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An Investigation of the Degradation of Fluorinated Ethylene Propylene (FEP) Copolymer Thermal Blanketing Materials Aboard LDEF and in the Laboratory

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Abstract

Samples of fluorinated ethylene propylene copolymer thermal blanketing material, recovered from the Long Duration Exposure Facility (LDEF), were investigated to determine the nature and the extent of degradation due to exposure to the low-Earth-orbit environment. Samples recovered from the ram-facing direction of LDEF, which received vacuum-ultraviolet (VUV) radiation and atomic-oxygen impingement, and samples from the trailing edge, which received almost exclusively VUV exposure, were investigated by scanning electron microscopy and atomic force microscopy. The most significant result of this investigation was found on samples that received only VUV exposure. These samples possessed a hard, embrittled surface layer that was absent from the atomic-oxygen exposed sample and from unexposed control samples. This surface layer is believed to be responsible for the "synergistic" effect between VUV and atomic oxygen. Overall, the investigation revealed dramatically different morphologies for the two samples. The sample receiving both atomic-oxygen and VUV exposure was deeply eroded and had a characteristic "rolling" surface morphology, while the sample that received only VUV exposure showed mild erosion and a surface morphology characterized by sharp high-frequency peaks. The morphologies observed in the LDEF samples, including the embrittled surface layer, were successfully duplicated in the laboratory.

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Introduction

High-velocity atomic-oxygen and vacuum-ultraviolet (VUV) radiation, acting separately or concertedly, have been found to contribute significantly to materials degradation in Earth-orbiting spacecraft.[1] Of the materials used in spacecraft construction, organic polymers used as electrical-insulation materials and as thermal control blankets show the most significant degradation due to these environmental effects.

For example, aluminized Kapton and silverized fluorinated ethylene propylene (FEP) Teflon thermal-blanketing materials, recovered from the Solar Maximum Mission satellite (Solar Max), showed significant degradation in the space environment.[2,3] The thermal blankets, which were in low Earth orbit for four years and two months, showed degradation characterized by extensive pitting and erosion with the most severe decay occurring on surfaces exposed to *both* VUV and atomic oxygen.

Dramatic materials degradation has also been observed on the recently recovered Long Duration Exposure Facility (LDEF) satellite. Consistent with what was seen on Solar Max, the Kapton and Teflon thermal-blanketing materials showed significant degradation. Like the materials recovered from Solar Max, the most severe degradation was found for the samples attached to the leading edge of the satellite, which received VUV *and* atomic-oxygen exposure; however, the trailing edge, which experienced predominantly VUV exposure, also showed significant amounts of erosion.

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[1] Teichman, L. A., Stein, B. A. ed., *NASA/SDIO Space Environmental Effects on Materials Workshop*, NASA CP-3035, National Aeronautics and Space Administration (NASA), Washington, D.C., Part 1, 1989.

[2] Santos-Mason, B. in *Proceedings of the SMRM Degradation Study Workshop*, SMRM-79-0001, Goddard Space Flight Center, Greenbelt, MD, 1985, pp. 273–286.

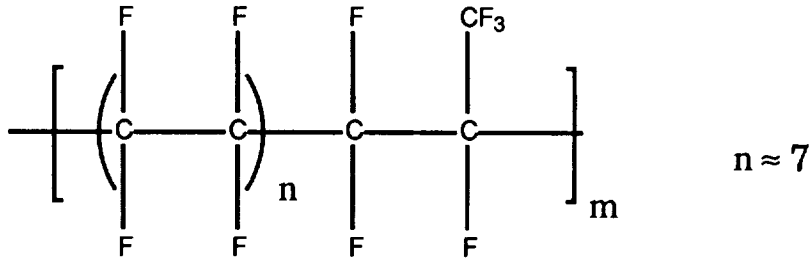
[3] Liang, R., Oda, K., Chung, S., Gupta, A., *18th Intern. SAMPE Tech. Conf.*, vol. 18, October 7–9, Society for Advanced Materials and Processing Engineering, Seattle, WA, 1986, pp. 1050–1055.

Based on these observations it is clear that atomic-oxygen and VUV radiation, acting either alone or concertedly, represent a significant source of degradation for a number of space-qualified polymers.

This publication contains an investigation of the effects of atomic-oxygen and VUV degradation on FEP copolymer, which is used in silverized Teflon thermal-blanketing materials. This study provides a detailed micrographic analysis, utilizing scanning electron microscopy (SEM) and atomic force microscopy (AFM) of the surface morphology changes in samples of FEP thermal blankets recovered from the LDEF satellite. The materials investigated include samples that received predominantly VUV exposure (trailing edge) and samples that received both atomic-oxygen *and* VUV impingement. Concomitant with this analysis is a comparison of the LDEF samples to similar samples of FEP exposed to VUV radiation and to concerted VUV and atomic-oxygen impingement in the laboratory. This comparison will suggest the degree to which degradation processes observed in space can be duplicated and quantified under ground-based testing conditions.

Material Background and Environmental Physics

FEP copolymer, used in space-qualified thermal blankets, is produced commercially (by DuPont) by the copolymerization of tetrafluoroethylene and perfluoropropylene in an approximately 6/1 mole ratio. The chemical structure is shown below:



$n \approx 7$

The polymer is insoluble and relatively amorphous (50–60% crystallinity) with a glass transition temperature (T_g) of 260°–280°C.

Thermal blankets in spacecraft applications are a laminate of 5-mil FEP with a reflective silver coating (~1000–2000 Å), which is vapor deposited on one surface. The layer of silver is subsequently coated with a layer of black paint (Chemglaze Z306). The FEP acts as a thermal (infrared) emitter, as well as a transparent "window," to protect the reflective properties of the silver and, as a consequence of this, receives direct exposure to the space environment. A scanning electron micrograph of a control sample of FEP thermal blanket (Figure 1) reveals this laminate structure.

The material, viewed at the edge in Figure 1 and showing some delamination at the corner, has a thickness of approximately 170 μm (as measured in the SEM) with 40 μm of black paint constituting the bottom layer and 120 μm of FEP constituting the top. The silver coating can be seen in Figure 1 as particles on the underside of the peeled away FEP layer.



Figure 1. SEM of a control sample of an FEP thermal blanket, viewed at the edge (at a magnification of 400), showing the laminate structure of the bulk material.

Photochemical processes leading to the degradation of FEP are initiated by the optical absorption of radiation by the polymer. The degree of degradation induced by a specific wavelength of light is dependent on, among other things, the absorption of light by the polymer at that frequency.[4] Optically, FEP is translucent with an optical absorption starting at about 300 nm and reaching 0% transmittance at ~260 nm. While a complete absorption spectrum of FEP in the VUV is unobtainable due to the sample thickness (the insolubility of the polymer prohibits the spin coating of thin samples), the absorption spectrum of polytetrafluoroethylene (PTFE) has been reported and is expected to be very similar.[5] The spectrum of PTFE shows a strong absorption at 161 nm (7.7 eV) (an absolute absorbance scale was not reported) with a weak tail extending to ~443 nm (5.5 eV) on the low-energy side, with a continuously increasing absorption at energies higher than 161 nm. Photoinduced degradation can occur only at wavelengths where there is significant overlap between the solar spectrum and the absorption of the material. In the VUV region of the solar spectrum, 100–180 nm, the major (~80%) component of the irradiance is due to the Lyman-α line of atomic hydrogen that occurs at 121 nm and has an irradiance of 0.5 $\mu W/cm^2$.[6] This region significantly overlaps the high-energy absorption continuum of the fluorocarbon and should contribute significantly to any photodegradation processes. At wavelengths higher than 150 nm, which overlap the intense 161-nm absorption of PTFE (a transition that will also be present in the FEP) as well as the low-energy absorbing tail of the polymer itself, the solar flux increases dramatically with the greatest flux occurring in the 200- to 300-nm region of the near ultraviolet (UV).

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[4] Turro, N. J., *Molecular Photochemistry*, Benjamin/Cummings Publishing, Menlo Park, CA, 1979.

[5] Seki, K., Tanaka, H., Ohta, T., Aoki, Y., Imamura, A., Fujimoto, H., Yamamoto, H., Inokuchi, H., *Physica Scripta 1990*, vol. 41, pp. 167–171.

[6] Heath, D., Thekaekara, M., White, O. R., ed., *The Solar Output and Its Variation*, Colorado Associated University Press, Boulder, CO, 1977, pp. 193–212.

Atomic oxygen is present in low Earth orbit (LEO) due to the UV photolysis of molecular oxygen:

$$O_2 + h\upsilon \rightarrow 2O$$

Atomic oxygen is a highly oxidizing, corrosive species that, at low Earth orbital velocity, has a translational energy of 5 eV. These high-energy atoms can, in and of themselves, erode materials. There is considerable evidence both from recovered space-exposed materials and laboratory simulations that a "synergistic" effect takes place between VUV radiation and atomic oxygen that results in a much greater rate of erosion than either effect taken separately.[7,8]

Material Exposed to the Space Environment: Samples Recovered from LDEF

LDEF was built to provide a stable platform for the exposure of space-related materials directly to the space environment. It was intended to provide low-cost, easy access to the LEO environment. A detailed description of LDEF is given elsewhere.[9] Briefly, the satellite is a 12-sided cylindrical structure approximately 30 feet long and 14 feet in diameter and was deployed in a three-axis gravity gradient, stabilized configuration with Earth-facing and space-facing ends, with row 9 pointing in the orbital (ram-pointing) direction. It was populated with 57 experiments involving over 200 investigators from nine countries and was deployed by the Space Shuttle Challenger (41-C) in April of 1984.

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[7] Koontz, S., Leger, L., Albym, K., Cross, J., *J. Spacecraft 1990*, vol. 27, pp. 346–348.

[8] Gjerde, H. B., Chun, T. R., Low, S. J., *18th Intern. SAMPE Tech. Conf.*, vol. 18, October 7–9, Society for Advanced Materials and Processing Engineering, Seattle, WA, 1986, pp. 1086.

[9] Clark, L. G., Kinard, W. H., Carter, D. L., Jones, J. L., *Long Duration Exposure Facility (LDEF). Mission 1 Experiments*, NASA-SP-473, NTIS order number: N84-24632/1, NASA, Washington, D.C., 1983.

The LDEF satellite was successfully retrieved and returned to Earth by the Space Shuttle Columbia (STS-32) in mid-January of 1990 after over 5-1/2 years of exposure to the LEO environment. The extended exposure of LDEF to this environment resulted in the satellite becoming a unique source of information regarding long-term space environmental effects on a broad range of materials, electrical power and data systems, and mechanical systems and structures.

The LEO environment caused obvious damage to many polymeric materials aboard the satellite, ranging from mild discoloration to complete loss (erosion) of films. Degradation of silverized FEP Teflon thermal blankets on the ram-facing surfaces of the satellite was quite apparent by the development of a noticeable hazy appearance in the previously highly specular films. The silverized FEP blankets on the trailing faces generally appeared unaffected although careful inspection revealed local regions of cloudiness in the material.

We analyzed samples of silverized FEP Teflon thermal blankets recovered from two specific areas of the LDEF satellite.[10] A sample was taken from row 2 on the trailing edge (P0004) of the satellite, which received predominantly VUV exposure (9346 equivalent solar hours) with very little atomic-oxygen fluence (4.7×10^9 atoms/cm^2), and a sample was taken from row 8 (a position ~40° off ram), which received both VUV (9346 equivalent solar hours) and significant atomic-oxygen fluence (6.4×10^{21} atoms/cm^2). Both of these samples received approximately the same equivalent solar hours of exposure, which, for the Lyman-α line (121 nm), amounts to approximately 17 J/cm^2 total radiant energy deposited.

SEM and AFM were used to interrogate the surface of the FEP thermal-blanketing samples recovered from LDEF. SEM provides a general qualitative overview of the surface morphology and a quantitative measure of the dimensions of the larger surface features (0.1 to 100 μm

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[10] Samples of FEP thermal blanket material recovered from LDEF were supplied by the LDEF project office.

range) while AFM operates on a smaller scale (~1 Å to 5 μm range), and yields a quantitative topological measurement of the roughness of the surface. These micrographic techniques are complementary and yield a relatively complete picture of the surface morphology.

SEM and AFM images of the FEP surface of the control sample are shown in Figures 2 and 3, respectively. The SEM image at 4000 times magnification shows a surface that is, on the whole, relatively smooth with some adhering dust particles and scratch marks due to handling and processing. Similarly, the AFM image shows a relatively smooth surface having a standard deviation of 9 nm from the average height of the sample (the standard deviation from the average height is a measure of surface roughness).

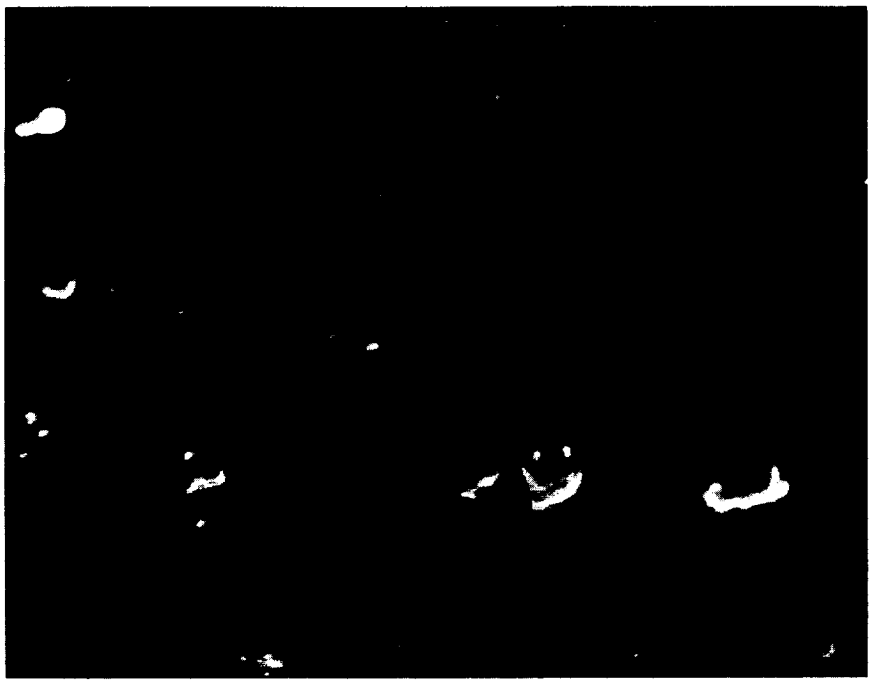


Figure 2. SEM of the surface of a control sample of FEP thermal blanket material (at a magnification of 2000).

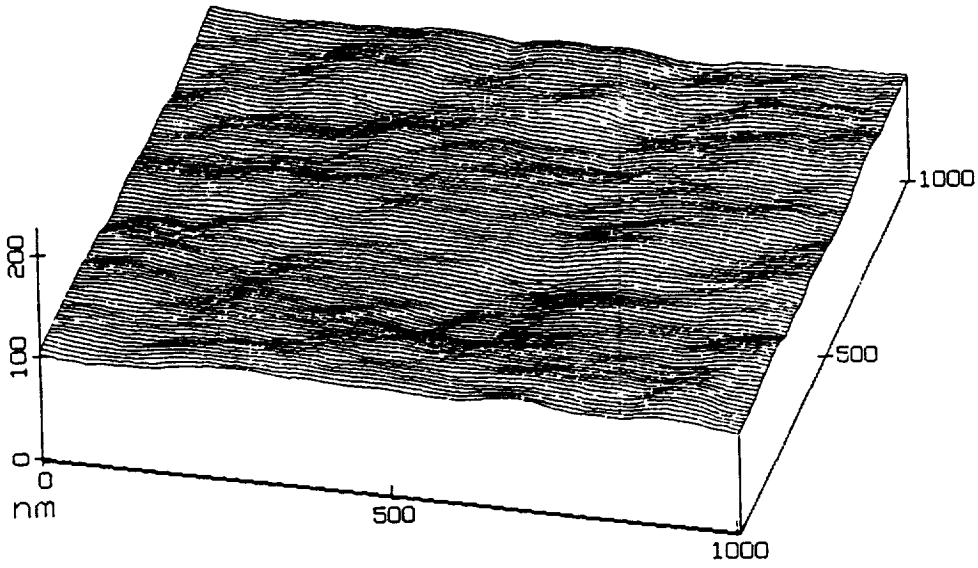


Figure 3. AFM of the surface of a control sample of FEP thermal blanket material.

In contrast, the sample that experienced atomic-oxygen *and* VUV exposure in the space environment is highly eroded. The SEM (Figure 4) shows dramatic roughening with sharp peaks angled in the direction of the atomic-oxygen flow.

The leading edge sample suffers an erosion of approximately 14 μm (0.5 mil) relative to the control sample. An atomic force micrograph (Figure 5) covering a 6 × 6 μm area of the same sample reveals that the peaks have a "rolling" quality with a very high degree of roughness as measured by the standard deviation of the peaks from the average height (451 nm as compared to 9 nm for the reference).

The observed morphological differences suggest that there is significant erosion of FEP from atomic-oxygen and VUV exposure in space (a fact already well established) and that the action of the oxygen flux is highly directional in its erosion of channels in the surface of the material. These data support recent ground-based testing results from the NASA

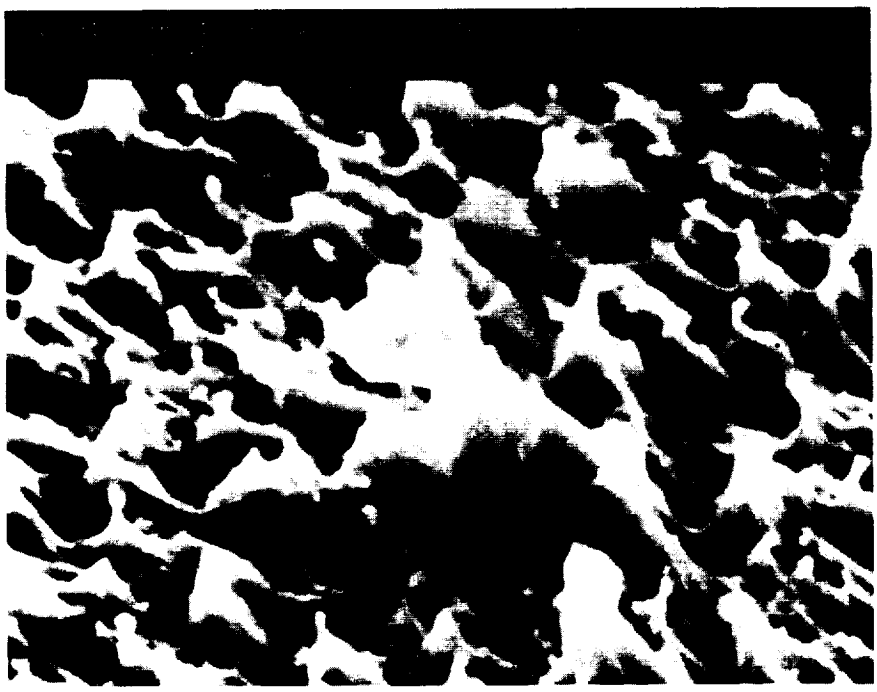


Figure 4. SEM of the surface of an FEP thermal control blanket recovered from the leading edge of LDEF (at a magnification of 10,000).

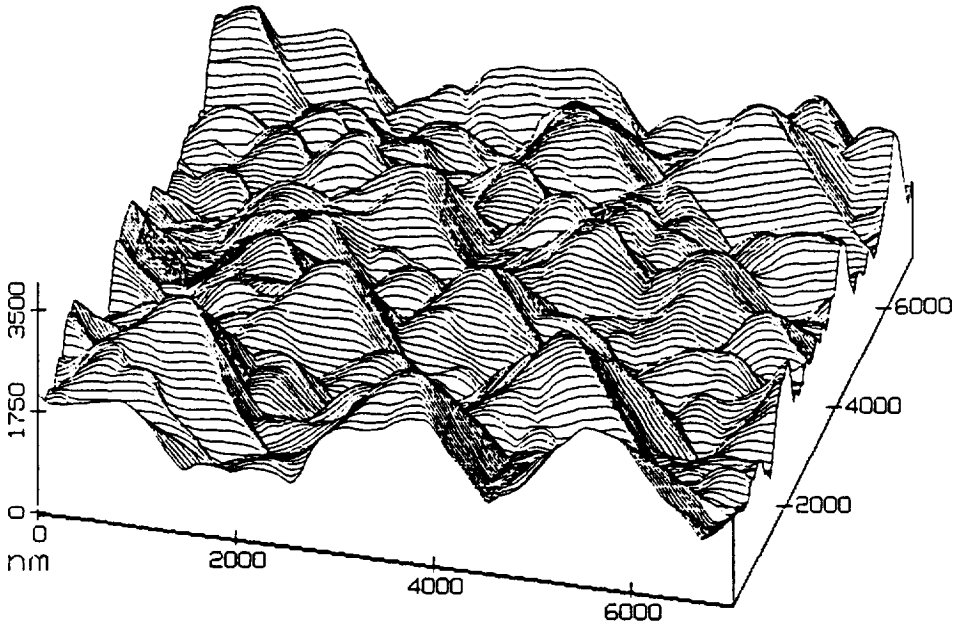


Figure 5. AFM of the surface of an FEP thermal control blanket recovered from the leading edge of LDEF.

Lewis Reseach Center that showed a directional dependence of atomic-oxygen erosion.

The LDEF samples that received predominantly VUV exposure (trailing edge) show a surface morphology that contrasts sharply with the morphology of the atomic-oxygen exposed sample. The SEM of the surface of this sample (Figure 6) shows some crazing and roughening of the surface; however, it is the AFM image (Figure 7) that shows the morphology in detail.

The VUV exposed surface shows a pattern of very sharp "jagged" peaks that are *superimposed* over a "rolling" background that is similar to the atomic-oxygen exposed sample. The peaks are very high frequency (a large number of peaks per unit area) with a moderate amount of roughness (standard deviation = 31.6 nm). This morphology suggests that VUV acting alone can erode FEP and that, due to the unique characteristics of the erosion morphology, its erosion mechanism is much different from that of simultaneous VUV and atomic-oxygen exposure.



Figure 6. SEM of the surface of an FEP thermal control blanket recovered from the trailing edge of LDEF (at a magnification of 2000).

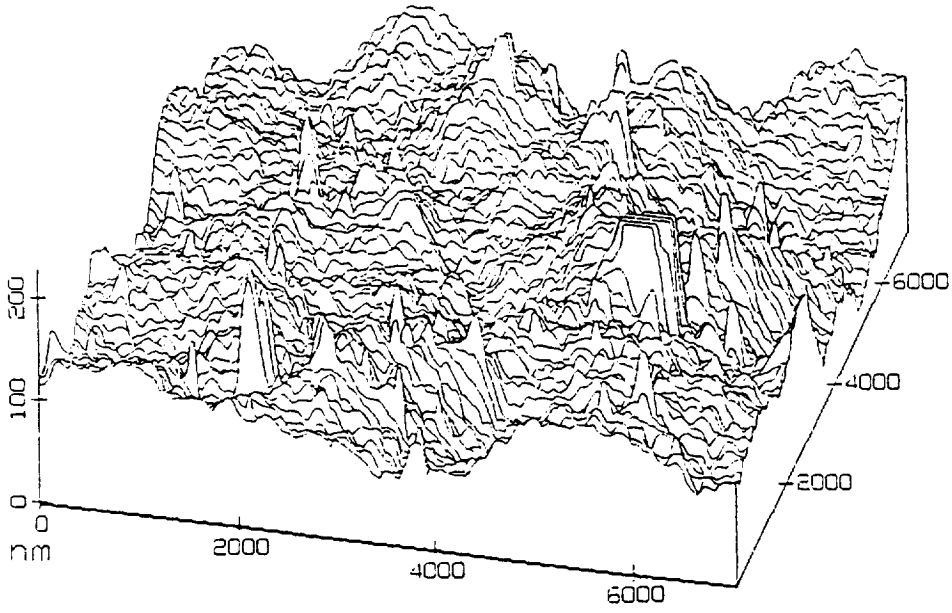


Figure 7. AFM of the surface of an FEP thermal control blanket recovered from the trailing edge of LDEF.

The most interesting aspect of the VUV-eroded material can be seen in the SEM in Figure 8. This micrograph shows the edge of the material where a cracked and hard surface layer covering the bulk material is apparent. A higher magnification image (Figure 9) shows this surface layer clearly and permits the thickness of the layer to be measured (1.14 μm).

This hard layer has no counterpart in either the reference sample (Figure 1) or the atomic-oxygen exposed sample and, therefore, must be formed from exposure to VUV radiation. It is likely that this hard layer is formed from the photochemical cleavage of the polymer chains at the surface. Recent electron spin resonance (ESR) studies have shown that VUV light induces chain scission in the polymer-yielding radical species. This chain cleavage process results in the erosion of the surface by the ejection of small molecules with concomitant cross-linking of the remaining polymer, resulting in the formation of a hard brittle layer.[10] This surface layer is absent in the atomic-oxygen exposed LDEF sample, leading to the conclusion that this surface structure is responsible for the

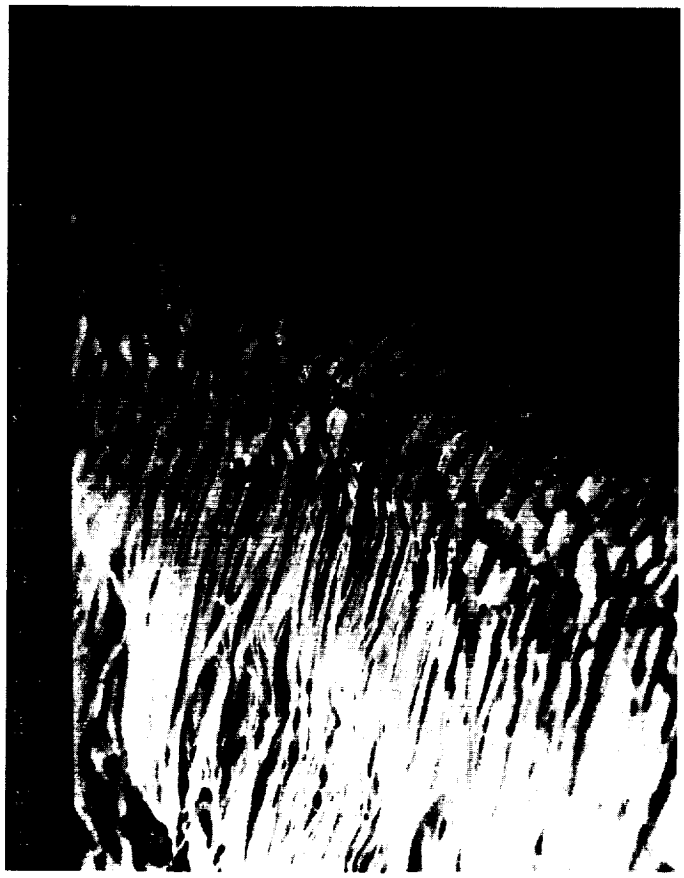


Figure 8. SEM of the edge of an FEP thermal control blanket recovered from the trailing edge of LDEF (at a magnification of 500).

"synergistic" effect observed between atomic-oxygen and VUV erosion sources. The embrittled surface layer may be more reactive towards atomic oxygen than the bulk material and, therefore, more easily eroded. In this mechanism the VUV radiation "prepares" a reactive surface on the FEP that is quickly etched away by the energetic atomic oxygen.



Figure 9. SEM of the embrittled surface layer on an FEP thermal control blanket recovered from the trailing edge of LDEF (at a magnification of 4000).

Laboratory Exposure of FEP Samples

The laboratory exposure of samples of FEP to high-energy oxygen atoms and VUV radiation is necessary in order to mimic the observed degradation seen in the space environment and to determine the relative importance of various environmental effects on the overall process. A deuterium-lamp radiation source (124 nm) was used to generate continuous fluxes of VUV radiation at frequencies similar to those experienced in space (Lyman-α, 121 nm). A molecular beam apparatus, which is capable of generating high-velocity oxygen atoms (5 eV) with simultaneous VUV exposure, was used to simulate the aspect of low Earth orbit. Details of this apparatus are given in footnote 11.

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11 Brinza, D., Coulter, D., Chung, S., Smith, K., Moacanin, J., Liang, R., "A Facility for Studies of Atomic Oxygen Interactions with Materials," *Proc. of the 3rd Inter. SAMPE Electronics Conf.*, vol. 3, Society for Advanced Materials and Processing Engineering, Los Angeles, CA, pp. 646–652.

For the VUV exposure, samples of commercial FEP (DuPont; 2.5 cm disks, 2-mil thick) were exposed with a 25-W deuterium lamp (2.9×10^{-8} W/cm^2 radiant flux at 124 nm) at a distance of 7.6 cm under an Argon atmosphere. The surface morphology of samples exposed for 92 days receiving approximately 0.24 J of 124-nm radiation was investigated.

For the atomic-oxygen exposure, a 1×2 cm sample of 2-mil FEP was exposed to an atomic-oxygen flux of 3.5×10^{19} atoms/cm^2 at an energy of 5 eV. The sample received simultaneous exposure to VUV radiation generated by the plasma that produced the atomic oxygen; the spectrum and flux of this radiation were unquantified.

Morphological Changes of the Surface: VUV Laboratory Exposure

Atomic force micrographs of an FEP sample exposed to VUV radiation in the laboratory are shown in Figure 10. The sample shows

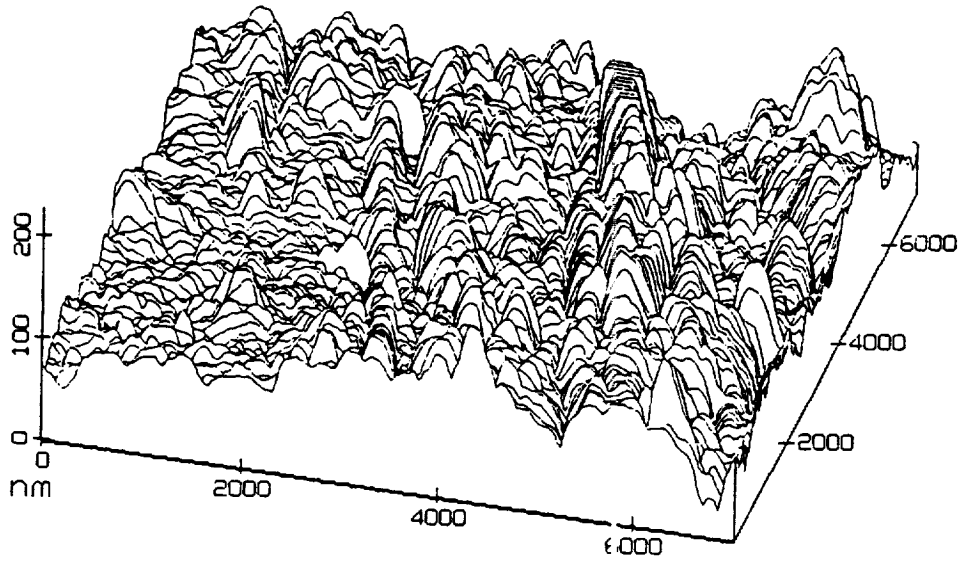


Figure 10. AFM of the surface of an FEP film exposed to VUV radiation in the laboratory.

dramatic morphological changes in the atomic force micrograph. The material shows deep erosion with separation between the peaks on the order of 100 nm and a peak-to-valley height of 250 nm. The sample has a standard deviation of 29.2 nm from the average plane and has a high frequency repeating pattern (many peaks per unit area).

The laboratory sample is remarkably similar to the LDEF sample (Figure 7) with comparable peak-to-valley heights and standard deviations (32 nm for the LDEF sample and 29 nm for the laboratory sample). The morphology is also qualitatively similar with high-frequency peaks that tend to be sharp and jagged. The laboratory sample lacks the "rolling" morphology that is seen as background in the VUV/UV-exposed LDEF sample (Figure 7) and is the dominant morphological pattern of the VUV and atomic-oxygen exposed sample (Figure 5). This is consistent with a suggestion that the "rolling" morphology is induced by atomic-oxygen exposure, while the sharp high-frequency pattern is the result of VUV/UV exposure.

More interestingly, the FEP polymer exposed to VUV light in the laboratory formed a hard surface layer analogous to that observed in the LDEF sample. This is seen on the sample edge in the SEM shown in Figure 11. The thickness of the layer is 2.8 μm, which is thicker than that seen on the LDEF sample; this may be due to the absence of any other erosive effects in the laboratory environment.

The ability to replicate most of the surface morphology of the trailing-edge exposed LDEF samples by exposure to VUV light in the laboratory implicates the action of VUV in the erosion of FEP in space and validates these laboratory techniques for the testing and study of space-related materials.



Figure 11. SEM (at a magnification of 4000) of the embrittled surface layer of an FEP film exposed to VUV radiation in the laboratory.

Morphological Changes of the Surface: Atomic-Oxygen and VUV Laboratory Exposure

The AFM showing the surface morphology of FEP simultaneously exposed to atomic-oxygen and VUV radiation sources in the molecular beam apparatus is shown in Figure 12. The surface morphology of this sample contrasts sharply to the VUV-exposed sample. Qualitatively, it has an incipient low-frequency "rolling" morphology similar to the atomic-oxygen-exposed LDEF sample. The laboratory sample received much less total exposure than the space-exposed sample, resulting in much less total erosion and a relatively small standard deviation from average, especially compared to the LDEF material (22 nm versus 452 nm). While more quantitative work is necessary to fully duplicate the atomic-oxygen and VUV synergistic effect seen in space, some of the qualitative morphological changes seen in these samples suggest that this effect is, in fact, being adequately replicated.

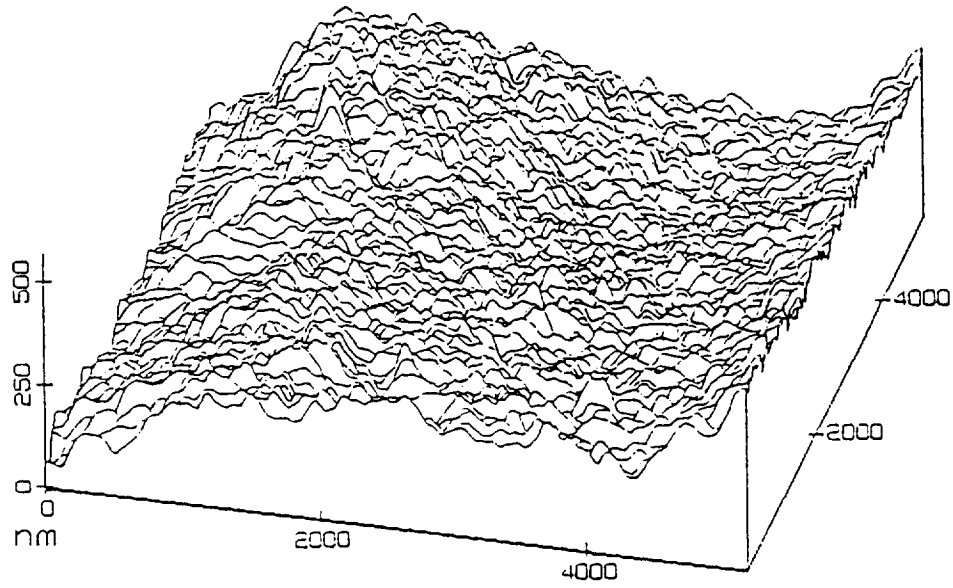


Figure 12. AFM of the surface of an FEP film exposed to VUV radiation and atomic oxygen in the laboratory.

Summary and Conclusions

Several significant conclusions can be drawn from this work. VUV radiation clearly results in the degradation of FEP copolymer used in thermal blankets for spacecraft applications. This mode of degradation results in the erosion of the polymer surface, yielding a surface morphology that is characterized by sharp high-frequency peaks. This pattern, produced in the laboratory, is almost identical to that seen in trailing-edge samples of silverized FEP recovered from LDEF. The most important observation is of a hard brittle surface layer on the FEP recovered from the trailing edge of LDEF and its subsequent formation in the laboratory with VUV exposure. The absence of this layer in the atomic-oxygen-exposed LDEF sample suggests that it may be responsible for the "synergistic" effect between VUV and atomic oxygen.

Samples of FEP recovered from the ram-facing direction of LDEF, which received both VUV and atomic-oxygen exposure, showed a very different morphology with a much greater depth of erosion and with a lower frequency of peaks per unit area as seen in the atomic force

micrograph. Qualitatively, the surface morphology is characterized by a "rolling" type of structure; a qualitative feature that has been, to some extent, duplicated under laboratory conditions.

Taken together, these results suggest that VUV radiation, separately and in concert with atomic-oxygen exposure, significantly erode FEP polymers (and, by inference, other saturated fluorocarbons). This erosion process can be satisfactorily simulated in the laboratory, a result that will greatly facilitate the testing and evaluation of new materials.

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16. Abstract

Samples of fluorinated ethylene propylene copolymer thermal blanketing material, recovered from the Long Duration Exposure Facility (LDEF), were investigated to determine the nature and the extent of degradation due to exposure to the low-Earth-orbit environment. Samples recovered from the ram-facing direction of LDEF, which received vacuum-ultraviolet (VUV) radiation and atomic-oxygen impingement, and samples from the trailing edge, which received almost exclusively VUV exposure, were investigated by scanning electron microscopy and atomic force microscopy. The most significant result of this investigation was found on samples that received only VUV exposure. These samples possessed a hard, embrittled surface layer that was absent from the atomic-oxygen exposed sample and from unexposed control samples. This surface layer is believed to be responsible for the "synergistic" effect between VUV and atomic oxygen. Overall, the investigation revealed dramatically different morphologies for the two samples. The sample receiving both atomic-oxygen and VUV exposure was deeply eroded and had a characteristic "rolling" surface morphology, while the sample that received only VUV exposure showed mild erosion and a surface morphology characterized by sharp high-frequency peaks. The morphologies observed in the LDEF samples, including the embrittled surface layer, were successfully duplicated in the laboratory.

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