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Ion Beam Sputter-Deposited Thin Film Coatings for Protection of Spacecraft Polymers in Low Earth Orbit

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NASA

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SUMMARY

Ion beam sputter-deposited thin films of Al_2O_3, SiO_2, and a codeposited mixture of predominantly SiO_2 with small amounts of a fluoropolymer were evaluated both in laboratory plasma ashing tests and in space on board Shuttle flight STS-8 for effectiveness in preventing oxidation of polyimide Kapton®. Measurements of mass loss and optical performance of coated and uncoated polyimide samples exposed to the low earth orbital environment are presented. Optical techniques were used to measure loss rates of protective thin films exposed to atomic oxygen. Results of the analysis of the space flight exposed samples indicate that thin film metal oxide coatings are very effective in protecting the polyimide. Metal oxide coatings with a small amount of fluoropolymer codeposited have the additional benefit of great flexibility.

INTRODUCTION

Anticipated space systems which must operate in the low earth orbital environment for many years, such as Space Station, will require the utilization of materials which are durable in their environment. Early Shuttle flights have demonstrated that many materials such as polyimide (Kapton®), carbon coatings, and some paints are gradually eroded and suffer changes in optical properties when exposed in low earth orbit (ref. 1). The observed rates of material loss are sufficiently high to potentially compromise the long term durability of polymers typically used in solar arrays or thermal blankets in low earth orbit (ref. 3). The postulated mechanism for the material loss is oxidation by ram impact (at approximately 4.5 eV) of the geosynchronous atomic oxygen which is the predominant environmental specie at altitudes between 180 km (97 nmi) and 650 km (351 nmi) (ref. 2).

One approach to preventing oxidation of materials in low earth orbit is to provide a protecting barrier over the oxidizable material. Such a barrier or protective coating must itself be unaffected by atomic oxygen bombardment. In addition such a coating should be flexible, thin, lightweight, adherent, UV tolerant, abrasion resistant, allow adhesive bonding, and not alter the substrate's optical properties if it is to be used for protecting polymers such as polyimide. Oxidation protective coatings consisting of predominantly metal-oxide with small amounts of fluoropolymer have been proposed as a protective yet flexible coating (ref. 4). Ion beam sputter codeposition was used to produce such metal-oxide polymer mixtures. Oxidation protective coatings must be adequately elastic to allow the typical flexure and handling required for a specific polymer application. In the case of polyimide (Kapton®) solar array blankets the hinge portion of a fold-up solar array blanket may be required to be bent to a 0.6 mm radius of curvature without coating failure. The use of mixed metal-oxide and fluoropolymer films as protective coatings may allow increased flexibility of the protective film but may require additional film thickness to assure long term (>10 yr) oxidation protection. The objective of this paper is to report the oxidation, flexibility, optical and intrinsic stress characteristics of various composition thin films that have been deposited by ion beam sputtering. Both ground laboratory and space flight test results will be presented. The ground laboratory tests utilized thin film sensing techniques and radio frequency plasma ashers to assess thin film performance. The space flight test consisted of a 41.17 hr exposure of four samples to the low earth orbital ram atomic oxygen on shuttle flight STS-8. Characterization of the samples exposed to space was performed to address two general questions. What are the changes in properties of the protected Kapton® as a result of the orbital atomic oxygen exposure and how effective is the coating in providing long term durability to the underlying Kapton®?

APPARATUS AND PROCEDURE

Protective Coating Deposition

Two ion beam sputter deposition systems were used to deposit all of the protective coatings evaluated in the ground-based and flight tests reported in this paper. Both systems utilized 8 cm beam diameter argon ion sources operated at 1000 eV with tantalum hot wire cathodes and neutralizers.

Figure 1 shows the ion beam sputtering system used to deposit protective coatings on the samples evaluated in space. These coatings consisted of thin films of SiO_2, Al_2O_3, and SiO_2 with small amounts of fluoropolymer on 0.127 mm thick Kapton®. This ion source was operated at 65 mA beam current for ion beam cleaning the substrates as well as for sputter deposition of the protective films. The sputter target, located 20 cm downstream of the ion source, was bombarded with an ion beam current density of approximately 2.8 mA/cm^2. Codeposition of SiO_2 and fluoropolymer was achieved by placing a small area sample of polytetrafluoroethylene (PTFE Teflon®) in front of the 15.24 cm diameter SiO_2 target. Deposition rates of 72 Å/min typically resulted for 100 percent SiO_2 targets.

Figure 2 shows the ion beam sputtering system used for the deposition of protective SiO_2/fluoropolymer codeposited films parametrically evaluated in ground based tests. This sputtering system was designed with a stainless steel annulus to help reduce backsputtered vacuum facility material which would deposit on the substrates being evaluated. In this system 15.24 cm diameter targets of SiO_2 and polytetrafluoroethylene (PTFE Teflon®) were placed 51.3 cm downstream of the ion source and were typically bombarded with a current density of 0.045 mA/cm^2 from a 54 mA ion beam. Deposition rates of approximately 26 Å/min resulted when using 100 percent SiO_2 targets.

Deposited film thicknesses from both sputtering systems were measured by means of a surface profiling instrument (Alpha-Step Profiler®, Tencor Instruments) which documented deposition step heights on optically flat SiO_2 substrates.

Evaluation of Protective Coating Performance

Ground-based laboratory tests. - The ability of the sputter deposited films to prevent oxidation, by atomic oxygen, of Kapton® or carbon was evaluated by means of an RF plasma asher (SPI Plasma Prep II®). Samples being evaluated were placed in the RF asher which was operated on an air discharge (at pressures of 100 to 130 μm pressure) for approximately 15 hr with only the protective coating surface of the Kapton exposed to the plasma. Weight loss measurements were then used to determine if the thin film coatings were protecting the polyimide. Because Kapton® can absorb up to 2.9 weight percent water, several days were allowed for reabsorption of moisture in a controlled laboratory environment prior to weight measurement after ashing.

An additional method to evaluate coating protection was used which relied on optical measurements during RF plasma ashing rather than weight loss of the substrates. The purpose of using this technique was to examine whether or not the protective film is preventing oxidation by using a more sensitive measure of survival, or lack of survival, of the protective film rather than waiting for bulk loss of the substrate material. To accomplish this, a diamondlike carbon film coated SiO_2 slide was used instead of Kapton® as the substrate. The diamondlike carbon film of approximately 1000 Å thickness was ion beam sputter deposited from a pyrolytic graphite target placed in the system shown in figure 1 (ref. 5). The protective coating to be evaluated was then deposited (as in fig. 2) over the diamondlike carbon film. A light beam passed through the protective coating would be partially absorbed by the diamondlike carbon film as long as the protective film prevented oxidation of the underlying carbon film. If the RF plasma ashes the protective film, then the carbon thin film ashes quickly, resulting in an increase in transmitted light. Figure 3 depicts the RF plasma ashing system with optical detection of protective film durability. An additional advantage of using an optical based durability sensing technique is that water absorption is not a concern because the substrate is not a polymer.

For actual flight utilization the film must provide protection even when it is in various degrees of mechanical strain, such as when the protected polymer is wrapped around components as in the hinge of a folded solar array. It is therefore desirable to evaluate the performance of the film with regard to the synergistic effects of atomic oxygen exposure and tensile stress. This was accomplished by wrapping coated Kapton® substrates around ceramic mandrels (0.76 mm diam) with the protective film on the external surface and placing them in the RF plasma asher. Figure 4 shows the sample configuration used for these tests. Characterization after ashing was performed by scanning electron microscopy.

Functional space applications of protective films over Kapton® would require that flexure of coated Kapton® be limited so as to not cause cracking of the protective coating, which would allow exposure and subsequent oxidation of the Kapton®. The minimum radius of curvature to which the coated Kapton® could be bent without tensile or compressive failure of the protective film was evaluated for various composition coatings by wrapping coated Kapton® around calibrated mandrels. The stressed coating was then examined for cracks by reflected light microscopy.

Intrinsic stress of protective films was determined from measurements of the dish shape distortion that occurs to an initially flat 7.62 cm diameter silicon wafer after it has been coated. The particular instrument used (Stress Gauge by Ionic Systems) measured the deflection (or bowing) of the silicon wafer by optical means using a fiber-optic proximity sensor located under the center of the silicon wafer.

Flight tests. - Samples were exposed to the ram atomic oxygen environment of 222 km (120 nmi) during Shuttle Flight 8 in three separate exposure periods on September 3, 4, and 5, 1983 for a total of 41.17 hr. This was accomplished by orbiting the earth with the shuttle bay doors open to allow the sample trays to ram with normal incidence into the environmental atmosphere. The samples consisted of one unprotected and three thin film protected Kapton® disks 2.54 cm in diameter and 0.127 mm thick which were mounted in aluminum trays which allowed space environmental exposure over a 2.06 cm diameter central portion of each sample. The unprotected sample of Kapton® was a control for the coated samples. The samples as mounted in their flight tray are shown in figure 5. This tray was located within the shuttle bay of STS-8 as shown in figure 6.

The characterization of changes in the properties of Kapton® resulting from the deposition of the protective coatings was documented by: optical microscopy; scanning electron microscopy (Amray® 1400); optical reflectance, absorptance, and transmittance (a Gier-Dunckle® integrating sphere in conjunction with a tungsten strip lamp and monochrometer); and infrared total reflectance and transmittance (a Perkin Elmer Model 1430 Infrared Sprectroscopy Data System). The characterization of the effectiveness of the applied coatings was accomplished by: optical microscopy; scanning electron microscopy; energy dispersive x-ray spectroscopy (Kevex® EDS System); mass change; and Rutherford backscattering spectrometry (for the SiO_2 and codeposited SiO_2-fluoropolymer films).

RESULTS AND DISCUSSION

Ground Based Laboratory Tests

The RF plasma asher was found to produce repeatable mass loss per unit area rates for Kapton® provided that power setting, sample spacial position, and sample orientation were held constant, and that sufficient time for water reabsorbtion was allowed. Figure 7 is a plot of the dependance of the rate of mass loss per unit area with position along the axis of the asher. The asher was used to evaluate the effectiveness of various protective films on Kapton® for selection of the thickness and composition of coatings to be tested in space on STS-8 based on mass loss per unit area rate data (ref. 4). The protective coatings selected on this basis are given in table I.

Use of the plasma asher with optical detection of the protective films typically resulted in one of the three types of plots of transmitted light (or short circuit solar cell current) versus time shown in figure 8. The decreased transmittance of light with time occurred only for pure protective films thicker than 200 Å. This optical technique was used to determine the minimum film thickness required for permanent oxidation protection as a function of SiO_2 and fluoropolymer composition (see fig. 9). The protective film was considered permanently protecting if a negligible increase in transmittance occurred after more than 12 hr of exposure to the asher plasma. This can be compared with complete ashing of a 340 Å diamondlike carbon film in 1.1 hr if no protective film is present or with complete ashing of a 100 percent fluoropolymer film of 465 Å and its underlying 340 Å diamondlike carbon film in 1.22 hr. As can be seen from figure 9 the addition of codeposited fluoropolymer necessitates thicker films for permanent protection of the diamondlike carbon. The addition of the fluoropolymer for purposes of increasing film flexibility is thus limited to approximately 15 percent fluoropolymer for lifetime reasons. The data points represent the minimum thickness films that are permanently protecting based on numerous ashing tests of films of various thicknesses for each SiO_2-fluoropolymer composition. A question that appears appropriate to ask concerning figure 9 is whether the minimum thickness requirement is the same for the protection of a Kapton® substrate as for a diamondlike carbon film substrate. This was experimentally verified for two compositions (0 and 15 percent fluoropolymer) by observing the indicated results for the curve of figure 9 when the films were deposited on Kapton®.

Because the energy of bombarding oxygen atoms in low earth orbit is 4.5 eV and the energy of the oxygen ions in the plasma asher is approximately 0.23 eV, the shape of the minimum thickness versus percent fluoropolymer curve may be slightly different for an actual in-space exposure. Ferguson (ref. 6) has found that the oxidation rate for Kapton® is energy dependant . The rate was found to be proportional to the 0.68 power of the impinging oxygen ion or atom kinetic energy. In spite of this energy dependance, the figure 9 plot would probably be only slightly different because the major constituent, SiO_2, should have a negligible ash rate since it is already in its highest oxidation state. Figure 9 may be more a measure of the ashing percolation limit of molecularly mixed SiO_2 and tetrafluoroethylene molecules than a result of the ash rate of materials. If the population of the fluoropolymer constituent is sufficiently high to allow oxidation of a line of sight path through the protective film, then the underlying carbon atoms will be oxidized. Otherwise the SiO_2 molecules will block the oxidation process. The greater the fluoropolymer content, the thicker the film must be to provide enough SiO_2 molecules to prevent oxidation pathways to develop through the film.

The addition of fluoropolymer codeposited with SiO_2 was found to increase the amount of strain the film could withstand without causing brittle fracture. Figure 10 shows plots of (a) the minimum radius of curvature to which a 1000 Å thick codeposited film on 0.127 mm thick Kapton® can be bent and (b) its estimated strain as functions of composition. The strain was computed assuming that the modulus of elasticity for each SiO_2-fluoropolymer film was the appropriate arithmetic average between SiO_2 and PTFE-Teflon® for its composition. As one can see from figures 9 and 10 the addition of 15 percent fluoropolymer to SiO_2 allows a factor of 3 gain in tolerable strain but is accompanied by an order of magnitude increase in thickness needed for permanent oxidation protection. Film thickness has no significant influence on the minimum radius of curvature because the film is essentially in pure tension during the flexure of the substrate Kapton® which is approximately 10^3 times thicker than the film as can be seen in figure 11(a). However the thicker the SiO_2 film the greater the influence the high modulus coating has on the low modulus substrate. This effect tends to reduce the actual strain applied to the film for constant radius of curvature bending. Figure 11(b), which depicts the fracture limit mean strain in the film as function of film thickness, indicates this effect. Film thicknesses of approximately 1000 Å were chosen to provide data for figure 10 because they were sufficiently thick to allow easy observation of fractures in the films.

The intrinsic stress in films resulting from the sputter codeposition of SiO_2 and fluoropolymer scission fragments from the polytetrafluoroethylene (Teflon®) target was found to be a function of both composition and film thickness as shown in figure 12. The thickness dependance shown is typical of other materials (ref. 7).

The evaluation of protective films subjected to simultaneous tensile stress and RF plasma ashing was found to show clearly the effect of oxidation of the underlying Kapton® if the film was stressed to failure causing undercutting oxidation through the open cracks. As can be seen in figure 13(a) closed fractures in an indium-tin-oxide film on Kapton® do not allow oxidation but open fractures do. Figures 13(b) and (c) show sufficient undercutting oxidation in failed films of pure SiO_2 and 14 percent fluoropolymer in SiO_2, respectively, on Kapton® to allow partial detachment of the protective films.

Flight Tests

The Kapton® samples and surface protective films tested in space on STS-8 are listed in table I. The Kapton® sample having no protective coating was the only sample to show a change in appearance as a result of the 41.17 hr orbital ram exposure. The exposed surface had a matte rather than a specularly reflecting appearance. Although, as shown in figure 14, the total reflectance and absorptance changed little, the diffuse reflectance increased greatly, thus causing the matte appearance. The transmittance for the unprotected Kapton® sample remained zero because of the opaque aluminum coating on its unexposed surface.

The optical properties of transmittance, absorptance, and total reflectance for the SiO_2, and $\geq$96 percent SiO_2 $\leq$4 percent PTFE coated samples remained unchanged as a result of the orbital ram exposure. Figure 15 compares the optical properties of the codeposited $\geq$96 percent SiO_2 $\leq$4 percent PTFE sample prior to film deposition, after deposition but prior to orbital ram exposure, and after orbital ram exposure for the wavelengths from 0.33 to 2.2 μm. Similar results were obtained for the SiO_2 coated sample and for the pre- and post-deposition Al_2O_3 coated sample. The Al_2O_3 coated sample was not optically evaluated postflight. Figure 16 compares the infrared transmittance and reflectance of Kapton® prior to and after deposition of the $\geq$96 percent SiO_2 $\leq$4 percent PTFE film for wavelengths from 2.5 to 50 μm. The infrared results were again similar for the Al_2O_3 and SiO_2 coatings. As can be seen from figures 14 and 15, there appears to be no significant alteration in the optical properties of Kapton® as a result of either depositing the thin film coating on the Kapton® or the orbital ram exposure. Based on the visible and infrared data the solar absorptance, α_S, for each of the three types of coated Kapton® was found to be identical to that of uncoated Kapton® ($\alpha_S = 0.331$) and unchanged by low earth orbital atomic oxygen ram exposure. The thermal emittance, ϵ_T, at 300 K was also found to be unchanged ($\epsilon_T = 0.80$) by either the presence of or type of protective coating. The limited size of samples available for postflight analysis precluded measurement of the infrared properties of the samples after orbital atomic oxygen exposure because portions of the samples were consumed for SEM and RBS analysis.

Figure 17 shows scanning electron micrographs to illustrate the effect of the $\geq$96 percent SiO_2 $\leq$4 percent PTFE protective coating on the durability of the Kapton® surface when exposed to atomic oxygen ram in space. The lack of noticeable damage for the >96 percent SiO_2 <4 percent PTFE coating was typical of that of the Al_2O_3 and SiO_2 coatings as well. The Al_2O_3 coated sample however did have a small defect which was noticed prior to flight testing. This defect site was a spot about 0.2 mm diameter where a particle of debris or dust prevented sputter deposition over the Kapton®. Postflight inspection of this site indicated the possibility of undercutting oxidation approximately 0.2 mm beyond the diameter of the original surface defect boundary. This may have been due to scattered atomic oxygen atoms which were able to laterally oxidize the Kapton® under the Al_2O_3 film. The surface texture present for the unprotected Kapton® after orbital ram exposure is the cause of the matte appearance and increase in diffuse reflectance resulting from atomic oxygen exposure.

Energy dispersive x-ray spectroscopy confirmed the presence of each of the protective films after orbital ram exposure.

The results of the pre- and post-flight mass determinations are given in table I. The unprotected Kapton® had high rates of mass loss in comparison to Kapton® with protective films. The relatively high mass loss rate found for the Al_2O_3 coated sample may in part be a result of the surface defect, a possible missing shard, or an anomalous water uptake before or after weight measurements. The amount of mass lost for this sample is six times the mass of the coating present. However even in optical microscopy there is vivid evidence that the coating is present, thus tending to confirm the suggestion that the measured high weight loss is an incorrect measure of the protection of the Kapton®. Results of the Rutherford backscattering analysis of unexposed and exposed areas of the SiO_2 and >96 percent SiO_2 <4 percent PTFE flight tested samples are given in table II. These data indicate that there was no loss in the thickness of these protective coatings.

CONCLUSION

Ion beam sputter deposited thin films of Al_2O_3, SiO_2, and SiO_2 - polytetrafluoroethylene molecular mixtures are effective in preventing oxidation of underlying materials such as Kapton® as demonstrated in both ground-based laboratory and flight tests. The addition of small amounts (≤15 percent by volume) of codeposited polytetrafluoroethylene to SiO_2 can be used to increase the strain that the film can withstand without fracture by a factor of 3. However the ground-based rf plasma ashing tests in conjunction with optical detection of the protective films indicate that the minimum protective film thickness required for permanent protection from oxidation is increased by an order of magnitude (from 50 to 600 Å) if one adds 15 percent polytetrafluoroethylene. The application of protective films of Al_2O_3, SiO_2, and ≥96 percent SiO_2 ≤4 percent polytetrafluoroethylene did not alter the optical properties of Kapton® over the wavelengths from 0.33 to 50 μm. In addition, no change occurred in optical properties of the protected Kapton® over the wavelengths from 0.33 to 2.2 μm. Postflight analysis indicates the three protective coatings remained intact and functional throughout the 41.17 hr orbital ram exposure to environmental atomic oxygen.

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TABLE I. - EROSION RATES OF UNPROTECTED AND PROTECTED KAPTON® SAMPLES EXPOSED TO LOW EARTH ORBITAL ENVIRONMENT

Protective coating on Kapton®	Coating thickness, Å	Measured mass loss, µg	Based on all characterization information	
			Conclusion	Estimated[a] erosion rate, cm^3/atom
None (unprotected)	0	5020	Kapton® eroded and textured	3.0×10^{-24}
Al_2O_3	700	567	Coating protects	$<2.0\times10^{-26}$
SiO_2	650	5.9	Coating protects	$\leq 8\times10^{-28}$
$\geq$96% SiO_2 $\leq$4% PTFE[b]	650	10.3	Coating protects	$\leq 8\times10^{-28}$

[a]Based on an estimated atomic oxygen fluence of 3.5×10^{20}.
[b]Polytetrafluoroethylene.

TABLE II. - SURFACE DENSITIES OF OXYGEN AND METAL ATOMS ON UNEXPOSED AND SPACE EXPOSED SAMPLES ANALYSED BY RUTHERFORD BACKSCATTERING

Protective coating on Kapton®	Oxygen atoms/cm^2		Metal atoms/cm^2	
	Unexposed area	Exposed area	Unexposed area	Exposed area
SiO_2	2.42×10^{17}	2.53×10^{17}	1.22×10^{17}	1.27×10^{17}
$\geq$96% SiO_2 $\leq$ 4% PTFE	3.21×10^{17}	3.17×10^{17}	1.20×10^{17}	1.25×10^{17}

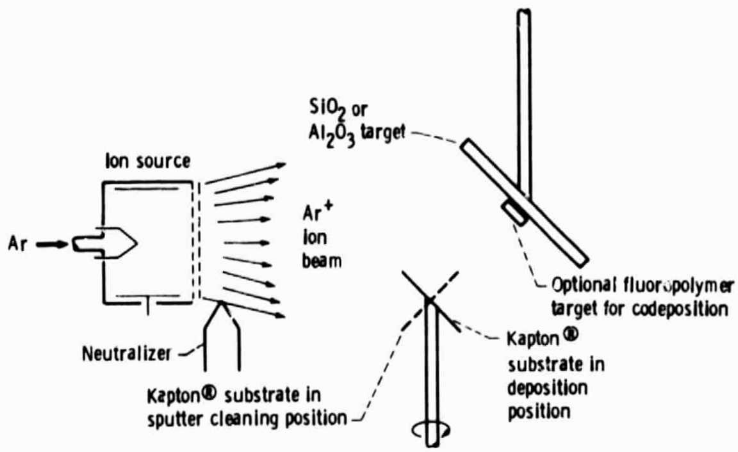

Figure 1. - Ion beam sputter cleaning and deposition system used for preparation of samples evaluated in-space.

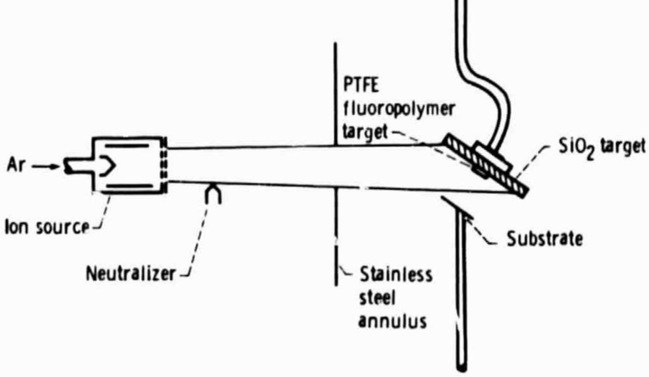

Figure 2. - Ion beam sputtering system for ground-based laboratory evaluation of protective films.

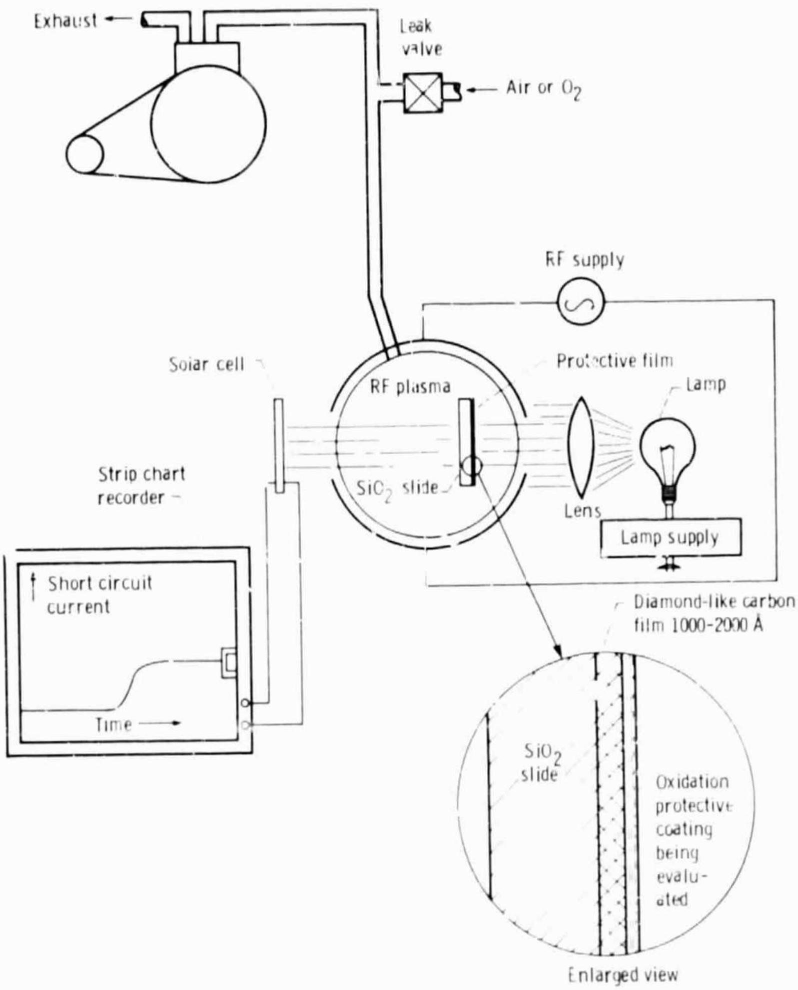

Figure 3. - RF plasma ashing system with optical detection of protective film durability.

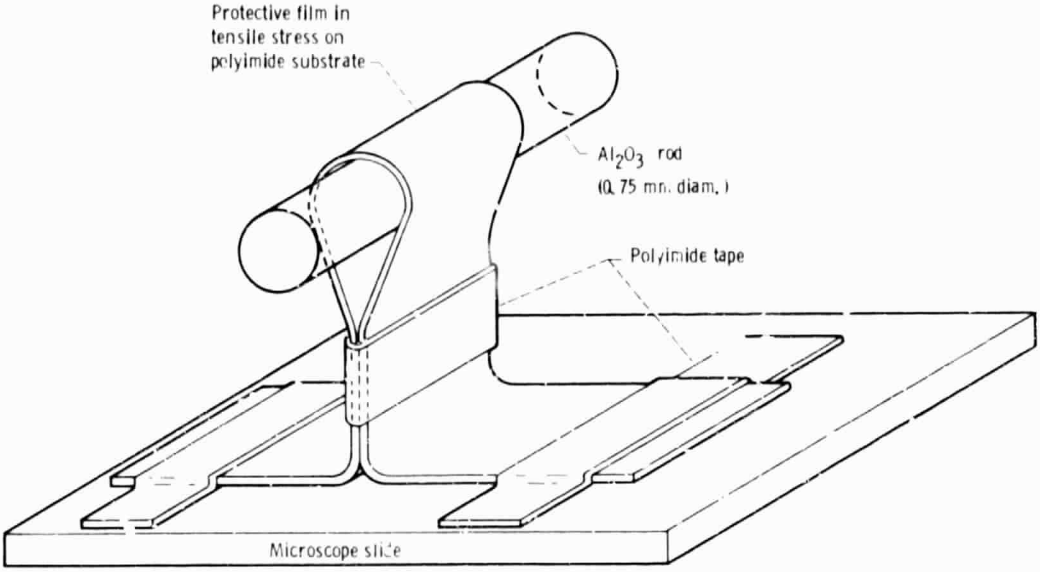

Figure 4. - Sample configuration used to evaluate the synergistic effects of tensile mechanical stress in the protective coating and atomic oxygen exposure.

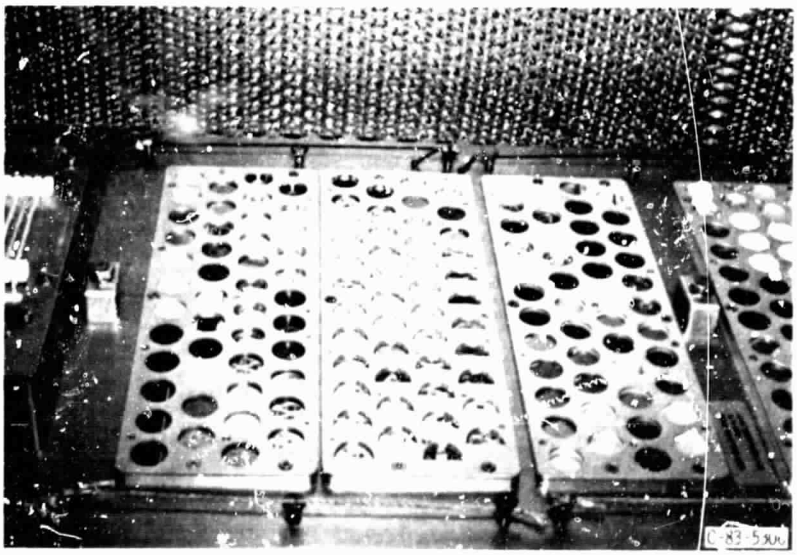

Figure 5. - In-space experiment sample tray.

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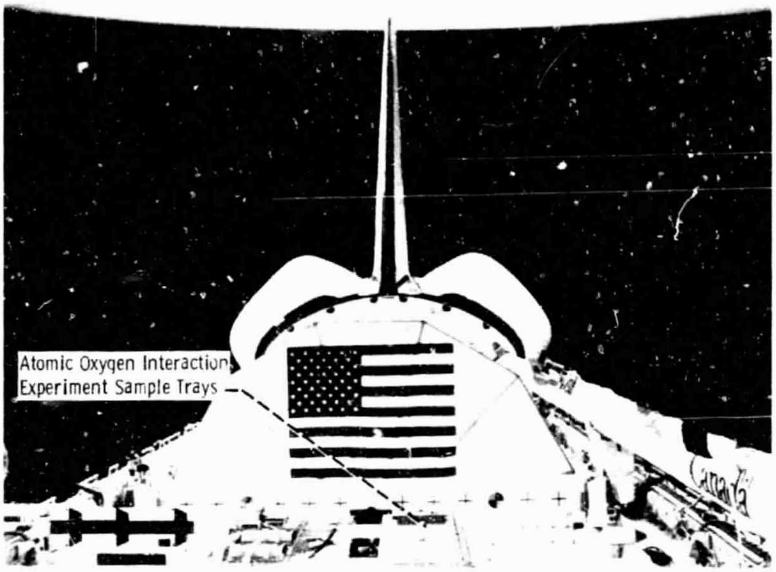


Figure 6. - In-space experiment in the bay of STS-8.

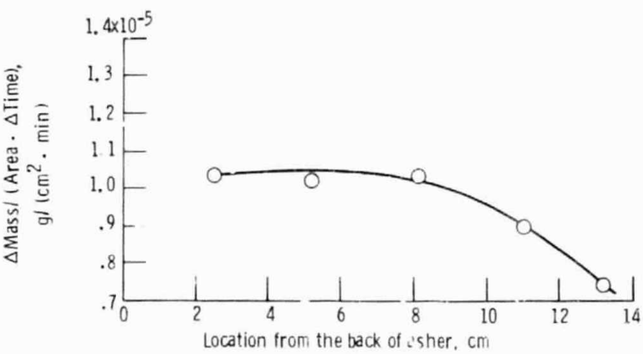


Figure 7. - Dependance of rate of mass loss per unit area of Kapton® (0.127 mm thick) with position along axis of R F plasma asher.

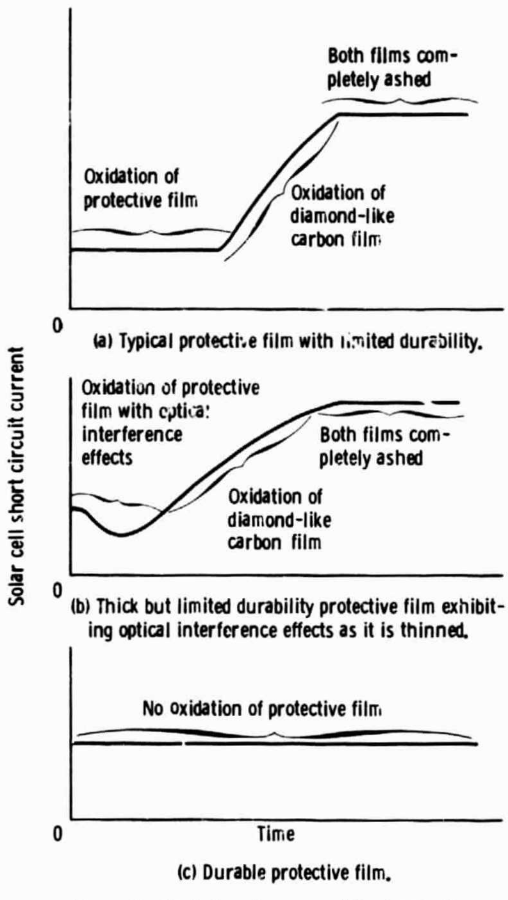


Figure 8. - Typical performance plots of protective films on Kapton ®.

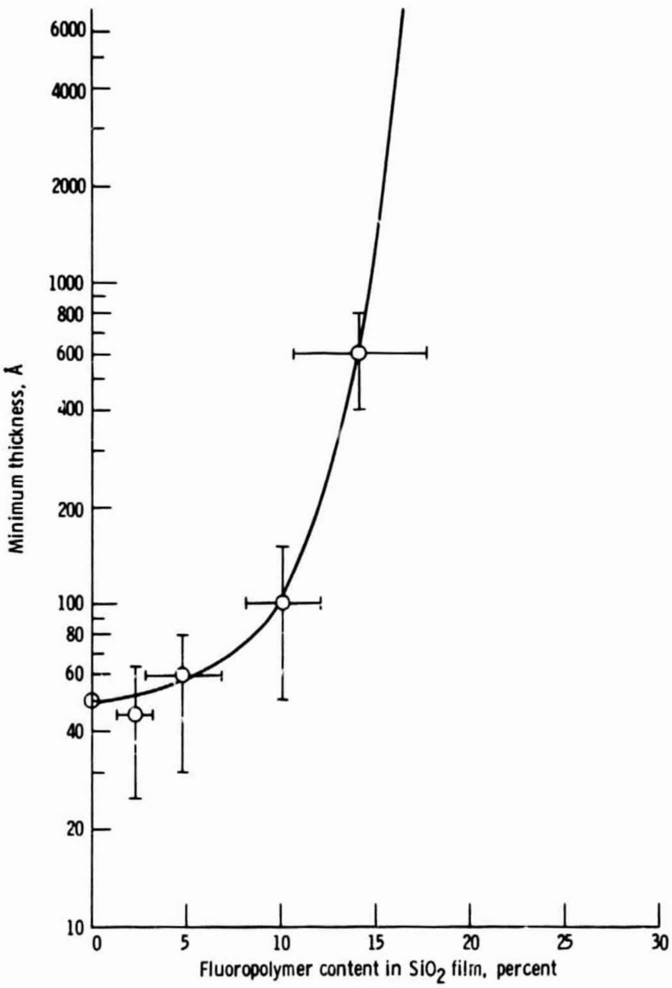


Figure 9. - Minimum film thickness for permanent oxidation protection of diamond-like carbon films versus volume percent fluoropolymer in SiO_2.

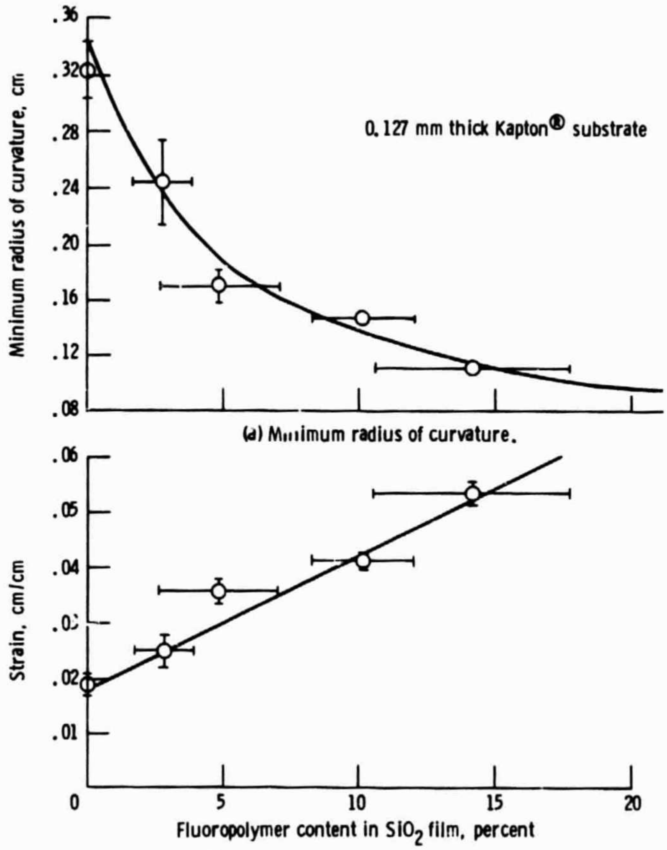
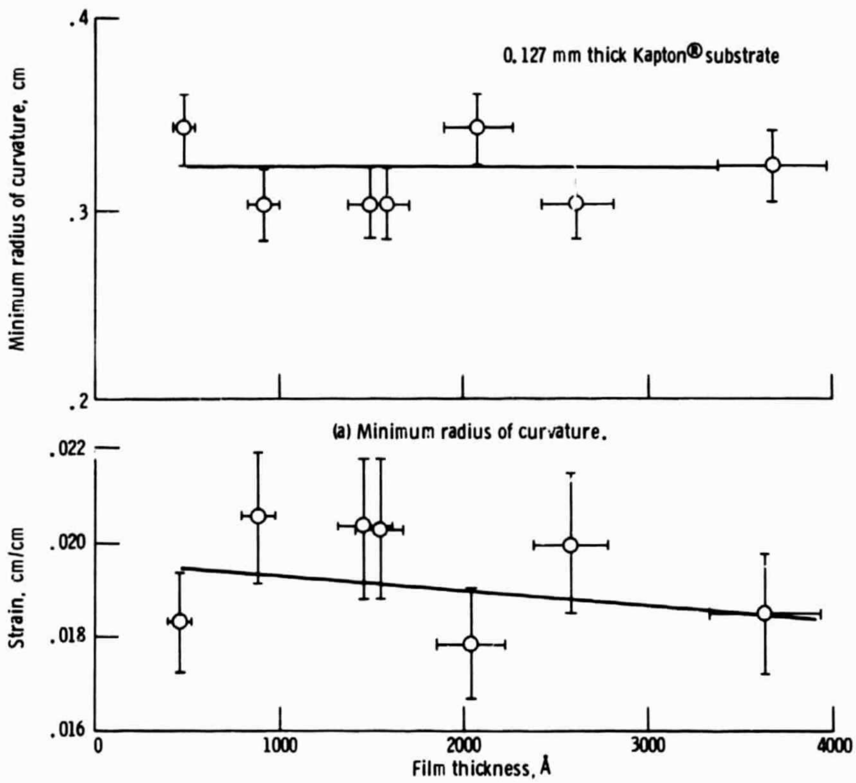


Figure 10. - Minimum radius of curvature and maximum strain that a codeposited SiO_2 - fluoropolymer film (1000 Å) can survive without brittle failure.



(b) Maximum strain without brittle fracture.

Figure 11. - Minimum radius of curvature and strain that a pure SiO_2 film can survive without brittle fracture as a function of film thickness.

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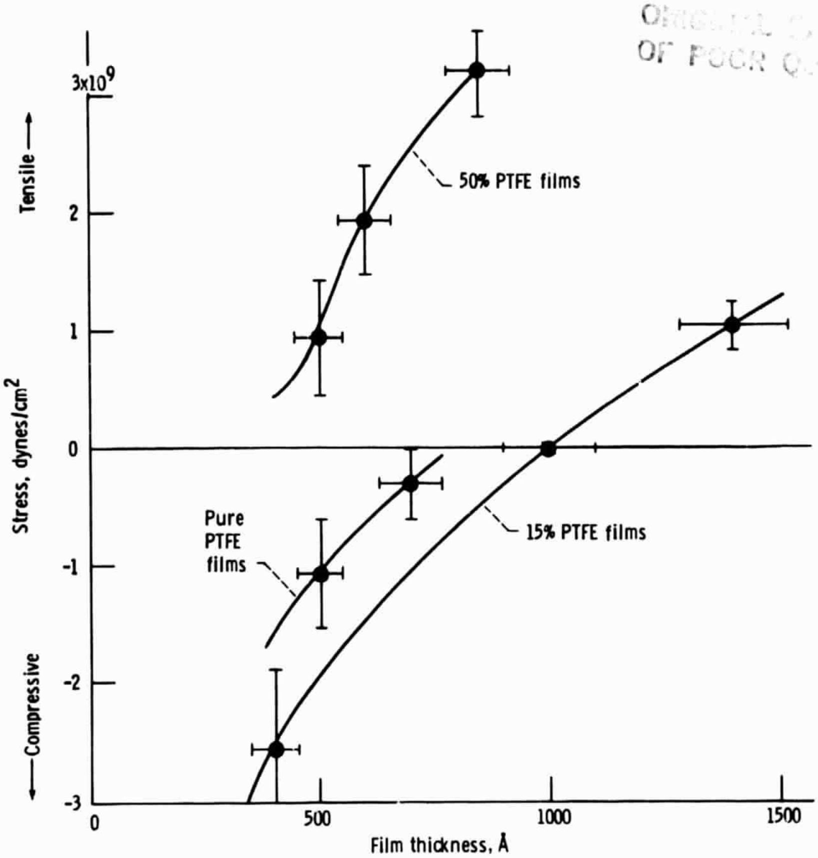
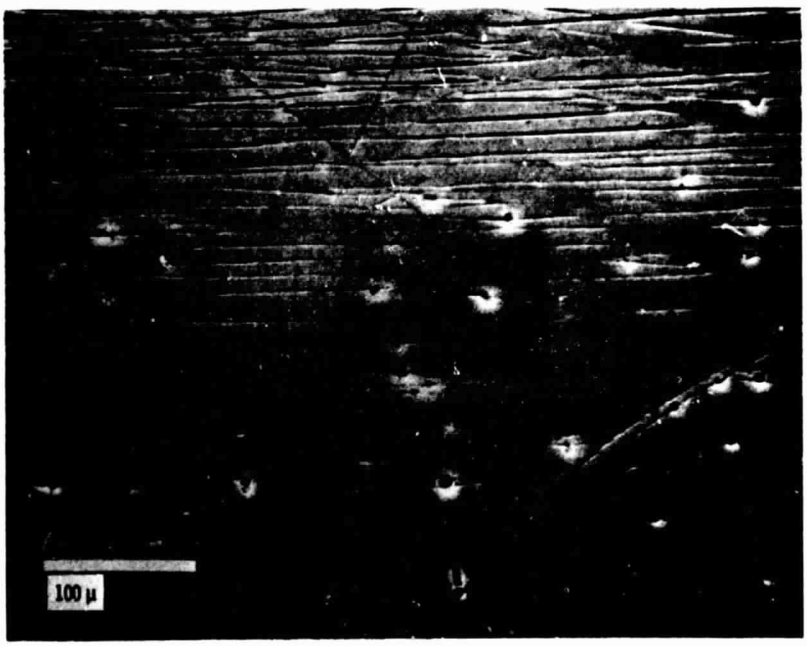


Figure 12. - Intrinsic stress of codeposited thin films on silicon substrates.



(a) Indium-tin oxide film with low stress in lower portion and high stress in upper portion of photograph.

Figure 13. - SEM photographs of protective films subjected to tensile stress failure and simultaneous ashing.

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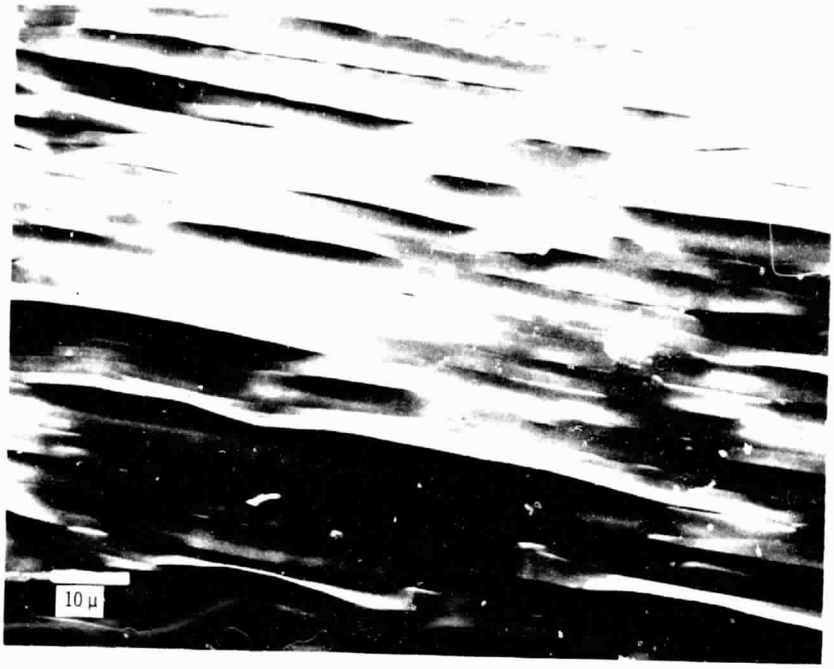


(b) 400 Å SiO_2 film.

Figure 13. - Continued.

(c) 1000Å film of 14% fluoropolymer in SiO_2.

Figure 13. - Concluded.

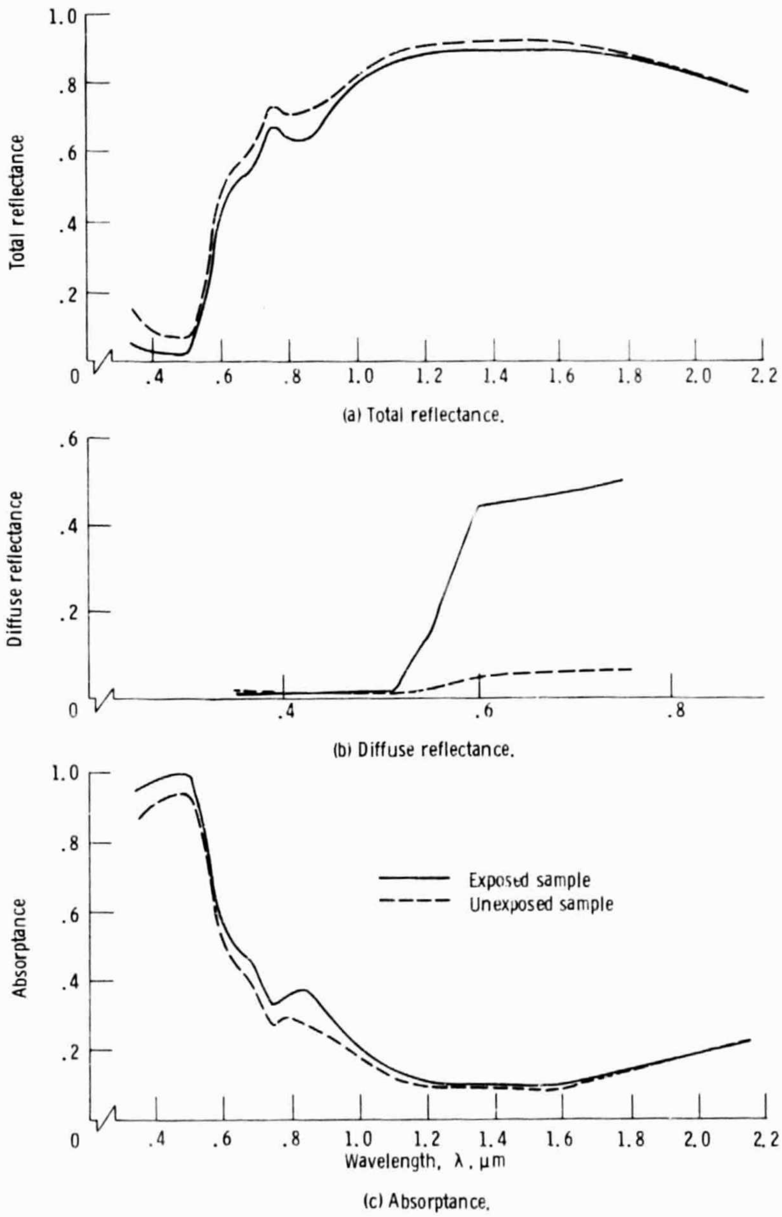
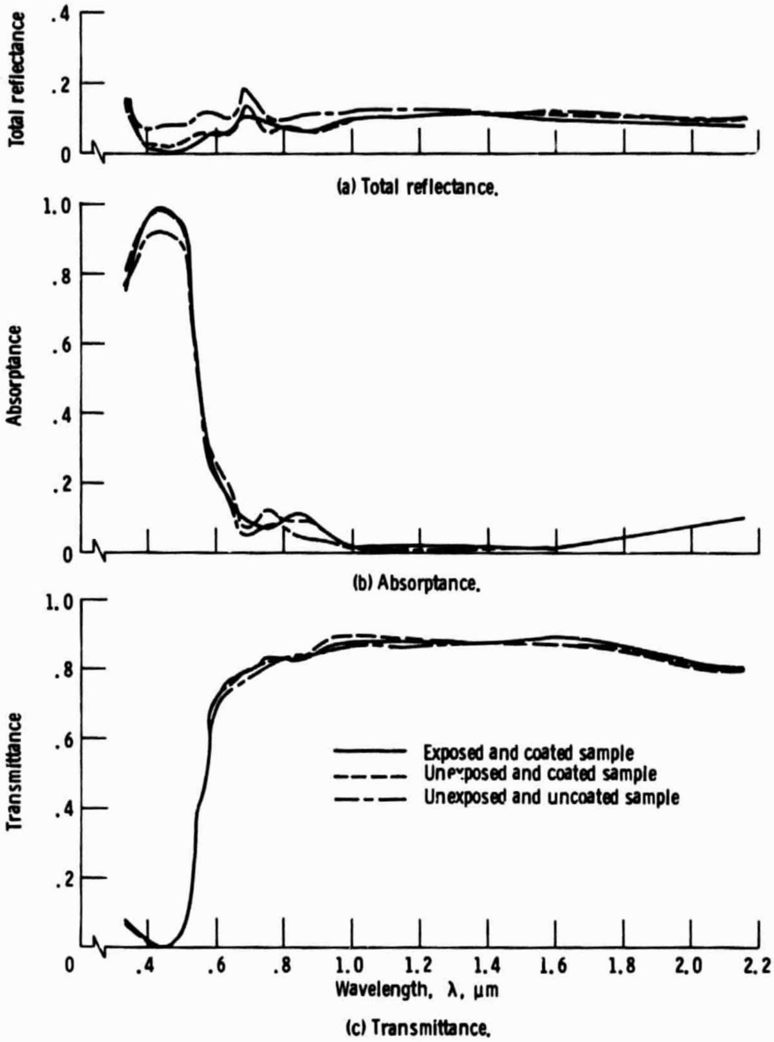


Figure 14. - Optical properties of unprotected Kapton® (0.127 mm thick with an aluminum film on the exposed surface) for samples unexposed and exposed to low earth orbital environment.



(c) Transmittance.

Figure 15. - Optical properties of ≥96% SiO_2 ≤4% PTFE coated Kapton® samples unexposed and exposed to low earth orbital environment compared with uncoated and unexposed Kapton®.

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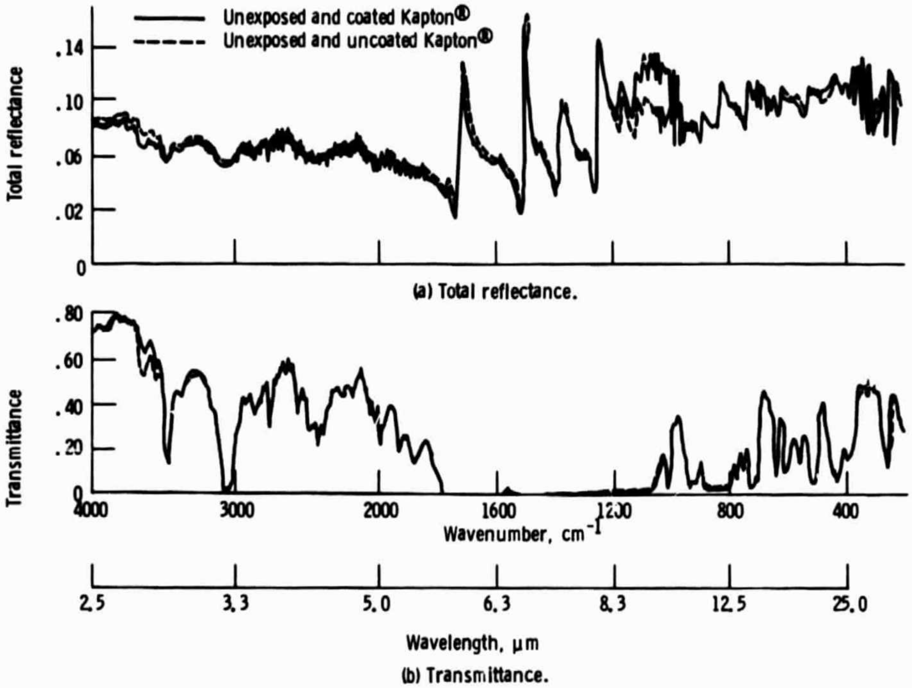

Figure 16. - Infrared reflectance and transmittance of Kapton® prior to and after deposition of a ≥ 96% SiO_2 ≤ 4% PTFE film 650 Å thick.

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16. Abstract

Ion beam sputter-deposited thin films of Al_2O_3, SiO_2, and a codeposited mixture of predominantly SiO_2 with small amounts of a fluoropolymer were evaluated both in laboratory plasma ashing tests and in space on board Shuttle flight STS-8 for effectiveness in preventing oxidation of polyimide Kapton®. Measurements of mass loss and optical performance of coated and uncoated polyimide samples exposed to the low earth orbital environment are presented. Optical techniques were used to measure loss rates of protective thin films exposed to atomic oxygen. Results of the analysis of the space flight exposed samples indicate that thin film metal oxide coatings are very effective in protecting the polyimide. Metal oxide coatings with a small amount of fluoropolymer codeposited have the additional benefit of great flexibility.

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