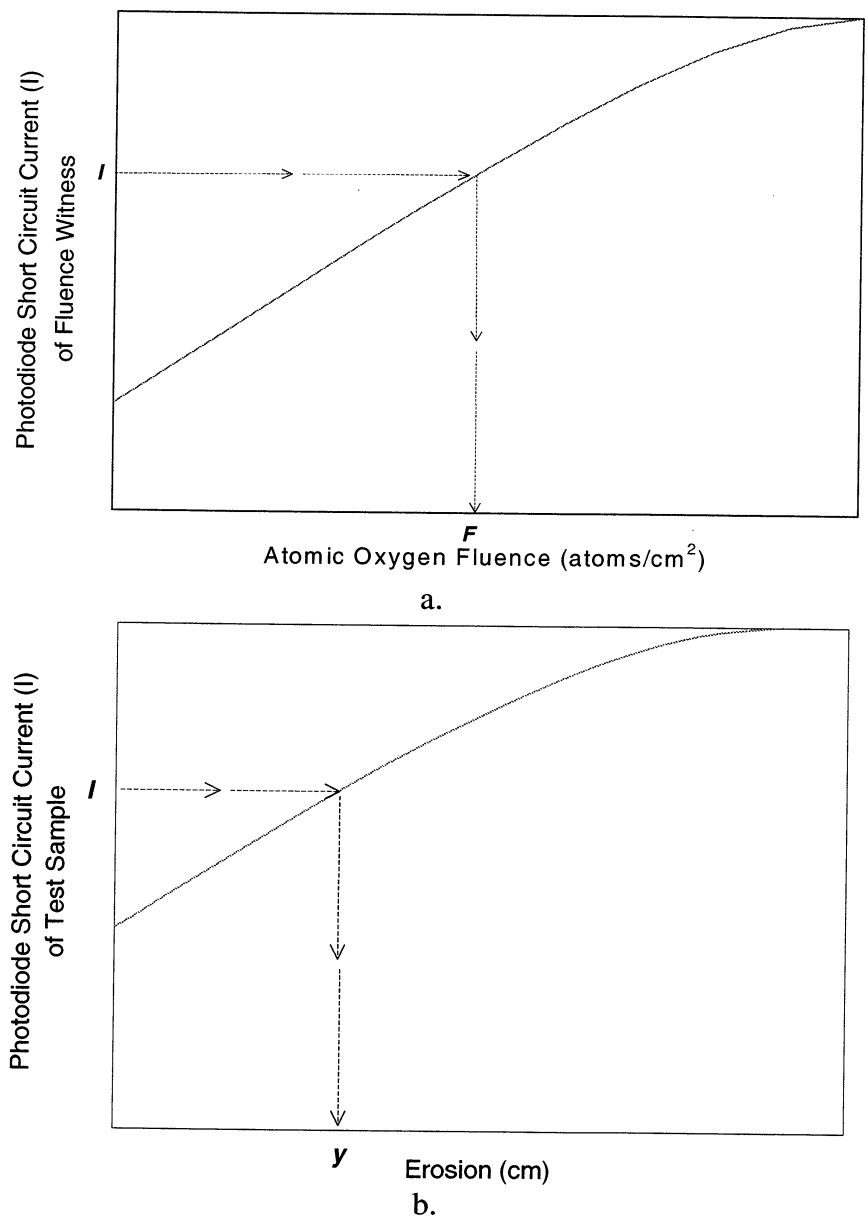


Figure 9. Method of determining atomic oxygen fluence and erosion depth based on photodiode short circuit current measurements: a. determination of atomic oxygen fluence, and b. determination of erosion depth.

The advantage of this active erosion and erosion yield measurement technique is its simplicity and reliance upon well-characterized fluence witness materials. Because the technique has not yet been tested in space, some potential complicating issues that may compromise the in-space results include reliance upon ground laboratory reflectance and absorptance data to predict optical characteristics in space. This may be an issue for long duration tests of materials that change optical properties with significant ultraviolet radiation exposure in space.

4. SUMMARY AND CONCLUSIONS

This paper reviews both passive and active atomic oxygen erosion measurement techniques. Typical passive erosion measurements for atomic oxygen erosion yield determination of polymers are based on mass loss. A technique based on protecting the flight sample pre-flight with isolated intimate contact particles, combined with post-flight AFM recession depth analyses, has been discussed. Although the recession technique is dependent on both atomic oxygen fluence and protective particle thickness, for low fluence measurements this technique is more sensitive than mass loss measurements for protective particles that are less than 17 μm thick. Error analyses indicate that AFM recession measurements for 10 μm thick protective particles have approximately one half the uncertainty in the erosion yield data as compared to mass loss measurements for an atomic oxygen fluence exposure of 1×10^{19} atoms/cm^2.

Active, or dosimeter measurements of atomic oxygen fluence have been made using QCM's and through the use of electrically conductive coating sensors. But, potential problems can exist with these techniques, and have been addressed. A new technique based on optical characterization of layered polymers is proposed by Glenn and discussed in detail. This technique could be used for either atomic oxygen fluence measurement over time, or erosion yield determination of test polymers over time.

5. REFERENCES

1. Banks, B.A., Mirtich, M.J., Sovey, J.S., Nahra, H. and Rutledge, S.K., NASA CP–3109, Vol. 2, 1990, pp. 179–184.
2. *U.S. Standard Atmosphere*, 1976, U.S. Govt. Printing Office, Washington, DC, 1976, p. 30.
3. Banks, B.A., Rutledge, S.K., Auer, B.M. and DiFilippo, F., *Materials Degradation in Low Earth Orbit (LEO)*, TMS Society, 1990, pp. 15–33.
4. Gregory, J.C., Proceedings of the NASA Workshop on Atomic Oxygen Effects, Nov. 10–11, 1986, JPL 87–14, 1987, pp. 29–30.
5. Stein, B.A. and Pippin, H.G., NASA CP–3134, Part. 2, pp. 617–641.
6. Silverman, E.M., NASA CR–4661, Part 1, August 1995.
7. de Groh, K.K. and Banks, B.B., J. Spacecraft & Rockets, Vol. 31, No. 4, 1994, pp. 656–664.
8. de Groh, K.K., Dever, J.A., McCollum, T.A., Rodriguez, E., Burke, C. and Terlep J.A., Solar Engineering 1992, Vol. 2, ASME, pp. 775–782.
9. Banks, B.A., de Groh, K.K., Auer, B.M. and Gebauer, L., NASA CP–3257, 1994, pp. 143–158.
10. ASTM E 2089–00 Standard Practices for Ground Laboratory Atomic Oxygen Interaction Evaluation of Materials for Space Applications, Annual Book of ASTM Standards, 2000.

11. de Groh, K.K., Banks, B.A., Clark, G.W., Hammerstrom, A.M., Youngstrom, E.E., Kaminski, C., Fine, E.S. and Marx, L.M., "A Sensitive Technique Using Atomic Force Microscopy to Measure the Low Earth Orbit Atomic Oxygen Erosion of Polymers," NASA/TM—2001-211346, Dec. 2001.
12. Minton, T.K., JPL Publication 95–17, Version 2, 1995.
13. Howland, R. and Benatar, L., *A Practical Guide to Scanning Probe Microscopy,* Park Scientific Instruments, 1996.
14. Banks, B.A., de Groh K.K., Baney-Barton, E., Sechkar, E.A., Hunt, P., Willoughby, A., Bemer, M., Hope, S., Koo, J., Kaminski, C. and Youngstrom, E., NASA/TM—1999-209180, May 1999.
15. Scheirs, J., ed., Modern Fluoropolymers, John Wiley & Sons, NY, 1997 (Chapter 4, B.A. Banks).

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (*Leave blank*)

2. REPORT DATE
March 2002

3. REPORT TYPE AND DATES COVERED
Technical Memorandum

4. TITLE AND SUBTITLE
Techniques for Measuring Low Earth Orbital Atomic Oxygen Erosion of Polymers

5. FUNDING NUMBERS
WU–755–A4–06–00

6. AUTHOR(S)
Kim K. de Groh, Bruce A. Banks, and Rikako Demko

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
John H. Glenn Research Center at Lewis Field
Cleveland, Ohio 44135–3191

8. PERFORMING ORGANIZATION REPORT NUMBER
E–13246

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
Washington, DC 20546–0001

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
NASA TM—2002-211479

11. SUPPLEMENTARY NOTES
Prepared for the 2002 Symposium and Exhibition sponsored by the Society for the Advancement of Materials and Process Engineering, Long Beach, California, May 12–16, 2002. Kim K. de Groh and Bruce A. Banks, NASA Glenn Research Center; Rikako Demko, Cleveland State University, Cleveland, Ohio 44115. Responsible person, Kim K. de Groh, organization code 5480, 216–433–2297.

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Unclassified - Unlimited
Subject Categories: 18 and 27 Distribution: Nonstandard
Available electronically at http://gltrs.grc.nasa.gov/GLTRS
This publication is available from the NASA Center for AeroSpace Information, 301–621–0390.

12b. DISTRIBUTION CODE

13. ABSTRACT ***(Maximum 200 words)***

Polymers such as polyimide Kapton® and Teflon® FEP (fluorinated ethylene propylene) are commonly used spacecraft materials due to their desirable properties such as flexibility, low density, and in the case of FEP, a low solar absorptance and high thermal emittance. Polymers on the exterior of spacecraft in the low Earth orbit (LEO) environment are exposed to energetic atomic oxygen. Atomic oxygen reaction with polymers causes erosion, which is a threat to spacecraft durability. It is therefore important to understand the atomic oxygen erosion yield (E, the volume loss per incident oxygen atom) of polymers being considered in spacecraft design. The most common technique for determining E is through mass loss measurements. For limited duration exposure experiments, such as shuttle experiments, where the atomic oxygen fluence is often so low that mass loss measurements can not produce acceptable uncertainties, recession measurements based on atomic force microscopy analyses can be used. Equally necessary to knowing the mass loss or recession depth for determining the erosion yield of polymers is the knowledge of the atomic oxygen fluence that the polymers were exposed to in space. This paper discusses the procedures and relevant issues for mass loss and recession depth measurements for passive atomic oxygen erosion yield characterization of polymers, along with techniques for active atomic oxygen fluence and erosion characterization. One active atomic oxygen erosion technique discussed is a new technique based on optical measurements. Details including the use of both semi-transparent and opaque polymers for active erosion measurement are reviewed.

14. SUBJECT TERMS
Atomic oxygen; Low Earth orbit; Polymer; Durability; Erosion; Atomic force microscopy; Dosimeter; Flight experiment; Space environment

15. NUMBER OF PAGES
20

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	