

Atomic Oxygen Resistant Coating from Poly(tetramethyldisilylene-co-styrene)

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ABSTRACT: A polydisilahydrocarbon, namely, poly(tetramethyldisilylene-co-styrene), synthesized from dimethyldichlorosilane and styrene in 1 : 0.5 mol ratio under dechlorination conditions was evaluated as an atomic oxygen (AO) resistant coating for polyimide film and C-polyimide composite. Exposure of the polymer coating on a quartz plate to an AO fluence of 2.1×10^{21} atoms/cm² resulted in practically no mass loss, indicating the AO resistance of the polymer. Ten percent solution of the polymer in toluene was applied on aluminized Kapton[®] polyimide film (125 μ m thick) to obtain a coating thickness of $\sim 5 \mu$ m on the unaluminized surface. In a similar way, the coating was applied on a C-polyimide composite. The coated and uncoated samples of Kapton[®] film and C-polyimide composite were exposed to AO in a plasma barrel system. The uncoated alu-

minized Kapton[®] film (125 μ m) lost 6.35 mg/cm² when exposed to AO fluence of 1.6×10^{21} atoms/cm² whereas the corresponding coated film lost only 0.14 mg/cm² even after exposure to AO fluence of 2.1×10^{21} atoms/cm². In the case of the C-polyimide composite, the uncoated sample lost 63.64 mg/cm² on exposure to AO fluence of 1.8×10^{21} atoms/cm² whereas the coated one lost only 0.21 mg/cm² even after exposure to AO fluence of 2.1×10^{21} atoms/cm². SEM studies suggest that the coating offers good protection to the substrates. Formation of cracks on some portions of the coating was noticed. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 94: 2368–2375, 2004

Key words: coatings; polysilanes; inorganic polymers; atomic oxygen resistant coating; low earth orbit

INTRODUCTION

In the last two decades, inorganic and organometallic polymers have gained significance as high temperature protective coatings, thermal protective systems, matrix resins, and preceramic polymers.^{1–5} In our laboratory, we synthesized novel polysilahydrocarbons in which the repeating units contain silicon atoms separated by two or more carbon atoms and studied the microstructure, mechanism of formation, and their conversion to thermally stable polycarbosilane.^{6–18} These polysilahydrocarbons were synthesized through the dechlorination of diorganodichlorosilane with styrene in the presence of sodium. Detailed microstructural analysis of the copolymers suggests that the copolymers in which disilyl and styryl units alternate with each other are formed when the monomer feed ratio of diorganodichlorosilane : styrene is 2 : 1. In addition to these alternating units, polysilyl blocks are present when the monomer feed ratio is more than 2 and polystyryl blocks are present when the monomer feed ratio is less than 2.

Polymeric films, advanced composites, and thermoplastic materials used in the construction of satellites and space stations placed in LEO are susceptible to atomic oxygen (AO) formed by the photodissociation of molecular oxygen formed in the upper atmosphere.^{19–23} Inorganic and organometallic polymers such as polysilanes,²⁴ polyimides,²⁵ polysiloxane,^{26–28} siloxane-epoxy,^{29,30} siloxane-imides,^{31,32} poly(carborane-siloxane)s,^{27,33–35} decaborane-based polymers,^{36–41} and phosphorus containing polyarylene ethers^{42,43} have been evaluated as AO resistant coatings. These polymers on reaction with AO form a protective oxide layer on the surface of the coating, which prevents further reaction of the coating with AO.

Polydisilahydrocarbons, containing alternating units of disilyl and styryl units, are of particular interest as they exhibit good film forming characteristics. The disilyl linkages present in polydisilahydrocarbons can trap AO, resulting in the formation of siloxane linkage that on further interaction with AO would result in the formation of a silica layer on the surface, preventing further oxidation of the coating. In the present article, we report the evaluation of poly(tetramethyldisilylene-co-styrene) (PTMDSS), synthesized through the dechlorination of dimethyldichlorosilane and styrene under dechlorination conditions, as an AO resistant coating for polyimide film and carbon-

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polyimide composites that are susceptible to AO attack.

EXPERIMENTAL

Materials

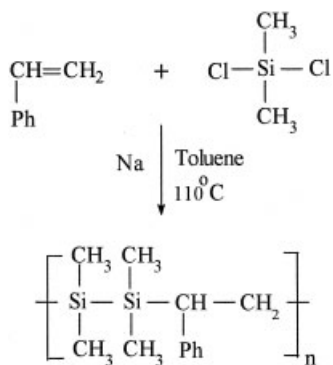
Kapton[®] film of thickness 125 μm aluminized on one side of the film, obtained from DuPont (Wilmington, DE), and carbon-polyimide composite, made in the Vikram Sarabhai Space Centre, India, from T-300 carbon fabric using nadic end-capped addition curable imide as the matrix resin, were used for AO exposure studies. Dimethyldichlorosilane (DMDCS) (Fluka, Buchs, Switzerland) was distilled before use. Toluene (Qualigens, Mumbai, India) was refluxed and distilled over sodium. Methanol (Qualigens) was used without further purification. Styrene (Alpha Bio Chem., Mumbai, India) was washed with 5% sodium hydroxide solution followed by water and dried over anhydrous sodium sulfate. It was purified by vacuum distillation over calcium hydride.

Synthesis of poly(tetramethyldisilylene-co-styrene) (PTMDSS)

PTMDSS was synthesized by reacting DMDCS and styrene in 2 : 1 mol ratio under dechlorination conditions using sodium in refluxing toluene following the procedure of Packirisamy et al.⁶ (Scheme 1). It was characterized by GPC and ¹H-, ¹³C-, and ²⁹Si-NMR spectra.

Characterization of PTMDSS

¹H-, ¹³C-NMR, and ²⁹Si-NMR spectra of PTMDSS were recorded at 300, 75, and 59.6 MHz in CDCl₃ using TMS as the internal standard on a Bruker 300 spectrometer. Molecular weight of PTMDSS was determined by GPC with a Waters ALC GPC 244 instrument using 10⁴, 10³, 500, and 100 microstyragel columns, and tetrahydrofuran as the elutant.



Scheme 1 Synthesis of PTMDSS.

Preparation of coatings

For preparing the coating, a 10% solution of PTMDSS in toluene was prepared and applied on a quartz plate, unaluminized side of an aluminized Kapton[®] film (125 μm), and C-polyimide composite using a brush. These samples were then dried in an air oven at 40°C for 12 h and then under vacuum at 60°C for 15 h.

AO fluence measurement

Uncoated Kapton[®] (125 μm) film, aluminized Kapton[®] film (125 μm), C-polyimide composite, and PTMDSS coated samples were exposed to AO in a plasma barrel system (March Plasmod Plasma Barrel System) operating at RF frequency of 13.56 MHz and 75 watts current input. The system was operated under oxygen flow of 7.23 standard cubic centimeter per min (SCCM). The mass loss of all the samples was measured at different time intervals using a Mettler Toledo balance, and exposure of the samples to air during weighing was kept to the minimum to prevent rehydration. From the mass loss of the uncoated Kapton[®] film, AO fluence, that is, the total number of incident AO per unit area expressed in atoms/cm², was calculated using the equation

$$\text{Fluence (F)} = \frac{\Delta M}{A \cdot \rho \cdot E}$$

where ΔM = initial mass of the sample – mass of the sample at the time of measurement (in grams), A = area in cm², ρ = density in g/cm³, and E = erosion yield in cm³/atom. Erosion yield of Kapton[®] as obtained from AO exposure study in the space environment is 3×10^{-24} g/cm³. Density of Kapton[®] is 1 g/cc. From these data, the AO fluence for a given period of exposure was calculated using the above equation. The mass loss of the coated and uncoated samples was plotted against AO fluence.

Surface analysis

A Hitachi Model S-2400 SEM was used for surface analysis. This instrument has a resolution of 4 mm at an accelerating voltage of 25 KV and ultimate vacuum of 10⁻⁷ torr. The specimen surface was made electrically conductive by coating a thin layer of gold using the plasma vapor deposition technique in a Fine Coat Ion Sputterer JF-1100.

RESULTS AND DISCUSSION

Poly(tetramethyldisilylene-co-styrene)

PTMDSS was synthesized by reacting DMDCS and styrene in a 2 : 1 mol ratio under dechlorination con-

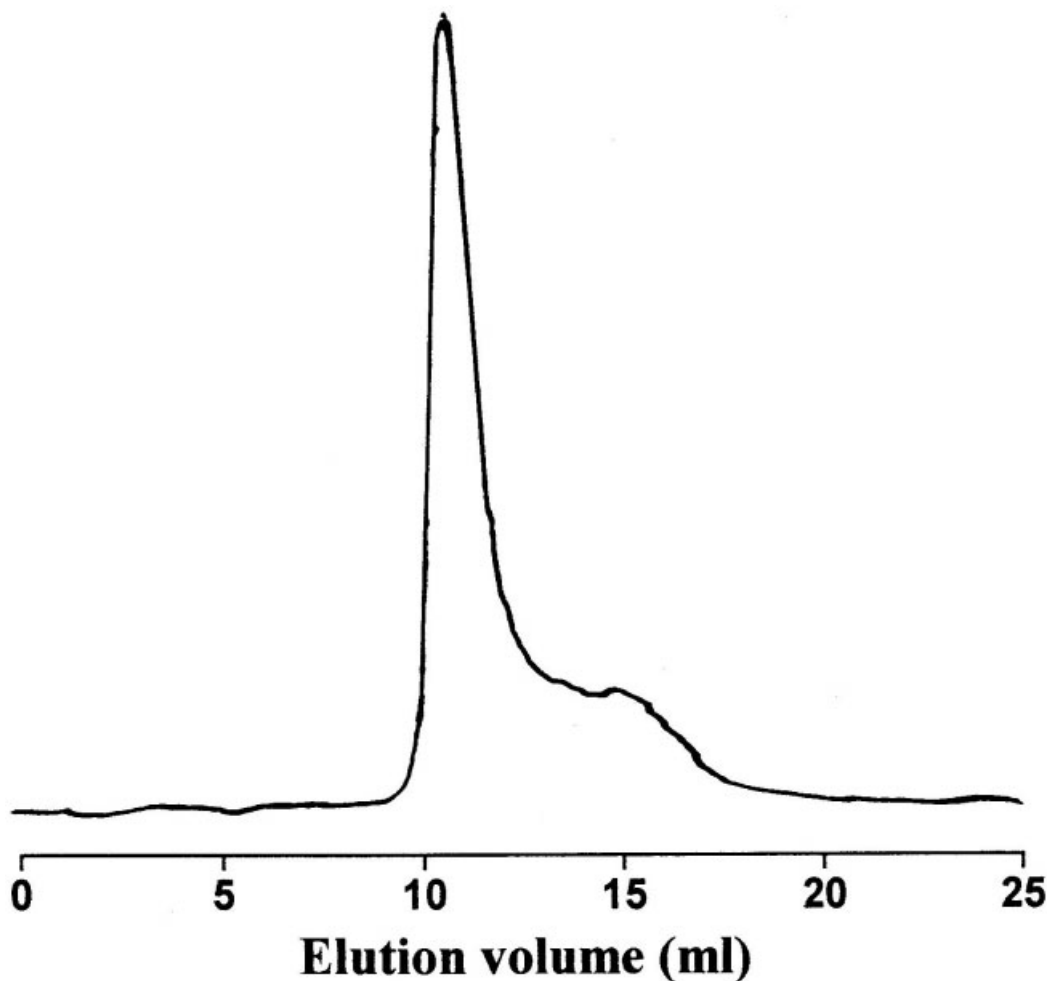


Figure 1 GPC curve of PTMDSS.

ditions as shown in Scheme 1. The copolymer is soluble in toluene, xylene, tetrahydrofuran, and chloroform. The copolymer is obtained in 60% yield. The copolymer was characterized by GPC, IR, and ^1H -, ^{13}C -, and ^{29}Si -NMR spectra. The GPC curve of the copolymer is given in Figure 1. The copolymer shows a bimodal molecular weight distribution, which is common with most of the polysilanes prepared by Wurtz-type polymerization.⁴⁴ The copolymer contains about 20 wt % of low molecular weight fraction. The high molecular weight fraction has M_w of 125,900 and M_n of 100,700 and the low molecular weight fraction has M_w of 6350 and 3300.

The composition of the copolymer as evaluated from ^1H -NMR spectrum on the basis of the relative intensities of the aromatic and aliphatic resonances is 0.5. The ^{29}Si -NMR spectrum of the copolymer obtained from DMDCS and styrene (1 : 0.5 mol ratio) under dechlorination conditions is shown in Figure 2. It shows signals at $\delta = -11.42$, -15.43 , and -15.77 ppm. Available literature data⁴⁵ suggest that substituents on the β -carbon affect the chemical shift of silicon

only marginally. Thus, two distinguishable Si atoms are present in the copolymers, namely, $-\text{Si}-\text{Si}-\text{CH}(\text{Ph})\text{CH}_2-$ and $-\text{Si}-\text{Si}-\text{CH}_2\text{CH}(\text{Ph})-$. Based on the ob-

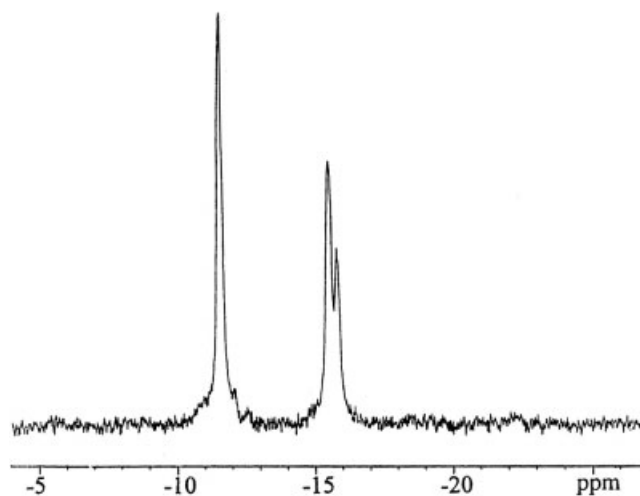


Figure 2 ^{29}Si -NMR spectrum of PTMDSS.

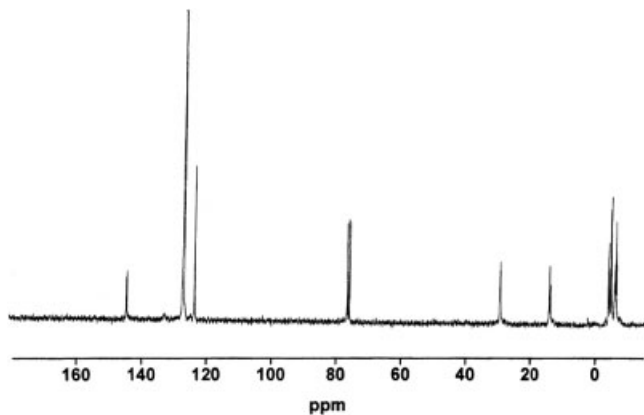
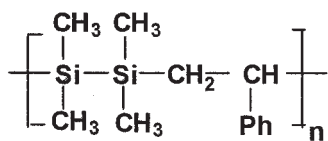


Figure 3 ¹³C-NMR spectrum of PTMDSS.

served deshielding of the phenyl substituent on α -carbon,⁴⁵ the resonance at $\delta = -11.42$ ppm was assigned to the $-\text{Si}-\text{Si}-\text{CH}(\text{Ph})-$ moiety and the resonances at $\delta = -15.43$ and -15.77 ppm were assigned to the $-\text{Si}-\text{Si}-\text{CH}_2-$ moiety. The multiplicity of signals in the up-field arises from the two possible stereochemical arrangements. A detailed account of the microstructure of PTMDSS is described elsewhere.⁶ In the ¹³C-NMR spectrum (Fig. 3) of the copolymer, methyl carbons show a large number of resonances in the region $\delta = -2$ to -6 ppm, and this can be attributed to the presence of different neighboring groups (CH and CH₂) as well as to the sensitivity of the stereochemical environment. Based on off-resonance decoupling experiments, the resonances at $\delta = 15.18$ and 30.4 ppm are assigned to methylene (SiCH₂) and methine (SiCH) carbons, respectively. The distinct feature of the spectrum in the aromatic region is the presence of a signal at $\delta = 125.64$ ppm. The structure of the copolymer as elucidated by ¹H-, ¹³C-, and ²⁹Si-NMR spectral studies is shown below.



AO exposure studies

The PTMDSS coated quartz plate, aluminized Kapton[®] film (125 μm) (coating applied on the unaluminized side), and C-polyimide composite were exposed to AO in a plasma barrel system along with the uncoated Kapton[®] film. As explained in the experimental section, the uncoated Kapton[®] film is used as the standard for calculating the AO fluence.

AO exposure of the PTMDSS coating on the quartz plate

Before using PTMDSS as an AO resistant coating, it is essential to find out if this polymer is resistant to AO.

For this purpose the polymer was coated on a quartz plate and exposed to AO. As quartz is resistant to AO attack, mass loss if any would be due to degradation of the polymer. The mass loss versus fluence data of the uncoated Kapton[®] film and the PTMDSS coating on quartz are compared in Figure 4. It is observed that the uncoated Kapton[®] film loses 6.370 mg/cm² on exposure to AO fluence of 21×10^{20} atoms/cm² whereas the PTMDSS coating on quartz loses only 0.03 mg/cm². This suggests that PTMDSS is resistant to AO attack, and the AO resistance of PTMDSS may be attributed to the presence of disilyl linkages. On exposure to AO, it is expected that insertion of oxygen to disilyl linkages would take place, resulting in the conversion of the disilyl linkage to a siloxane linkage. On further exposure to AO, the coating would undergo oxidation, resulting in the formation of a silica layer. The silica layer formed on the surface of the coating would prevent further attack of the coating by AO and thus protect the substrate from AO attack.

AO exposure of uncoated and PTMDSS coated aluminized Kapton[®]

High performance polymeric films are being extensively used in aerospace systems for various applications. Kapton[®], an organic polyimide film, is being used as a flexible substrate for light weight, high power solar arrays because of its inherent strength,

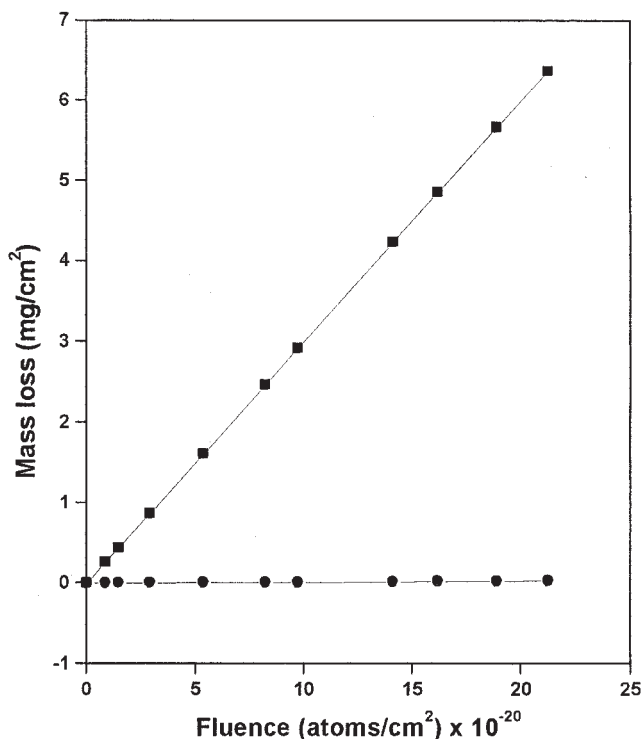


Figure 4 Mass loss versus atomic oxygen fluence for (■) Kapton[®] film and (●) PTMDSS.

temperature stability, excellent insulation properties, UV stability, IR transparency, radiation resistance, and good space heritage. Aluminized Kapton[®] is used as an external thermal control material on almost every satellite.⁴⁶ Here, aluminum in the back surface acts as a reflector and Kapton[®] is used to limit the emittance of the light. Although the Kapton[®] film performs fairly well under extreme conditions, its long-term stability in the LEO environment is a major concern. Hence, it is preferable to protect the Kapton[®] film from AO attack in LEO. As aluminum is resistant to AO attack, the unaluminized side of the films would be susceptible to AO attack. The unaluminized side of the aluminized Kapton[®] films were coated with PTMDSS.

Figure 5 presents the mass loss versus fluence for uncoated and PTMDSS coated aluminized Kapton[®]. It is observed that PTMDSS coated aluminized Kapton[®] loses 0.136 mg/cm² when exposed to an AO fluence of 2.1×10^{21} atoms/cm² whereas the uncoated aluminized Kapton[®] loses 6.350 mg/cm² when the samples were exposed to AO fluence of 1.6×10^{21} atoms/cm² and the uncoated sample was badly damaged. This suggests that PTMDSS offers good protection to Kapton[®] against AO attack. As PTMDSS is resistant to AO (as evidenced by the exposure studies of PTMDSS on the quartz plate), the mass loss of coated aluminized Kapton[®] probably occurs due to the attack of the film by AO through certain defect sites on the coating.

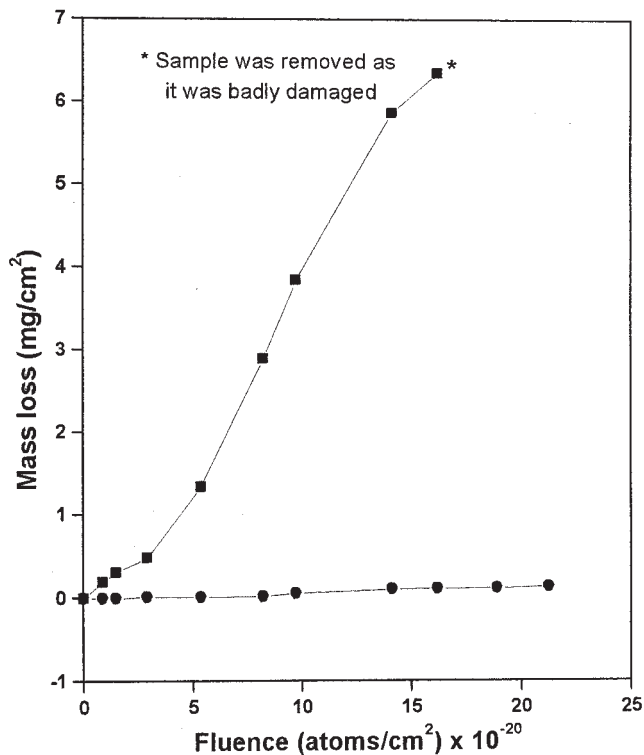
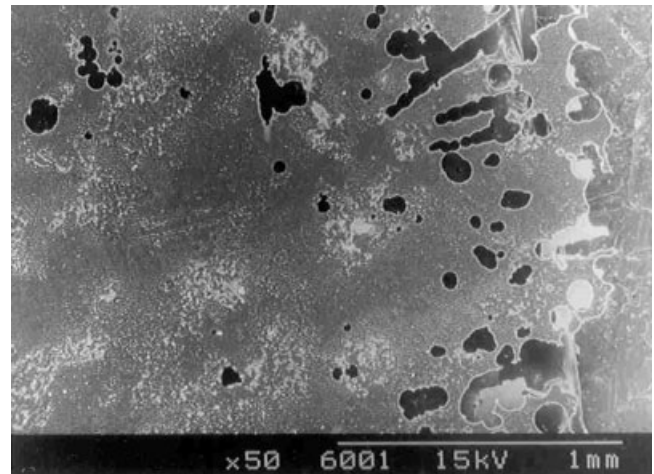
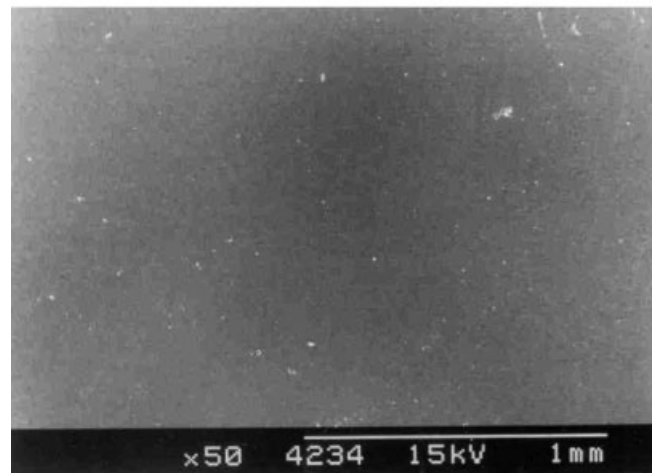


Figure 5 Mass loss versus atomic oxygen fluence for (■) uncoated and (●) PTMDSS coated aluminized Kapton[®] film.



(a)



(b)

Figure 6 SEM of (a) uncoated aluminized Kapton[®] film and (b) PTMDSS coated aluminized Kapton[®] film exposed to atomic oxygen.

Scanning electron micrographs of the uncoated and coated samples exposed to AO are given in Figure 6. It is observed that the uncoated sample is completely damaged due to erosion by AO whereas in the coated sample no such damage is observed.

AO exposure of uncoated and PTMDSS coated C-polyimide composite

Advanced composites are extensively used for spacecraft structural, power, and thermal subsystem applications.⁴⁷ Of the various composite materials, polymer matrix composites are preferred for spacecraft applications because of their combination of light weight, dimensional stability, high structural rigidity, and low thermal expansion.⁴⁷ In the fabrication of polymer matrix composites, a variety of epoxies, polyimides, polysulfones, and phenolics are used as matrix resins,

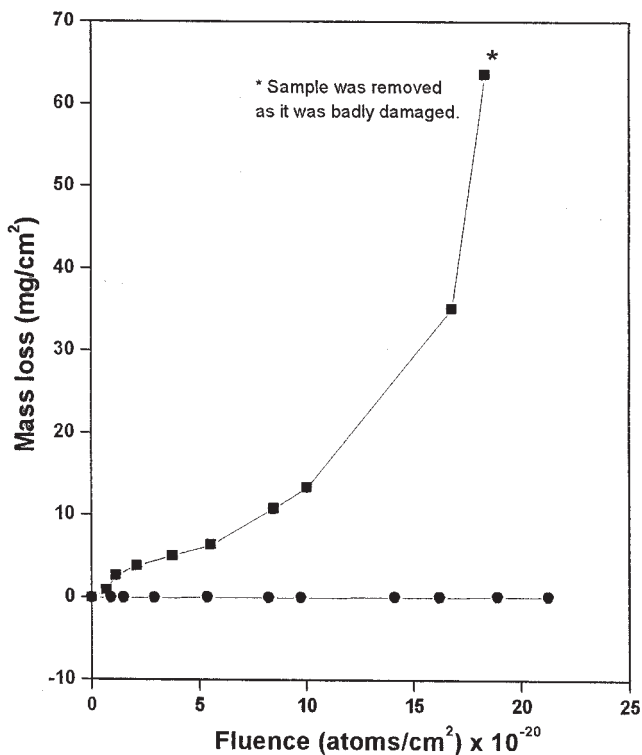


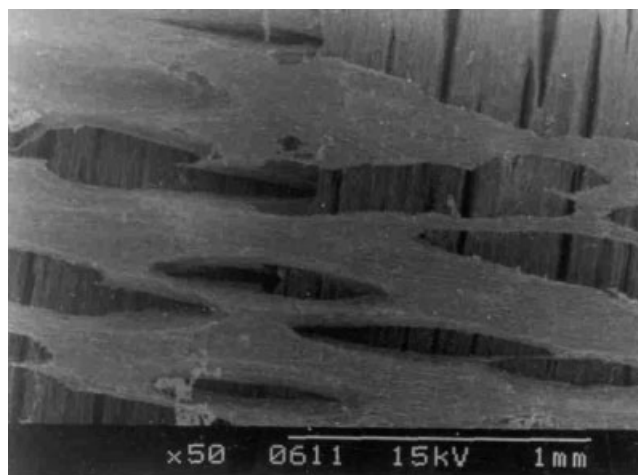
Figure 7 Mass loss versus atomic oxygen fluence for (■) uncoated and (●) PTMDSS coated C-polyimide composite.

and glass, aramid, graphite/carbon, and boron fibers are used as reinforcements. Typical applications of polymer composites for spacecraft structures include primary truss assemblies, core structures, boom, sandwich panels, bulkheads, and stiffeners. Types of payload advanced composites especially for dimensional stability include communication antenna reflectors, radio frequency components, optical telescope components, space-based radar, and precision mounting payload platforms and support structures. Rigid structural support for photovoltaic solar arrays and for solar dynamic power system concentrators also now utilize composites. Polymer matrix composites are susceptible to AO attack in the LEO environment. The high energy AO interacts with both matrix resin and reinforcements, resulting in the formation of volatile oxides on the surface, which leads to surface erosion. These effects are mainly responsible for reduction of mechanical strength, and changes in surface morphology and optical and thermal properties. Thus, polymer matrix composites need protection from AO attack. In the present study, PTMDSS has been evaluated as an AO resistant coating for C-polyimide composite.

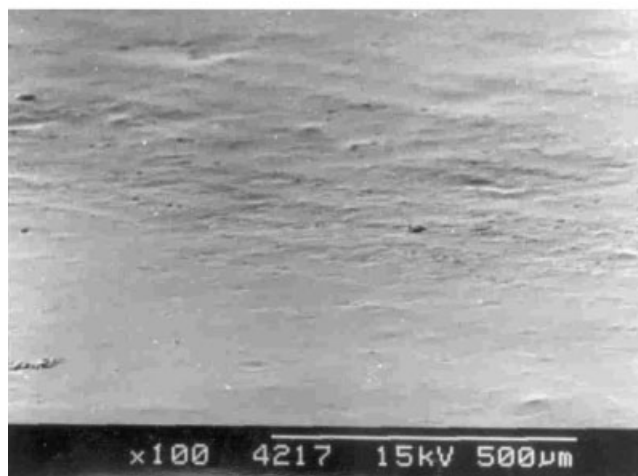
Figure 7 presents mass loss versus fluence of uncoated and PTMDSS coated C-polyimide composite. It is interesting to note that the uncoated composite undergoes mass loss of 63.64 mg/cm² on exposure to an AO fluence of 1.8 × 10²¹ atoms/cm² and the composite has to be removed from the plasma chamber as it got completely

damaged. On the other hand, the PTMDSS coated composite loses only 0.212 mg/cm² even after exposure to an AO fluence of 2.1 × 10²¹ atoms/cm².

The scanning electron micrographs of uncoated and coated C-polyimide composites exposed to AO are given in Figure 8. It is observed that in the case of the uncoated samples, the matrix resin is eaten away and the C-fabric is badly damaged. In contrast to the above observation, in the case of the coated sample no such damage is observed. The mass loss data and SEM studies suggest that the PTMDSS coating offers excellent protection to the C-polyimide composite against AO attack. SEM studies reveal the presence of cracks in certain areas of the coating on the C-polyimide composite through which AO could get access to the substrate causing erosion (Fig. 9).



(a)



(b)

Figure 8 SEM of (a) uncoated C-polyimide composite, and (b) PTMDSS coated C-polyimide composite exposed to atomic oxygen.

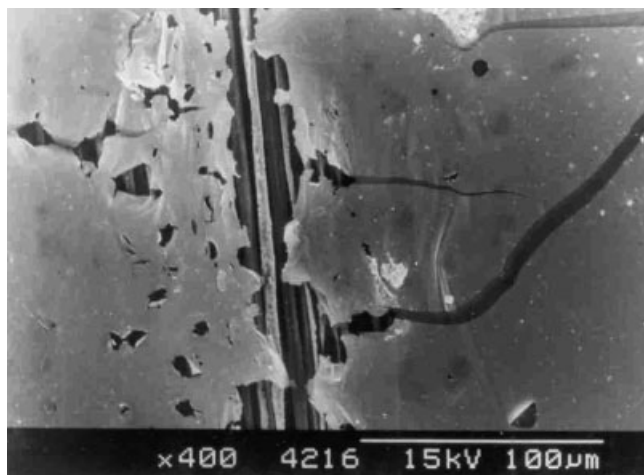


Figure 9 SEM of a defect site of PTMDSS coated C-polyimide composite exposed to AO.

CONCLUSION

The mass loss measurements of the PTMDSS coating on quartz (a substrate that is resistant to AO) on exposure to AO reveal that the polymer is stable towards AO attack. Hence, it was evaluated as an AO resistant coating for substrates such as Kapton[®] polyimide film and C-polyimide composite that are susceptible to AO attack in the LEO environment. The mass loss measurements at different AO fluence levels suggest that the uncoated samples undergo mass loss continuously, whereas only negligible mass loss is observed for the coated samples. SEM studies of the uncoated and PTMDSS coated samples exposed to AO suggest that PTMDSS offers very good protection to the substrates.

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