Plasma-Deposited Protective Coatings for Spacecraft Applications

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This article describes the properties of thin films that can be applied to polymers used on spacecraft to protect against environmental influences that may affect the life and performance of a space mission. These protective films, derived from volatile compounds via microwave glow discharge, include amorphous hydrogenated silicon and inorganic silicon compounds (silicon nitride, oxide, and oxynitride). The paper describes the performance of these coatings in the presence of deleterious effects of the space environment, including atomic oxygen degradation, electrostatic charging, thermal excursions, and vacuum. The coatings are thin and adhere tightly to the substrate. Application of the coatings does not appear to alter the thermal radiative properties of the substrate. Electrical performance of underlying material in the microwave frequency range is unaffected. Accordingly, these materials provide promising candidates for exterior surfaces of spacecraft to protect the underlying materials from the space envrionment.

Introduction

THE design of spacecraft structures is driven by environmental factors that affect their functional performance. Although a great deal of work is being directed toward the solution of the problem of degradation of polymeric and other materials by atomic oxygen in low Earth orbit (LEO),[1-4] this is only one of the many environmental factors that can affect the physical and functional performance of spacecraft. Other factors include thermal cycling, vacuum, radiation (both charged particles and energetic photons), debris, and micrometeoroid impacts. Also, man-made environmental factors such as electromagnetic interference (EMI) must be considered. Therefore, coatings that may be proposed for exterior surfaces of spacecraft must be able to withstand all of the effects of the space environment, although the relative importance of each effect will be dependent on the orbit and application of any particular hardware. In particular, evaluation of atomic oxygen resistant coatings must include the performance in the presence of other factors, perferably taken together.

All spacecraft are affected by atomic oxygen degradation. Those spacecraft in LEO are most prone to long-term attack, but even short times spent in a parking orbit for a geostationary spacecraft can result in exterior surface damage. When organic materials are exposed to the highly oxidizing O atoms, especially at the elevated velocities of spacecraft, the observed result has been rapid erosion (loss of mass) and surface roughening. This, in turn, leads to irreversible degradation of the physical characteristics (optical, thermal, electrical, and mechanical) for which the surface or structural members were designed.

At higher altitudes, the Van Allen belts and solar wind (e.g., in geostationary orbit) have other deleterious effects on polymers, namely, electrostatic charging and eventual surface flashover. These events have been shown to cause loss or near loss of spacecraft.[5]

This paper describes the performance of thin film protective layers[6] that can be applied to polymers used on spacecraft such as Kapton® polymide and epoxy resin. These protective films, derived from volatile compounds via microwave glow discharge, include amorphous hydrogenated silicon (a-Si:H) and inorganic silicon compounds (silicon nitride, P-SiN; oxide, P-SiO_2; and oxynitride, P-SiON).

The next section outlines the techniques for producing coatings. This is followed by a description of the performance of coated and uncoated specimens under test conditions simulating the harmful environment of space, namely, exposure to erosion by atomic oxygen, to charging by energetic particle flux, and to thermal cycling. In addition, structural, RF, and thermal radiative property measurements are reported before concluding that a very favorable overall balance of properties shows these materials to be very promising candidates for use on spacecraft.

Protective Coatings for Spacecraft Materials

It is possible to apply very thin coatings onto the exposed surfaces of organic materials in order to protect them against the harmful effects just described. Experiments with thin layers of polytetrafluoroethylene (PTFE, produced by plasma spraying),[2] sputtered or vapor deposited indium-tin oxide (ITO),[2] and a SiO_2-PTFE mixture prepared by ion beam sputtering[7] have been reported to protect against attack by atomic oxygen. It was found, in both ground-based laboratory and in-space tests, that these thin protective coatings can act as barriers against oxidative erosion of the underlying polymer.

The properties required for such a protective barrier coating include the following: the barrier must be resistant to atomic oxygen attack, the barrier must be flexible and abrasion resistant, the barrier must be be tolerant but not alter the substrate's optical properties, the barrier must be thin, lightweight, and strongly adherent, and, finally, surface conductiv-

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ity should be high in order to prevent the buildup of harmful potential gradients that result from charging.

The object of the present study was to evaluate the performance of thin barrier coatings applied to spacecraft or spacecraft components by low-pressure plasma (glow discharge) deposition (patents pending). Performance characteristics of various coating materials were measured in a simulated LEO environment.

The use of glow discharges for preparing thin solid films is well known.[8-11] The workpiece to be coated is placed into a reactor chamber that is evacuated. A glow discharge is established in a reagent gas flow, which results in a plasma-chemical reaction leading to the desired thin film deposit. Although plasma excitation in the audiowave, radiowave, or microwave frequency ranges is possible, microwave frequency is preferred because of the higher deposition rates obtainable. The following types of thin films have been prepared: 1) plasma polymers, 2) inorganic insulating films, and 3) semiconducting and conducting materials. In category 1, films may be derived from hydrocarbon, fluorocarbon, organosilicon, or organometallic monomer compounds. Typical category 2 materials are silicon compounds such as P-SiO_2, P-SiN, or P-SiON. These may be prepared by reacting silane (SiH_4) with an appropriate second gas (or mixture) such as N_2O, NH_3, N_2, etc. Films in category 3 include amorphous hydrogenated silicon and germanium, respectively prepared from SiH_4 and GeH_4 gas, and metal films prepared from volatile organometallic compounds. Except those prepared from hydrocarbon reagents, in principle, many of the aforementioned materials can provide suitable barriers against attack by O atoms. One of the most promising of these materials is amorphous silicon, a semiconductor, since changes in the processing conditions can significantly alter its electrical properties. It has been shown that procedures, such as the addition of gaseous dopants like phosphine or diborane, changes in reagent flow rate, substrate temperature, power density in the plasma, and reactor pressure can very significantly alter the electrical conductivity of this material.[8]

A series of specimens from categories 1-3 was fabricated in this study. A microwave plasma reactor was used to deposit various thin film materials onto organic polymers that are important to applications in spacecraft, and onto some test devices. Microwave power, at 2.45 GHz, was coupled into the plasma chamber using a slow-wave applicator (periodic traveling wave structure). As described elsewhere,[9] the resulting microwave plasma allows rapid and uniform film deposition over large surface areas. The plasma deposition process is not a line-of-sight technique like some others, such as magnetron sputtering. Accordingly, the plasma process is less sensitive to shadowing, which can result in pinholes in the coating.[12] More details of apparatus and plasma coating procedures may be found in Refs. 8-11.

The materials tested included coatings of plasma polymers, inorganic silicon compounds, and a-Si:H, respectively. The silicon compounds investigated were P-SiN, SiO_2, and P-SiON; the most promising plasma polymer was organosilicon (plasma polymerized hexamethyldisiloxane, PP-HMDSO). The protective coatings were applied to copper radiating patches, duroid substrate, epoxy, and Kapton. The typical film thickness was 0.5-0.7 μm.

Mass Loss Due to Atomic Oxygen Exposure

Exact simulation of the oxygen atom environment of LEO is very difficult to achieve in an Earth-based laboratory. Several approaches have been described[12-16] using varying techniques that appear to yield results that compare well to true space exposure. An evaluation of these techniques is beyond the scope of this paper but has been described elsewhere.[17] However, to assess the performance of the plasma-deposited coatings, three techniques were used to simulate the atomic oxygen erosion environment of LEO. These were RF oxygen plasma, microwave oxygen plasma, and an atomic oxygen beam facility. Each of these three techniques has its own particular characteristics that approximate the LEO environment to a greater or lesser extent, as described in Ref. 17. However, they are all capable of generating high concentrations of oxygen atoms, which is necessary to simulate the LEO environment. The coatings were subjected to the three testing techniques to ensure that the results of the testing were not influenced by the limitations of any one simulation technique.

The RF oxygen plasma facility at Lockheed Missiles & Space Company[13,14] was used to evaluate coatings of SiO_2 and a-Si:H on Kapton substrates. The operation and capabilities of this facility have been described elsewhere[13,14] and are not repeated here. However, the removal of organic materials by oxygen plasma is well known as it is among the earliest applications of plasma chemistry. For example, it is being used very extensively by industry for stripping photolithographic resist in microelectronic fabrication. This process has also been shown[13,14] to be an effective screening process for atomic oxygen resistant materials. Results from these works have shown that materials that are attacked by atomic oxygen in Shuttle flight experience are also attacked by an oxygen plasma. The Lockheed facility and technique have been calibrated through the evaluation of erosion of standard materials such as Kapton. The equivalent oxygen flux has been shown to compare to LEO atomic oxygen flux of approximately 6×10^{15} atoms/cm^2-s.

Material specimens of approximately 1 in. × 6 in. wrapped in aluminum foil to eliminate effects of the edges and the uncoated back side were exposed for periods of 1, 3, 8, and 24 h, corresponding to fluence (time integration of flux) levels of 2.2×10^{19}, 6.5×10^{19}, 1.7×10^{21}, and 5.2×10^{21} atoms/cm^2. During the testing, a reference standard of Kapton was exposed for the same durations to calibrate the chamber. Mass loss measurements were taken of the materials after exposure with particular care to obtain measurements of the dry surface before it had opportunity to absorb water from the air during measurement. In addition, specimens were dried in a desiccator for at least 24 h prior to initial measurement. Whereas the uncoated Kapton reference exhibited significant mass loss, the coated materials showed very little change in mass, as shown in Table 1. The slight reduction in mass for the silicon dioxide is attributed to removal of atmospheric contaminants from the specimen surface by the plasma. The slight increase in mass by the amorphous silicon coatings can be shown to be due to oxidation of the outermost surface layer, which will be described later.

Microwave plasma apparatus used to coat the materials is also useful as an atomic oxygen simulation facility due to its high yield of O atoms compared with conventional lower frequency excitation.[18,19] In this mode, the reagent gas or vapor used to generate the coating is replaced with oxygen. By exposing coated and control specimens to this latter plasma, an evaluation of the response to a LEO environment was achieved in the laboratory. Coated polymer specimens that had been stored in a desiccated chamber were first weighed and then exposed to oxygen plasma in the microwave plasma chamber described in Ref. 18 to simulate the LEO atomic oxygen environment. The mass loss of the various specimens were determined from weight loss using an electronic microbalance. A Kapton reference specimen was also exposed for an equivalent period of time for a calibration reference. The mass loss for

Table 1 Change in mass after RF oxygen plasma exposure

		Mass change, μg/cm^2			
Coating	Thickness, μm	60 min	180 min	480 min	1440 min
SiO_2	0.7	−8.7	−11.0	−6.1	−8.8
a-Si:H	0.7	+2.2	+7.2	+25.0	+21.5
a-Si:H	1.0	−4.7	−5.0	−5.5	−2.5
a-Si:H	1.2	0.0	+9.9	+19.0	+24.1
Uncoated (ref)	—	−108.0	−324.0	−864.0	−2592.0

untreated substrate materials was approximately 0.77 ± 0.2 mg/cm^2. All of the coatings investigated are seen to reduce the weight loss very substantially, in some cases to an immeasurably small value, as shown in Table 2.

Finally, coated specimens were also exposed in an atomic oxygen beam simulation facility[15] to supplement the tests conducted in the oxygen plasma reactors. Briefly, this facility consists of three major components: a microwave plasma torch, a sampler-skimmer interface, and a vacuum chamber with the associated support electronics. The plasma torch is used to generate a stream of essentially neutral oxygen atoms in a helium carrier gas. The sampler-skimmer system strips a portion of the lighter carrier gas from the plasma and produces a diverging atomic oxygen beam, which is then directed at the specimen in the connecting vacuum chamber. The input power to the plasma determines the energy level of the incoming atoms, which was held constant for these tests at 2.2 eV. Flux at the specimen location is determined by both input flow rate and specimen position. The flux used in these tests was approximately 8×10^{16} atoms/cm^2-s, which provides an acceleration factor on test time over that experienced in a nominal Shuttle orbit, but it was similar to that of the RF plasma facility.

Specimens of Kapton coated with SiO_2, a-Si:H, and PP-HMDSO were exposed to a total fluence of 3×10^{20} atoms/cm^2. Another specimen coated with SiO_2 was exposed to a fluence of 3×10^{21} atoms/cm^2. The exposed area consisted of a circle of approximately 1 cm in diameter in the center of a larger specimen, which made mass loss measurement using an electronic balance more difficult than for the larger specimens exposed in the RF or microwave plasma facilities. However, measurements taken after exposure showed no meaningful loss of mass for any of the coating materials. For the case of an uncoated 25-μm-thick Kapton sample, the same fluence resulted in complete removal of the material where the beam had impinged.

The results of mass loss from the three testing techniques are compared in Table 3. Since the fluence of each technique is not directly measurable, the equivalent fluence is estimated from the mass loss experienced by an uncoated Kapton specimen exposed to the same test conditions for an equal time. Measured changes in mass are within the tolerance of the sensitive electronic balance equipment in all cases for the coatings but were easily measured for the equivalent uncoated specimens.

Although each of these three techniques for atomic oxygen simulation provides very similar results, there are significant differences in each that complement the others. In particular, both the RF and microwave plasma techniques subject the specimens simultaneously to vacuum ultraviolet (VUV) radiation, which would therefore include synergistic effects between atomic oxygen and VUV. The atomic oxygen beam facility provides a directed, impinging beam much like that actually experienced by a spacecraft in LEO. However, a specimen placed on the powered electrode of a capacitively coupled RF discharge reactor also experiences bombardment by a flux of directed impinging ions. This arises due to the development of a negative dc bias on this electrode, which can accelerate positive ions from the plasma to energies of tens or even hundreds of eV.[20] This energetic particle bombardment does not occur in the microwave reactor. Coupled with the VUV, the RF discharge provides a good simulation of the atomic oxygen fluence experienced by spacecraft.

Table 2 Change in mass after microwave oxygen plasma exposure

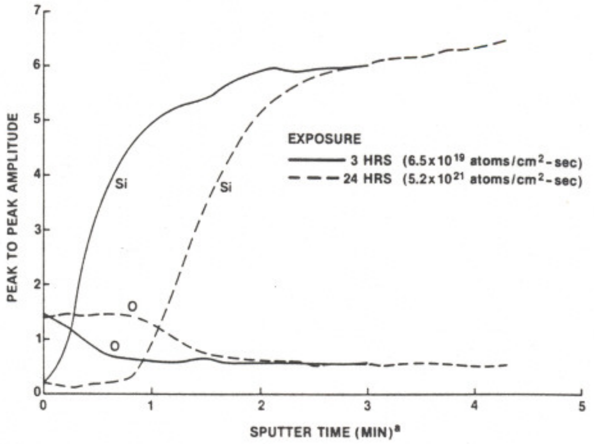
Sample number	Sample description (coating type)	Mass loss,[a] mg/cm^2
1	Control (untreated)	0.77
2	a-Si:H	0.01
3	a-Si:H	0.00
4	P-SiN	0.00
5	P-SiO_2	0.02
6	P-SiON	0.02
7	PP HMDSO	0.05

[a] O_2 flow: 200 sccm; pressure: 100 mTorr; microwave power: 300 W.

Table 3 Mass loss summary

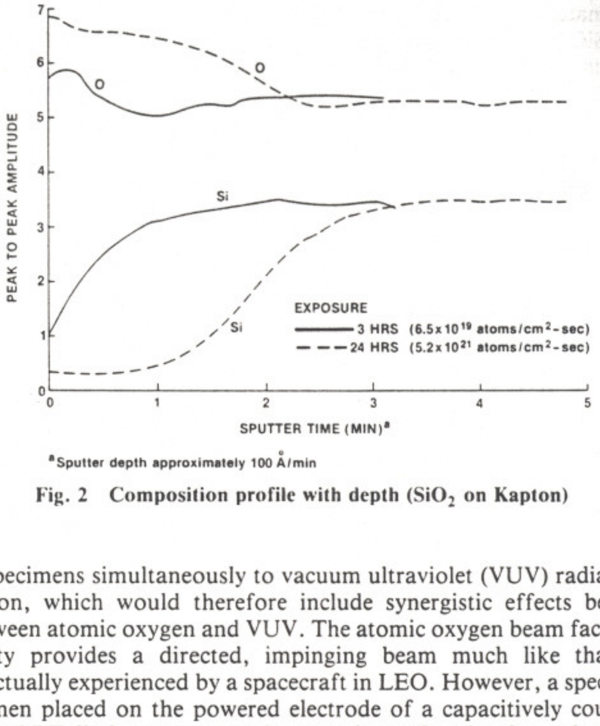
Simulation technique	Coating	Equivalent fluence, atom/cm^2	Normalized mass loss[a]
Microwave plasma	SiO_2	2×10^{21}	0.02
	a-Si:H	2×10^{21}	0.01
RF plasma	SiO_2	5×10^{21}	0.003
	a-SiO_2	5×10^{21}	0.009
Atomic oxygen beam	SiO_2	3×10^{21}	0.02
	a-Si:H	3×10^{20}	0.09

[a] Normalized to mass loss for Kapton.



[a] Sputter depth approximately 10 Å/min

Fig. 1 Composition profile with depth (a-Si:H on Kapton).



[a] Sputter depth approximately 100 Å/min

Fig. 2 Composition profile with depth (SiO_2 on Kapton)

Surface Analysis

Detailed examinations, using a scanning electron microscope (SEM), of the coated surfaces both before and after simulated atomic oxygen exposure in both the atomic oxygen beam and the plasma facilities showed no apparent change from the unexposed to the exposed. No distinguishing features were apparent that would indicate significant erosion of the surface, supporting the mass loss measurements reported earlier.

In order to establish the cause of the mass gain for the a-Si:H coated Kapton after exposure to the RF plasma, the specimen was examined with an Auger probe to investigate surface changes resulting from oxygen exposure. A plot of Auger signal amplitude vs depth with the beam locked on first the silicon then oxygen peak for material exposed for 3 and 24 h is shown in Fig. 1. It is clear from this figure that the outer layer is oxidizing to an increasing depth with increasing exposure time, supporting the suggestion that the increase in mass seen after oxygen exposure was in fact real and due to oxidation. Although it is difficult to accurately calibrate the depth of penetration of the beam with sputtering time, it is estimated that the depth of penetration of the oxide layer after 24-h exposure is approximately 80–100 A. A similar plot of Auger signal from Kapton coated with SiO_2 is shown in Fig. 2. A lower increase in oxygen content after increased exposure is apparent, suggesting that the initial coating may not have been stoichiometric but silicon rich. The depth of oxidation is similar to that for the amorphous silicon coating. In both of these cases, the oxidation of the outer layer of the coating is a positive feature for the coating, providing a healing mechanism for damage that might result from nicks, scratches, or mechanical abrasion of the coating surface.

Electrical Performance Testing

As exterior coatings, materials are subjected to both natural and induced electrical environments. The natural environment can produce a charge buildup on the exterior surfaces of spacecraft, particularly if these are composed of nonconducting materials such as Kapton or Teflon® films. In addition, thermal protection is often used in front of radiating components such as antennas to aid in control of surface inaccuracy resulting from thermal distortion. Transparency to microwave transmission is a primary requirement of such sun shields. In order to assess the electrical performance and response of the coating materials, three techniques were used.

To measure the effect of electrical surface conductivity, specimens were exposed to corona discharge using a charging voltage of −4000 V. The decay rate of the resulting potential was then measured using a noncontacting electrostatic voltmeter (Monroe Instruments Corp. Isoprobe). Results of these tests are shown in Table 4.

Whereas the three control specimens retained surface charge for lengthy periods (they become electrets), coated specimens disspiated their surface charge quite rapidly. Not surprisingly, this decay was most rapid in the case of the semiconducting film a-Si:H, but even for the insulating materials such as P-SiO_2 and P-SiON, it was far more rapid than for the untreated control specimens. The charge decay rate of coated specimens was even more rapid, in practically all cases investigated, following exposure to oxygen plasma. In particular, for specimens 4, 7, 9, and 16 in Table 4, the surface potential had dropped substantially 5 min after charging, following 20 min of exposure to oxygen plasma treatment and renewed corona charging. Based on these results, these coatings should be even more effective in space in preventing charge buildup than in the laboratory.

Table 4 Results of surface charge retention testing

Sample number	Description	Negative surface potential at the indicated time, V 0 s	30 s	5 min	2 h	4 days
1	Kapton control	2560	2560	2480	2080	820
8	Epoxy control	2500	2480	2400	1890	0
13	Mylar control	2460	2460	2440	2200	1700
2	Kapton/Si:H	1800	900	0	—	—
4	Kapton/P-SiN	2400	2340	1810	990	0
5	KaptonP-SiO_2	400	220	70	0	—
6	Kapton/P-SiON	990	910	610	550	0
7	Kapton/PP HMDSO	2560	2560	2480	2000	1250
9	Epoxy/a-Si:H	2000	1250	780	90	0
16	Mylar/P-SiON	450	330	80	0	—
17	Mylar/P-SiO_2	750	720	510	0	—

Table 5 Results of electrostatic discharge simulation testing

Specimen[a]	Coating	Coating thickness	Discharges	Substrate current
1	a-Si:H	1.0 μm	0	—
	Reverse side	—	3	175 A
2	a-Si:H	1.5 μm	5	85 A
	Reverse side	—	6	183 A
3	P-Si:N	0.5	0	—
	Reverse side	—	4	53 A

[a] Kapton substrate.

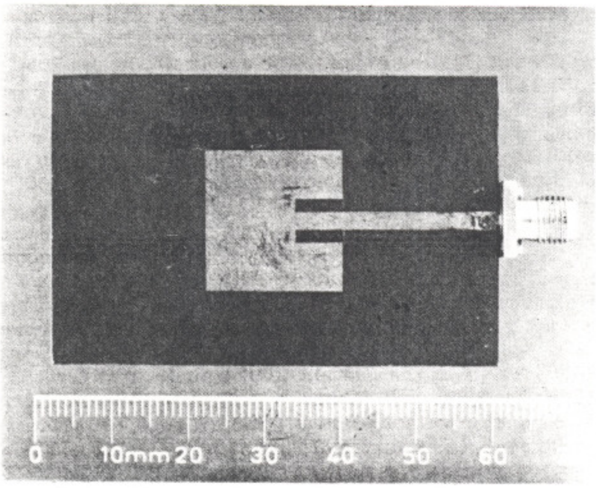


Fig. 3 Microstrip radiating patch.

To further substantiate the electrical surface conductivity tests, two coating materials, a-Si:H and P-SiN, deposited over Kapton were tested in an electrostatic discharge simulation apparatus.[21] Two specimens of a-Si:H coating that were somewhat different in composition and morphology (due to changes in processing parameters) and thickness were evaluated. These specimens, together with control specimens of uncoated Kapton (reverse side of coated specimen to reduce variability), were exposed to a 20-keV electron beam at current densities of 25, 50, and 100 nA/cm^2 for 20 min at each current density. Discharge currents measured from the substrate and the mask are shown in Table 5. In two cases, the coatings eliminated arc discharges completely, whereas in the third case, the coating reduced the arc discharge strength by 50%. The difference in discharge occurrence between specimens 1 and 2 (see Table 5) may not be due entirely to the difference in coating thickness. It may be due in part to the variability sometimes observed in performing experiments of this type, which suggests the need for further experimentation to establish more precisely the role of coating thickness. A further observation was that the coated specimens exhibited reduced long-term charge retention. When removed from the chamber, the specimens showed no visible signs of exposure to the electron beam.

The electromagnetic transparency at microwave frequencies of the coating that might be applied to a sun shield in front of an antenna is an important consideration. Coatings of SiO_2 and a-Si:H were applied directly to prototype radiation patches for a microstrip planar array designed to operate at L-band, as shown in Fig. 3. Insertion loss measurements were made both before and after coating, with no measurable difference recorded. The coated radiating patches responded with no apparent effect of the coating, although, particularly in the case of the a-Si:H, the coating has been shown to be sufficiently conductive to eliminate or reduce electrostatic charge buildup.

Thermal Radiative Properties

Passive thermal control of an orbiting satellite is established by balancing energy absorbed with thermal radiant energy emitted by the external surfaces at the desired operating temperature for the spacecraft. The solar absorptance α_s and the

Table 6 Thermal radiative properties of coated substrates

Substrate	Coating	Thickness, μm	Solar absorptance, α	Thermal emittance, ε	α/ε
Kapton (ref)	—	—	0.20	0.52	0.38
Kapton	SiO_2	0.7	0.22	0.51	0.43
Kapton	SiN	0.5	0.21	0.58	0.36
Kapton	PP HMDSO	0.7	0.32	0.72	0.44
Kapton	a-Si:H	0.5	0.23	0.56	0.41
Gr/epoxy (ref)	—	—	0.88	0.83	1.06
Gr/epoxy	a-Si:H	0.3	0.79	0.80	0.99
Gr/epoxy	a-Si:H	0.5	0.79	0.76	1.04
Copper (ref)	—	—	0.37	0.04	9.25
Copper	SiO_2	0.5	0.36	0.15	2.40
Copper	a-Si:H	0.5	0.37	0.24	1.54
Duroid (ref)	—	—	0.82	0.91	0.90
Duroid	SiO_2	0.5	0.82	0.87	0.94
Duroid	a-Si:H	0.5	0.67	0.70	0.95

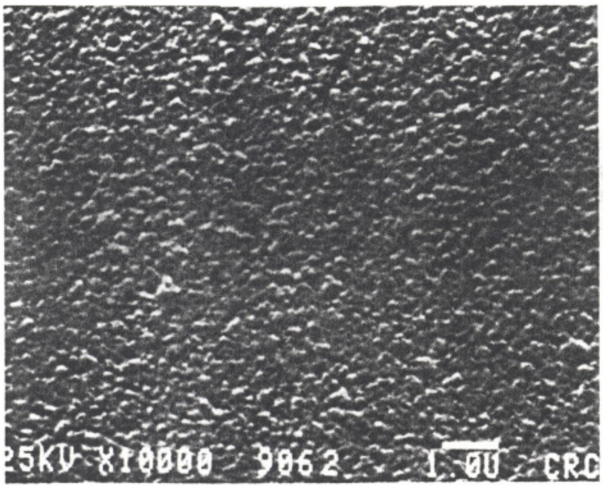


Fig. 4 Photomicrograph of a-Si:H coating over Kapton after thermal cycling showing no evidence of cracking or other damage.

hemispherical emittance ϵ_T must remain constant to maintain thermal balance. Accordingly, any coating applied to the external surface of the spacecraft must not alter this ratio.

A hand-held Gier-Dunkle Solar Reflectometer, Model MS-251, was used to measure solar absorptance, and a Gier-Dunkle Infrared Reflectometer, Model DB-100, was used to measure emittance. Since absorptance is somewhat thickness dependent for a translucent film such as Kapton, an uncoated standard of the same material was established for a reference. The results are shown in Table 6. Most of the protective overcoats did not appreciably perturb the thermal radiative characteristics (ratio of α/ϵ of the underlying materials). It has been shown that these values do not change after exposure to oxygen plasma.[6] This, of course, is vitally important for thermal blanket applications, among others.

Thermal Cycling

Coatings of SiO_2 and a-Si:H over Kapton were subjected to thermal cycling to evaluate resistance to cracking and spalling. Specimens of each material were cycled in a dry nitrogen environment from −196 to 125°C for a total of 25 cycles each. Total time for each cycle was approximately 1 h. Temperature was measured with an iron-constantan thermocouple. The specimens were analyzed with the aid of a scanning electron microscope after cycling to evaluate performance. The photomicrograph shown in Fig. 4 verifies that no cracking occurred, confirming that the coatings are tightly adherent to the substrate.

Conclusions

Evaluation of thin barrier coatings applied to typical spacecraft polymeric materials using a microwave plasma reactor have been conducted. The coating materials have been shown by three techniques (RF plasma, oxygen beam, and microwave plasma) to be resistant to simulated atomic oxygen attack in low Earth orbit. Photomicrographs of the coated surfaces before and after exposure to the atomic oxygen attack obtained using the scanning electron microscope confirm that the surfaces were not attacked by the beam. All coatings convert to a SiO_2 at the surface, which is chemically inert toward atomic oxygen. Besides being inert, the material acts to prevent charge buildup that might lead to harmful electrostatic discharge, yet it is transparent to microwave radiation. The coatings do not appreciably alter the exterior thermal radiative properties of the underlying material and resist damage due to thermal cycling. In conclusion, these materials provide promising candidates for protective coatings for exterior surfaces of spacecraft to protect the underlying materials from the deleterious effects of the space environment.

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