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CANDIDATE ELASTOMERIC MATERIALS FOR
LONG DURATION MISSIONS. TEST SERIES
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**EFFECTS OF ATOMIC OXYGEN AND ULTRAVIOLET
RADIATION ON CANDIDATE ELASTOMERIC MATERIALS
FOR LONG-DURATION MISSIONS
TEST SERIES NO. 1**

By R.C. Linton, M.M. Finckenor, R.R. Kamenetzky, and P. Gray

Materials and Processes Laboratory
Science and Engineering Directorate

June 1993



National Aeronautics and
Space Administration

George C. Marshall Space Flight Center



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13. ABSTRACT (Maximum 200 words)

Research has been conducted at the Marshall Space Flight Center on the behavior of elastomeric materials after exposure to simulated space environment. Silicone S383 and Viton V747 samples were exposed to thermal vacuum, ultraviolet radiation, and atomic oxygen and then evaluated for changes in material properties. Characterization of the elastomeric materials included weight, hardness, optical inspection under normal and black light, spectrofluorescence, solar absorptance and emittance, Fourier transform infrared spectroscopy, and permeability. These results indicate a degree of sensitivity to exposure and provided some evidence of UV and atomic oxygen synergism.

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The inception of this work was under the stewardship of Dr. A.F. Whitaker, Project and Environmental Engineering Division Chief of the Materials and Processes Laboratory. The ideas and assistance of Bobby Cothran of the University of Alabama-Huntsville are much appreciated, as are the contributions of J. Vaughn of the Space Environment Effects Branch as co-investigator on the STS-41 Intelsat solar array experiment and for the Princeton Plasma Physics Laboratory atomic oxygen system development. The authors also wish to thank R. Thompson of Sverdrup Technology for VCMO analysis, F. Ledbetter of the Polymers and Composites Branch, and L. Norman of Sverdrup Technology for ASTM-spec permeability measurements, and D. Burch of the Engineering Physics Branch for general laboratory technical support.

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TECHNICAL MEMORANDUM

EFFECTS OF ATOMIC OXYGEN AND ULTRAVIOLET RADIATION ON CANDIDATE ELASTOMERIC MATERIALS FOR LONG-DURATION MISSIONS TEST SERIES NO. 1

INTRODUCTION

The Marshall Space Flight Center (MSFC) is conducting research into the behavior of elastomeric materials after prolonged exposure to the space environment. In the Engineering Physics Division of the Materials and Processes Laboratory, candidate materials are being exposed to thermal vacuum, ultraviolet radiation, and atomic oxygen and then evaluated for change in material properties.

To provide a focus in the scope of this study, the materials selected for testing have been limited to Viton 747 and silicone S383, two of the more viable candidate materials. In this report, preliminary results of tests providing ultraviolet radiation and atomic oxygen exposure to the selected elastomeric materials are discussed in the dual context of relevant material effects and the implications for future testing.

EXPERIMENTAL PROCEDURE

Preliminary screening of the elastomeric materials included testing for compliance with MSFC Spec. 1443. The 1-in diameter Viton V747 samples were punched out of sheet stock, gently wiped with ethanol, and baked in a vacuum oven for 24 h at 325 °F (163 °C). The same procedure was followed for the silicone S383, except the vacuum bakeout was performed at 400 °F (204 °C). Tests for volatile condensable materials with optics (VCMO) were conducted using the engineering physics VCM test stand. The VCMO tests were conducted at 167 °F (75 °C) for 24 h at a nominal pressure of 10^{-6} torr (vacuum-baked silicone S383 material failed initial VCMO testing at 125 °C). An optical mirror in the test chamber was water cooled to 50 °F (10 °C). This mirror was evaluated for reflectance in the 121.6 to 200 nm range of vacuum ultraviolet (VUV) wavelengths before and after the VCMO test. Both the Viton and silicone materials, when vacuum baked as described, passed the VCMO test with acceptable levels of total mass loss (TML) and VCM loss and with less than ± 3 -percent change in reflectance in the VUV wavelengths.¹

The next step in the space environment test program was the UV radiation exposure. Current test methodology for UV imposes caution in the allowable levels of incident flux to avoid excessive specimen heating in the bulk or at the surface. For these UV tests, the chosen acceleration factor was 2.5 "UV-suns," with one UV-sun being defined as the simulated solar UV irradiance level at 1 au in the wavelength range of 220 to 400 nm (0.0165 W/cm^2). The selected source for these tests, an Oriel model 81280 enhanced UV source with a 1-kW mercury-xenon lamp, provides a relatively "cool" beam of predominantly UV radiation, concentrated in the spectral range 220 to 400 nm.

Two Viton and two silicone 1-in diameter specimens were exposed in this UV test. These were prepared from the same sheet stock and handled in the same manner as the VCMO test samples in terms

of cleaning and thermal vacuum bakeout. These specimens were characterized prior to exposure by a variety of techniques, including mass, optical, hardness, and permeability measurements. The optical measurements were generated using a Gier-Dunkle reflectometer model DB100 for infrared thermal emittance (ϵ) and reflectometer model MS251 for solar absorptance (α). The hardness measurements were made using a shore A hardness tester. The permeability measurements were made using a configured specimen holder with a Varian model 936-SP helium leak detector.

During UV exposure, the specimens were mounted in an aluminum holder, held in place by a retainer plate which covered only a thin (0.125-in) outer annulus of the specimens. This specimen holder was attached to a water-cooled plate in the exposure chamber of the UV test system. A schematic of this UV test system is shown in figure 1. The system is oil diffusion pumped with a liquid nitrogen cold trap, and, during the UV exposure, the pressure was held at a nominal 10^{-7} torr. The specimens were exposed to the UV radiation through a 6-in diameter UV-grade quartz window. The Oriel enhanced UV source uses a dichroic mirror to maximize the throughput of the source UV radiation at the expense of the unwanted longer wavelength radiation. A typical spectrum of the Oriel source, used with a 1-kW mercury-xenon high-pressure arc lamp, is provided in figure 2, shown in comparison to the zero air-mass solar spectral irradiance in the wavelength range. For this test, the spectral irradiance from the Oriel source at the distance of the specimen plate (50 cm) was measured prior to and after material exposure with an Optronics model 730A spectroradiometer and monitored periodically during the test with hand-held Beckman HD110 radiometers. The reduced irradiance due to window transmission loss is shown in figure 3. Also recorded during the test were the specimen plate temperature and the chamber pressure.

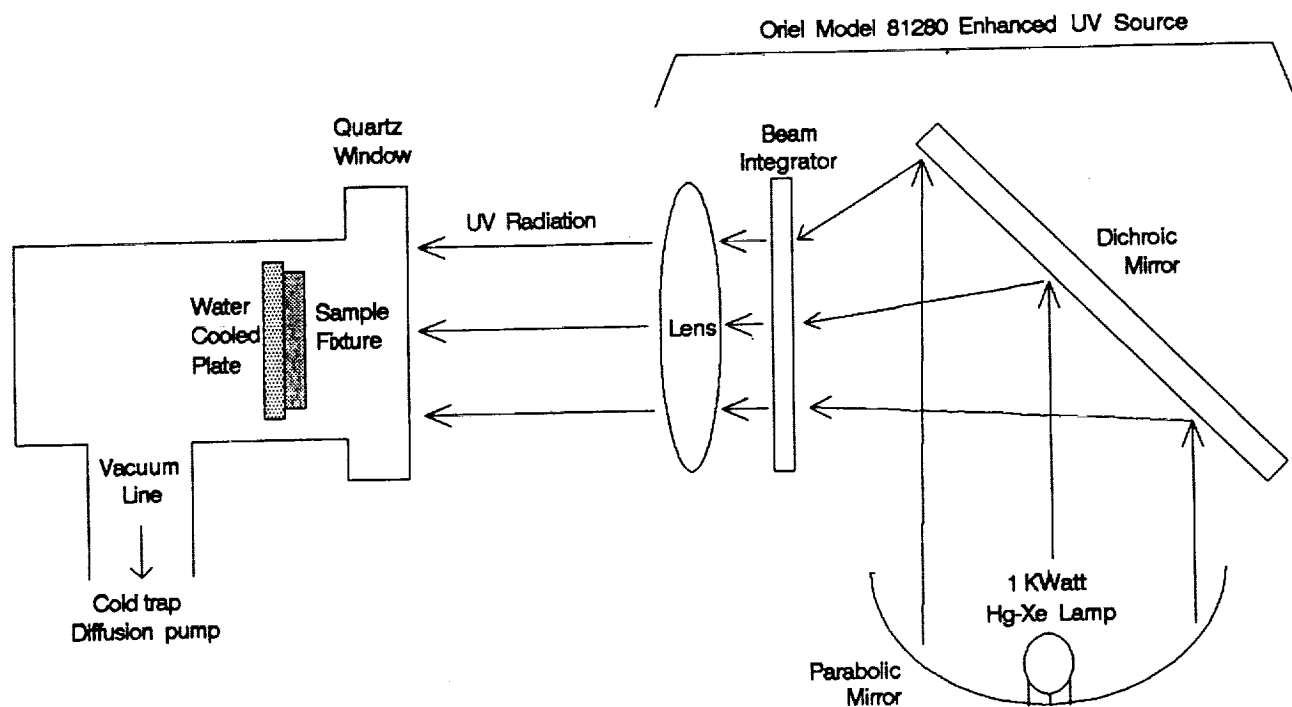


Figure 1. Ultraviolet radiation test setup.

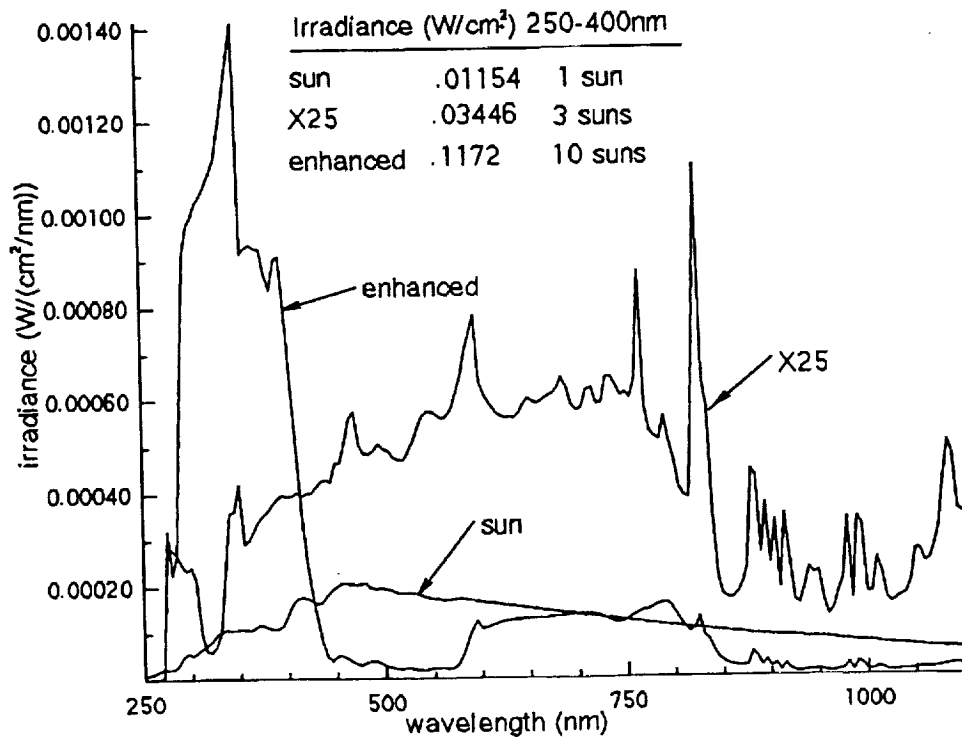


Figure 2. Irradiance comparison of UV sources.

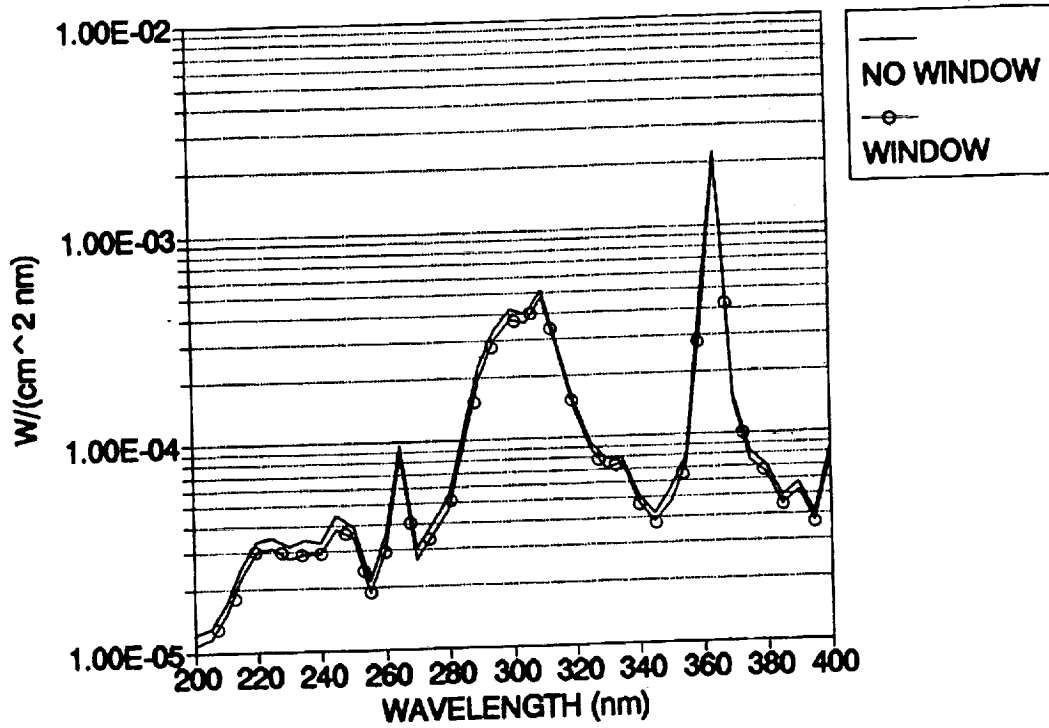


Figure 3. UV absorbance of quartz window

All four samples were exposed to 1,714.5 equivalent Sun hours of UV radiation at a sample temperature of 80 °F (27 °C). These samples were then evaluated for changes in material properties. Results will be presented later in this report.

The test plan was designed to investigate the effects of UV radiation and atomic oxygen both individually and in combination. One sample each of the UV-exposed Viton and silicone S383 and one sample each of previously unexposed Viton and silicone S383 were subsequently exposed to atomic oxygen. This exposure to atomic oxygen was performed in an asher configured to provide exposure external ("downstream") to the generated plasma. The drift tube asher, shown in figure 4, provides a neutral atomic oxygen flux of approximately 5×10^{16} atoms/cm²/s, in an environment free of radio-frequency field heating and the ionic plasma for more versatile use. In comparison, predicted levels of RAM-impinging atomic oxygen flux for Space Station *Freedom* (S.S. *Freedom*) in low-Earth orbit (LEO) are 10^{14} to 10^{15} atoms/cm²/s. The samples were exposed for 72 h in the asher, accumulating an integrated flux of 1.3×10^{22} atoms/cm², equivalent to approximately 5 years of RAM time in LEO for S.S. *Freedom* (for nominal solar activity, 1998 launch; for $+2\sigma$ solar activity, equivalence is approximately 3 years). An atomic oxygen exposure test with no prior UV exposure was later repeated on Viton and silicone samples, with nearly identical levels of atomic oxygen.

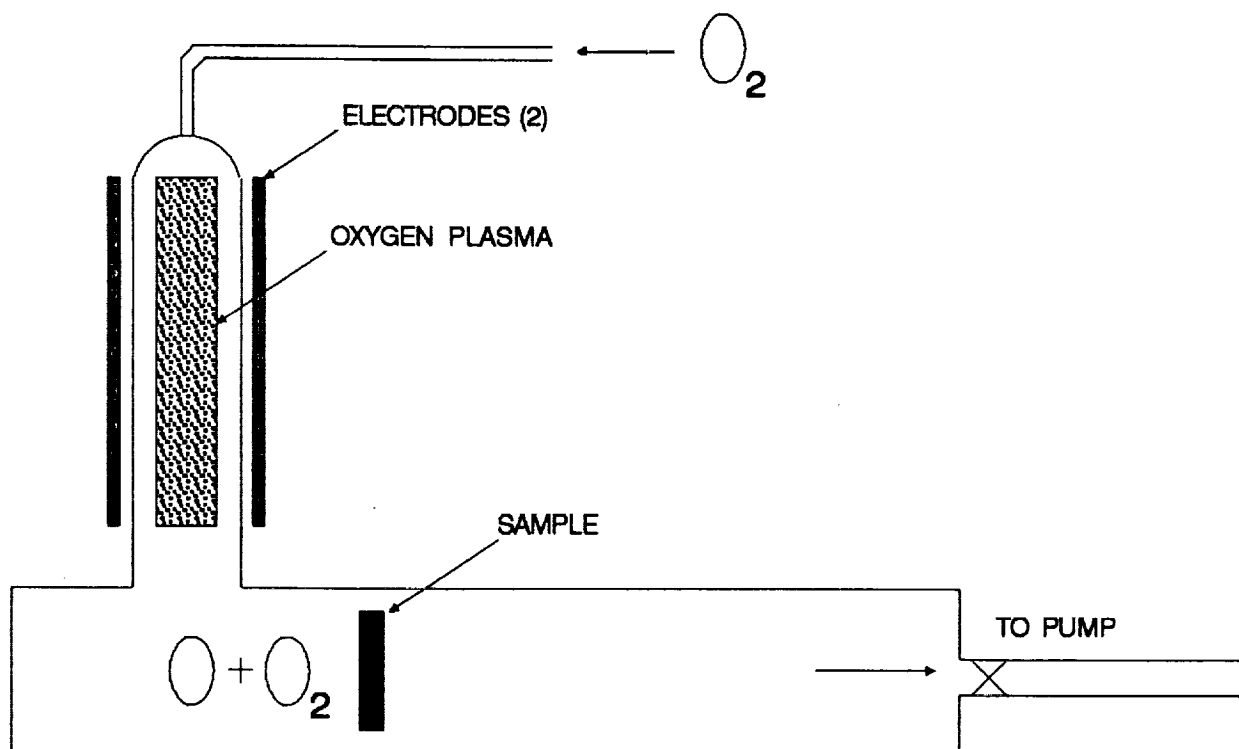


Figure 4. Atomic oxygen drift tube simulator.

EFFECTS OF SIMULATED SPACE EXPOSURE

To evaluate the effects of exposure, selected material properties were measured before and after the individual environmental tests. These characterizations included weight, hardness, optical inspection and black light-illuminated fluorescence, solar absorptance and emittance, FTIR, and permeability. Distinctive effects of exposure on the Viton and silicone samples were found as a result of both the UV radiation and the atomic oxygen exposure and the successive exposure to these environments.

Both the Viton and the silicone samples lost weight as a result of the UV exposure test. In preparing these samples for microbalance weighing prior to and following exposure, conventional techniques were employed to compare weights independent of ambient laboratory moisture gain. The samples were temporarily stored in desiccators, vacuum dried, and the "true" dry weight was extrapolated by weight gain during measurements. Following the atomic oxygen exposure, weight loss measurements indicated small levels of change for the silicone sample and more substantial changes, as expected, for the more reactive fluoroelastomer Viton.

The measured shore A hardness of the silicone S383 sample increased slightly on the side exposed to UV radiation. However, the UV exposure had no measurable effect on the hardness of the Viton samples.

Based on observations of stimulated fluorescence in similar silicone materials exposed on the Long Duration Exposure Facility (LDEF),² the samples were inspected under black light illumination prior to, during, and following the UV exposure test. Measurements of the fluorescence in the seal materials were obtained using an SLM Aminco SPF-500C dual monochromator spectrofluorometer. No stimulated fluorescence was detected with the Viton samples (fig. 5). The silicone material had weak natural red fluorescence prior to UV testing, which changed to a relatively bright yellowish flow within hours of onset of UV exposure. This stimulated fluorescence is in distinct visual contrast to the dull red, unexposed outer annulus of the exposed specimens. Figure 6 shows the natural red fluorescence peaks of the control sample compared to the stimulated yellow fluorescence of the UV exposed sample. Under conventional lighting, visual inspection revealed a slightly darkened color in the silicone specimens exposed to UV radiation. In contrast, the Viton samples exposed to UV radiation were very slightly faded.

The samples were then inspected under a Zeiss microscope. While no obvious differences were noted between exposed and unexposed regions of the Viton samples, subtle differences were detected for the exposed and unexposed regions of the silicone specimens. The visual contrast between exposed and unexposed areas of the silicone samples was more distinct under phase-contrast, and less distinct patterns of processing-imprinted striations in UV-exposed areas were noted.

Black light observations, before, during, and after the atomic oxygen exposure revealed no detectable changes in the fluorescent behavior or coloration of the samples. Changes in the visible appearance of both the Viton and silicone samples were detected. In contrast to the dull appearance of the protected outer annulus of the silicone S383 sample, the combined UV and atomic oxygen exposed area became distinctly more glossy in appearance, presumably as a result of atomic oxygen-induced "glassification." In addition, the atomic oxygen exposure induced a small pattern of surface microcracking (fig. 7). No microcracking was found in the exposed Viton sample. The Viton sample changed in appearance as a result of the atomic oxygen exposure, developing a grayish-white powdery appearance in contrast to the protected shiny black outer annulus.

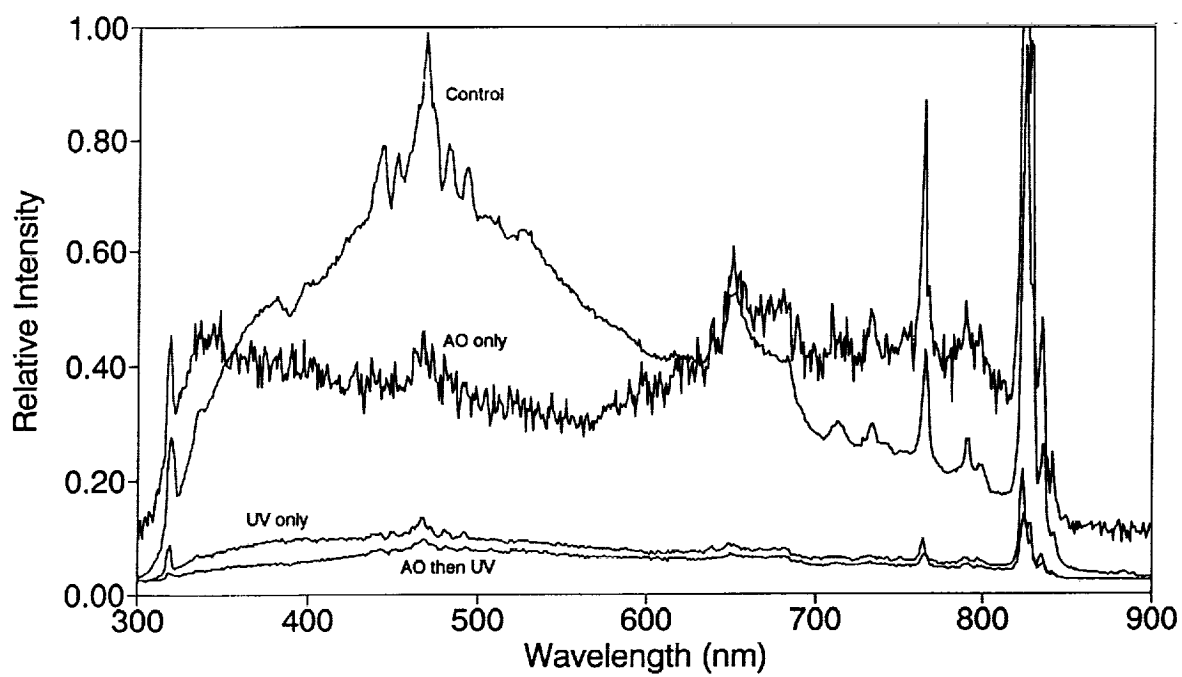


Figure 5. Fluorescence spectra, Viton V747.

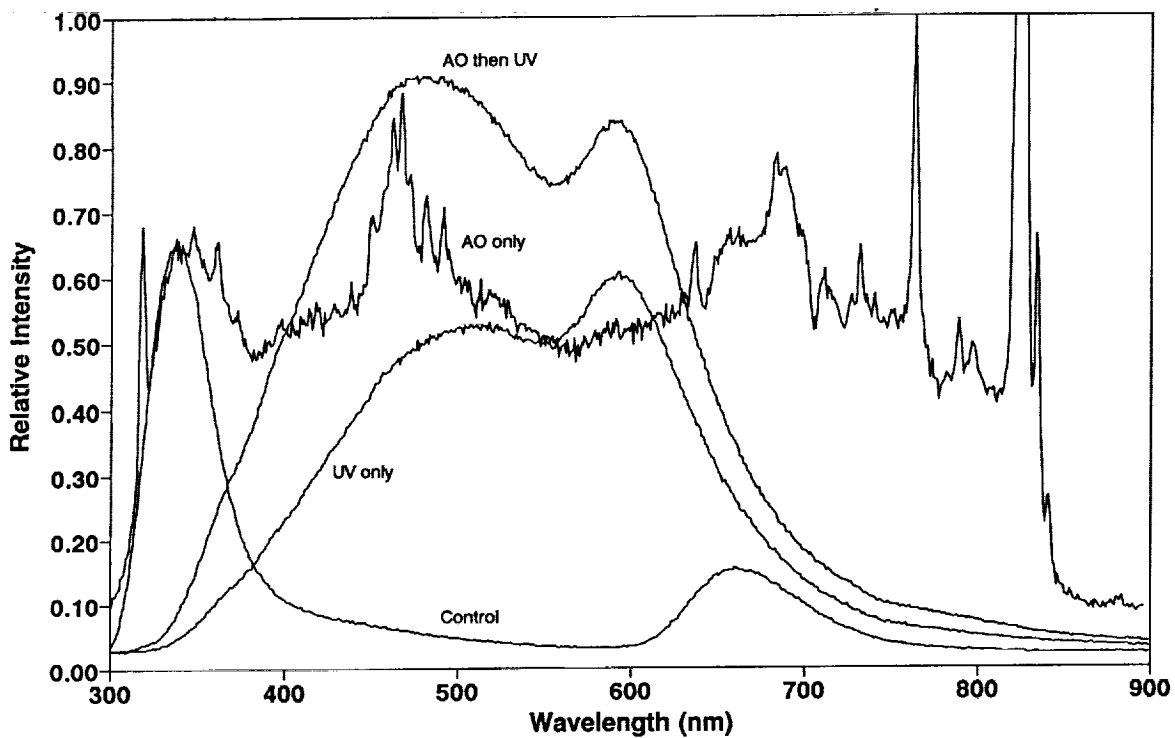


Figure 6. Fluorescence spectra, silicone S383.

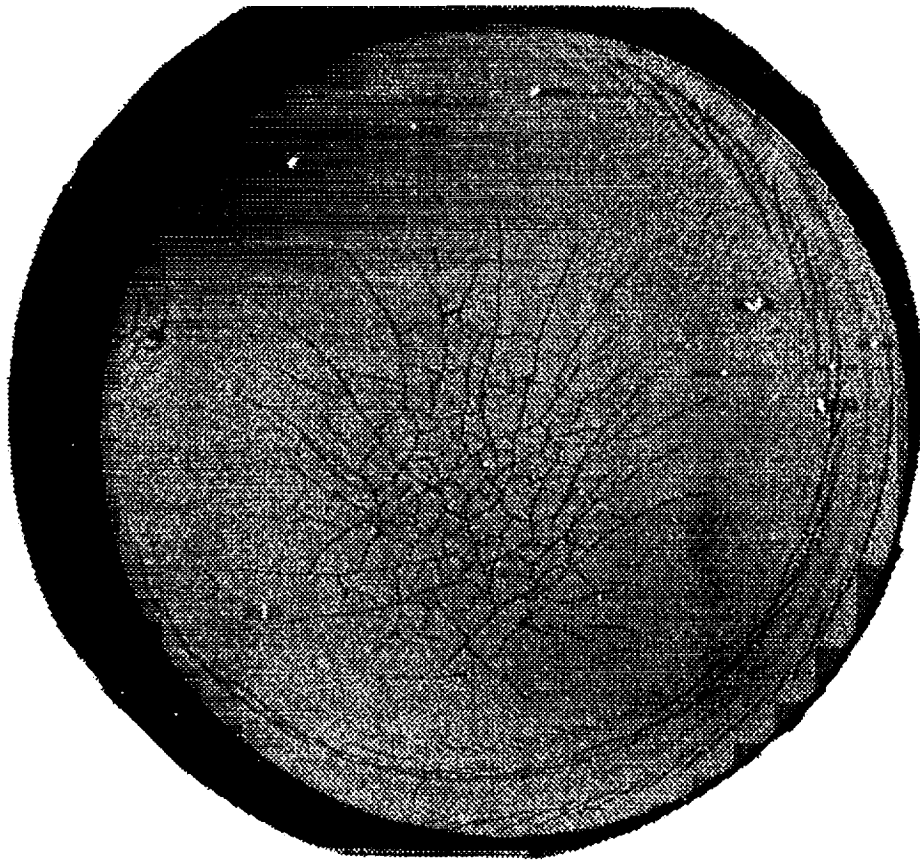


Figure 7. Microcracking in S383 after UV and atomic oxygen exposure.

Although a silicone S383 specimen, later exposed to only atomic oxygen, developed a glossy surface appearance similar to that observed for the specimen exposed to both UV and atomic oxygen, the previously discussed microcracking was not detectable as a result of this exposure. In further contrast to the UV exposure, the visual fluorescent color (dull red) of this specimen did not significantly change. The Viton 747 specimen exposed to only atomic oxygen was eroded in a manner similar to that of the UV and atomic oxygen-exposed specimen, but the rich black appearance of this exposed specimen contrasts with the grayish-white powdery appearance of the specimen exposed to both UV and atomic oxygen.

Portable solar and infrared reflectometers were used to measure the solar absorptance and infrared thermal emittance of the samples. Comparing measurements before and after the UV exposure test indicated no significant change in the absorptance and only a small change in the emittance of the samples. Some small changes in the solar absorptance and thermal emittance of the atomic oxygen-exposed samples were detected with the portable reflectometers.

A Perkin Elmer 1800 series fourier transform infrared (FTIR) spectrophotometer was also used to evaluate the degradation of the S383 elastomeric material subjected to UV radiation, atomic oxygen exposure, and the combination of UV radiation followed by atomic oxygen exposure (measurements made by D. Trinh, Analytical and Physical Chemistry Branch, Materials and Processes Laboratory). The S383 IR transmission spectra for each test case, along with the S383 control, are shown in figure 8. These spectra clearly show the susceptibility of the carbon-hydrogen bond ($2,966\text{ cm}^{-1}$ and $1,400$ to $1,500\text{ cm}^{-1}$) and the Si-CH₃ functional group ($1,257\text{ cm}^{-1}$ and 783 cm^{-1}) to UV and atomic oxygen attack, the worst case being the combination of UV radiation followed by atomic oxygen exposure. UV

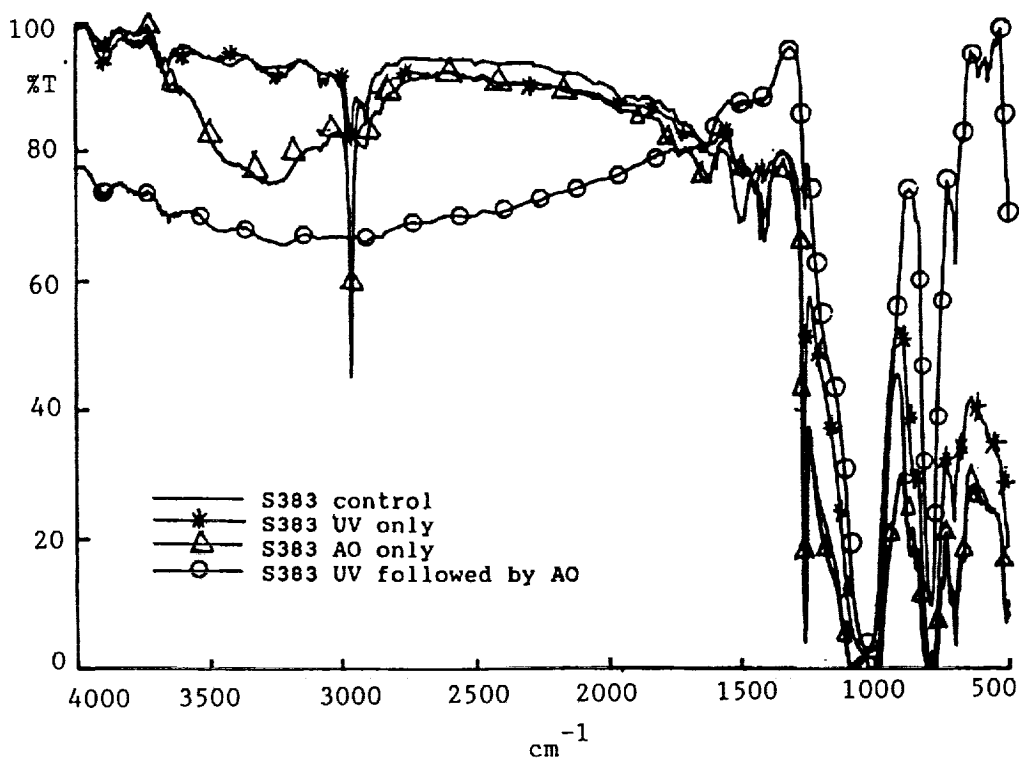


Figure 8. IR spectra of S383 control and exposed samples.

radiation alone apparently tended to produce more bond damage than did atomic oxygen exposure alone. The Si-O₂ functional group (~1,000 cm⁻¹) did not show evidence of significant damage in any test case. In addition to bond disintegration, the atomic oxygen only test case shows evidence of the early development of a carbonyl functional group (1,633 cm⁻¹) and the development of O-H bonds (3,000 to 3,500 cm⁻¹). More testing and analysis is planned to more clearly identify synergistic effects of UV and atomic oxygen exposure.

FTIR analysis was also performed on the exposed Viton elastomeric samples. Unlike the simplicity of the S383 material structure, Viton is a complex copolymer of vinylidene fluoride and hexafluoropropene which is vulcanized by the addition of a diamine derivative to ultimately produce in-chain polymeric crosslinking. Because of this structural complexity, FTIR identification of certain nitrogen functional groups would be highly speculative. However, the FTIR spectra do indicate general trends in the relative transmission, or lack thereof, of the various bonding groups as a function of exposure conditions (fig. 9). UV induced degradation of Viton appeared to be dependent on the amount of radiation the sample received. The FTIR spectrum of samples exposed to 1,715 equivalent Sun hours (ESH) indicated virtually no difference in chemical structure over the unexposed Viton transmission spectrum. Viton samples exposed to approximately 5,778 ESH, however, showed the nearly complete elimination of nitrogen bonds at or near 1,700 cm⁻¹, 2,270 cm⁻¹, and 3,700 cm⁻¹. (A subsequent exposure test will be discussed in the test series No. 2 report.) The C-F bond at approximately 1,200 cm⁻¹ and 1,400 cm⁻¹ and the =CH₂ wagging deformation at 888 cm⁻¹ appear to be unaffected by the 1,715 ESH or 5,778 ESH of UV radiation.

The FTIR spectrum of the Viton samples exposed to only atomic oxygen compared identically to the control Viton spectrum with the exception of the addition of a transmission peak around 1,520 cm⁻¹ which is most probably an N=0 peak. Viton samples exposed to 1,715 ESH followed by atomic oxygen

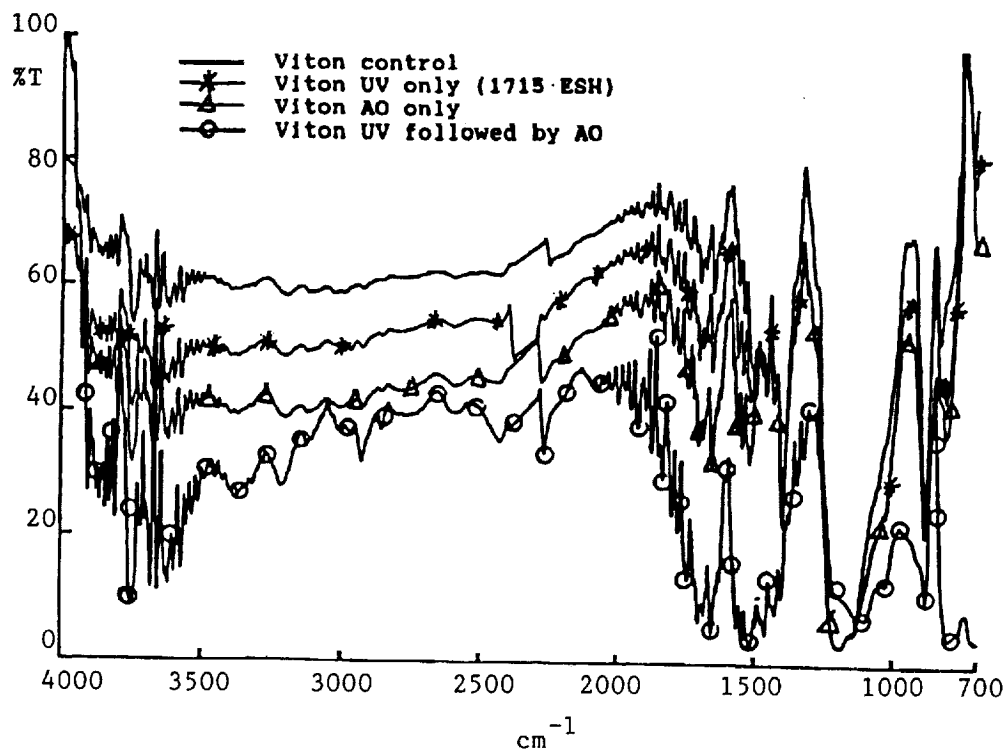


Figure 9. IR spectra of Viton control and exposed samples.

exposure also showed the additional N=0 peak and, in general, showed a large increase in transmission for all nitrogen peaks. The C-F stretch bond and the =CH₂ wagging deformation were only slightly altered. The FTIR spectrum of the Viton exposed to the 5,778 ESH followed by atomic oxygen exposure looked very similar to that of the sample exposed to 5,778 ESH alone except for the addition of two small transmission peaks at the N=0 wavelengths. More work is required to better understand any synergistic UV and atomic oxygen effects at the chemical bond level.

Procedures were developed for repeatable permeability measurements using a Varian model 936-SP helium leak detector. A perforated sample holder provided a sealing surface on the outer annulus of the sample while restricting deformation due to the operating pressure differential. These permeability measurements, providing rapid response in the laboratory, were conducted in tandem with supplementary measurements using a permeability test stand located in the Nonmetallic Materials Division of the Materials and Processes Laboratory, configured to comply with ASTM D1434-82. Permeability measurements made with the helium leak detector indicated no significant change in the Viton or silicone samples as a result of the UV exposure (figs. 10 and 11). Permeability measurements using the ASTM D1434-82 facility indicated slight changes (of about 6 percent) in the permeability of the silicone as a result of the UV exposure. Significant changes (of about 60 percent) were detected in these permeability measurements of the Viton as a result of the UV exposure (see the appendix).

During the asher exposure following the 1,715 ESH UV test, the silicone S383 and Viton samples were inadvertently exposed to atomic oxygen from both sides. Following the asher exposure test, permeability measurements using the helium leak detector indicated significant increases in the leak rate for the silicone sample and failure to seal for the Viton sample. Noting the faded appearance of the samples, the permeability measurements were repeated using Apiezon M grease on the outer annulus of the

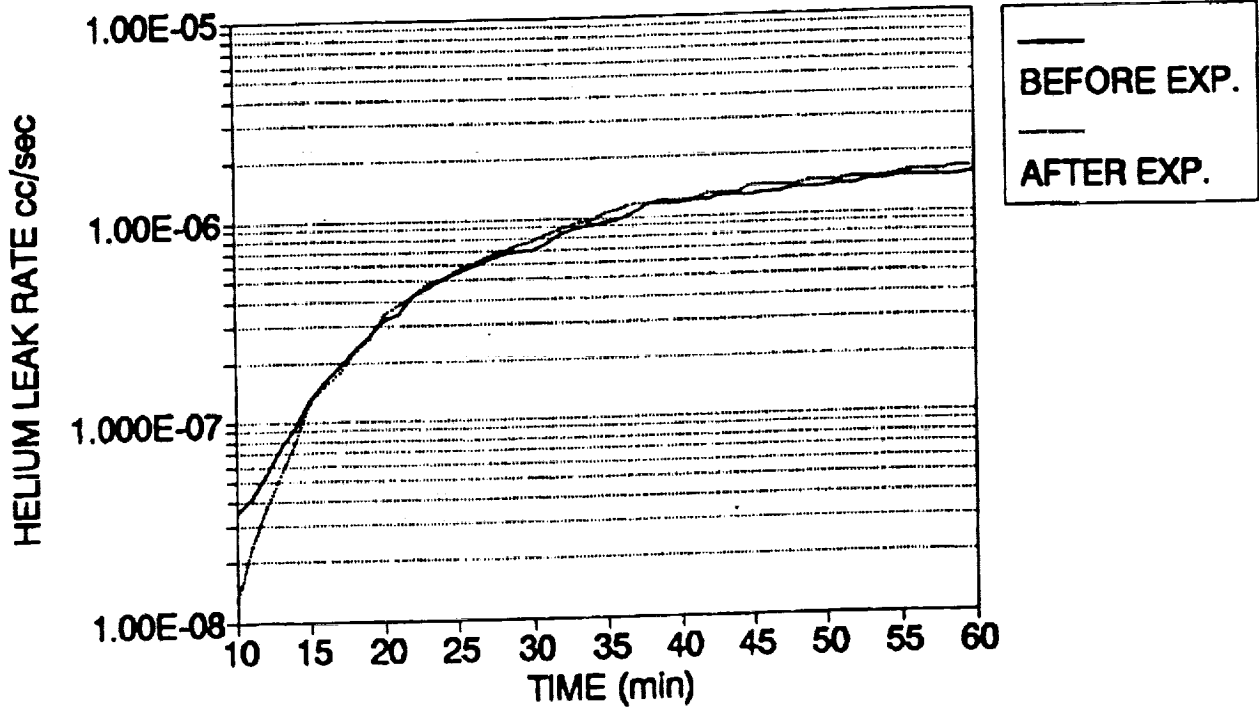


Figure 10. Helium permeability test, Viton 747 long-term UV exposure.

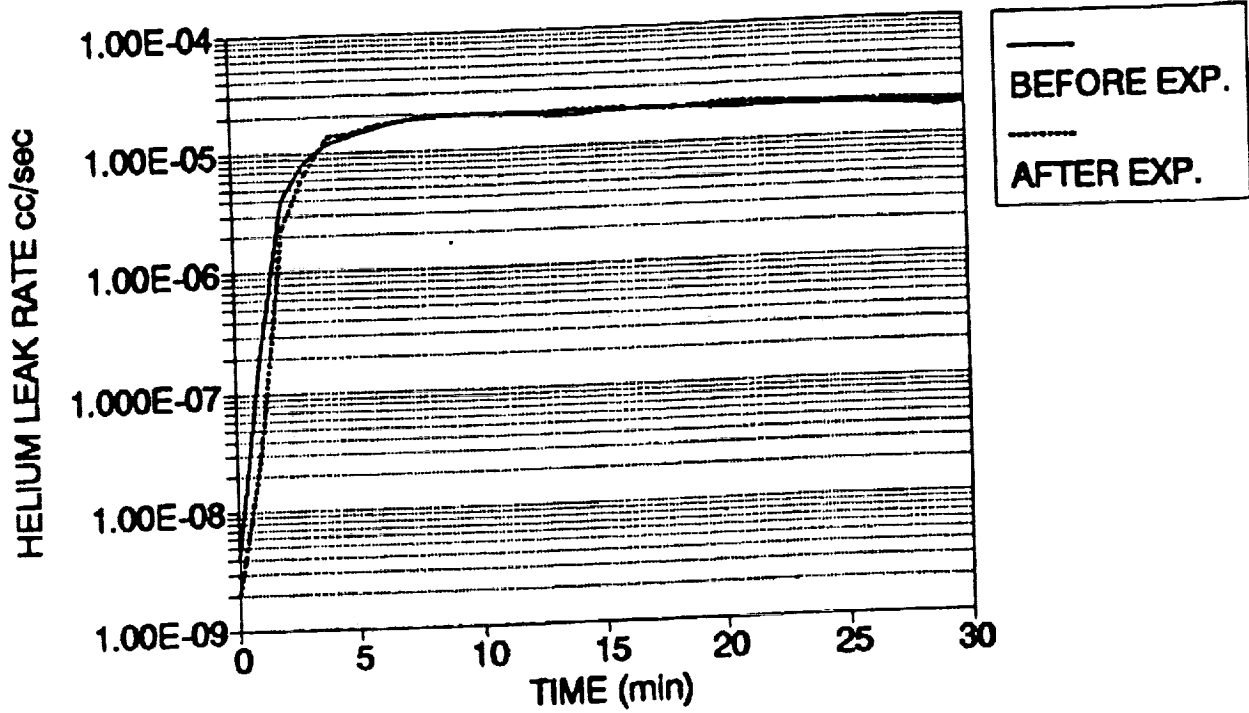


Figure 11. Helium permeability test, silicone 383 long-term UV exposure.

samples to facilitate sealing. This approach led to some improvement in the indicated leak rate of the silicone sample, though still nearly twice the level of pre-atomic oxygen exposure. The greased Viton had measurable leak rates only slightly higher than for control material. A subsequent exposure test of unexposed elastomeric materials with nearly identical atomic oxygen exposure revealed that this exposure alone leads to similar loss of sealing for Viton and silicone samples.

Silicone S383 and Viton 747 seal materials have also been exposed to atomic oxygen on a short-duration shuttle mission and in other laboratory exposure chambers. Selected silicone and Viton seal material specimens were flown on STS-41 as part of the Intelsat Solar Array Experiment. The silicone seal surface appeared glassy where exposed and gained 0.3-mg of weight due to exposure. The silicone experienced no measurable change in permeability. Analysis of the Viton samples revealed that the seal surface was visibly eroded and diffuse where exposed. The Viton lost 7-mg of weight due to exposure and also showed no measurable change in helium permeability.

These samples were sent to the University of Alabama-Huntsville (UAH) (contract NAS8-36955) for electron spectroscopy for chemical analysis (ESCA). Analysis of the silicone S383 seals indicates oxidation of the silicone material, consistent with results from LDEF analysis of RTV-511 polymeric material. Approximately 2:1 changes in the atomic ratio of oxygen (increase) and carbon (decrease) compare to only slightly higher silicone. This is consistent with the formation of Si-O₂ on the surface. Analysis of the Viton STS-41 seals shows a nearly 2:1 decrease for fluorine, a very small decrease for oxygen, and a 3:1 increase for carbon. There is, however, contamination as shown by measurable silicone for both control and flight Viton samples.

Silicone S383 and Viton 747 seal samples were also exposed in two ground-based 5-eV neutral atomic oxygen beam facilities, one at Princeton Plasma Physics Lab, Princeton, NJ, the other at Physical Sciences, Inc., Andover, MA. The ESCA results of the silicone ground-based exposures are comparable to that of STS-41. However, the ESCA results of the Viton testing from both ground-based facilities varied considerably from the flight experiment. Silicone was present in all samples, control and exposed, possibly confusing the results. UAH reported outgassing during seal analysis in the ESCA chamber, despite standard thermal vacuum preparation.

These results have indicated a degree of sensitivity to exposure for the selected elastomeric materials and provided some evidence of UV and atomic oxygen synergism. While these preliminary results also provide some evidence of significant loss of sealing capability for ungreased seals exposed directly to atomic oxygen, the results do not otherwise provide evidence of unacceptable degradation in seal performance. Additional testing with longer-duration exposure, combined ultraviolet radiation and atomic oxygen exposure, and higher specimen temperatures is currently underway to help resolve the issues of elastomeric environmental resiliency.

REFERENCES

1. Burns, H.D.: "Outgassing Tests of Viton V246-75 and S383-70 Silicone Seal Materials." MSFC EH12 memo No. 91-129, July 23, 1991.
2. Linton, R.C.: "Preliminary Results of LDEF Experiment A0034." Conference Proceedings CP-3134, First LDEF Post-Retrieval Symposium, Orlando, FL, June 1991.

Table 1. Summary of exposure effects for Viton V747.

	Control	UV Only	Atomic Oxygen Only	UV + Atomic Oxygen*
Weight Loss	—	4.08 mg	1.66 mg	13.42 mg
Emittance	0.862	0.860	0.872	0.888
Solar Absorptance	0.895	0.894	0.895	0.884
Shore A Hardness	77.82	77.64		
Normal Light Appearance	Shiny black	Shiny black	Diffuse black	Gray, powdery
Black Light Appearance	Black	Black	Black	Black

Table 2. Summary of exposure effects for silicone S383.

	Control	UV Only	Atomic Oxygen Only	UV + Atomic Oxygen*
Weight Loss	—	4.85 mg	1.29 mg	5.79 mg
Emittance	0.902	0.898	0.900	0.878
Solar Absorptance	0.672	0.675	0.660	0.676
Shore A Hardness	71.68	73.20		
Normal Light Appearance	Red	Slightly darker	Glassy red	Glassy red and cracking
Black Light Appearance	Weak red	Bright yellow	Weak red	Bright yellow

*UV exposure followed by atomic oxygen exposure.

APPENDIX

Sverdrup Technology, Inc

File No. BLN-033-91

Date: October 28, 1991

MEMORANDUM

To: Don Morris
From: Lynn Norman *Bob*
Thru: Dr. Ron Belz, Manager Materials & Processes Dept.
Subject: Permeability Testing of Space Station Seal Materials
(R. C. Linton Conversation)
Reference: Task No. 533-001

Results of the helium Permeability tests performed on Space Station Freedom candidate seal materials are listed below.

Sample Identification	Helium Permeability (E-08 cc cm/sec cm ² Bar)
Silicone S383 Baseline	158.5
Silicone S383 A0 exposure (STS-41)	160.8
Silicone S383 A0 exposure (PPPL)	159.5
Silicone S383 A0 exposure (PSI)	165.2
Silicone S383 UV radiated	168.5
Viton V747 Baseline	22.2
Viton V747 UV radiated	35.0

As you can see, the silicone samples show minor deviations from the baseline value no matter what the exposure. The Viton sample, however, showed a marked increase in permeability after being exposed to UV radiation (a 60% increase).

I have included a copy of the procedure for calculating permeability based on the volumetric flow rate of the permeating gas as well as a copy of ASTM D-1434. From what I understand, Perry is measuring the volumetric flow rate (VFR) of Helium through the

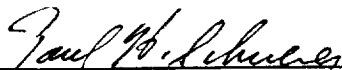
we would be able to directly compare our results. Trying to compare VFR with permeability isn't feasible since permeability takes into account the sample thickness, transmitting area, ambient temperature and pressure, and test temperature and pressure; VFR does not. I have included the ASTM test procedure because the Manometric method of measuring permeability sounds similar to the way Perry is measuring the VFR. If you have any questions, please call me or Don Morris at 4-2674.

APPROVAL

**EFFECTS OF ATOMIC OXYGEN AND ULTRAVIOLET RADIATION ON CANDIDATE
ELASTOMERIC MATERIALS FOR LONG-DURATION MISSIONS
TEST SERIES NO. 1**

By R.C. Linton, M.M. Finckenor, R.R. Kamenetzky, and P. Gray

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.



P.H. SCHUERER

Director, Materials and Processes Laboratory