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**OPTICAL AND ELECTRICAL PROPERTIES OF  
ION BEAM TEXTURED KAPTON AND TEFLON**

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# OPTICAL AND ELECTRICAL PROPERTIES OF ION BEAM

## TEXTURED KAPTON AND TEFLON

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### ABSTRACT

An electron bombardment argon ion source was used to ion etch polyimide (Kapton) and fluorinated ethylene propylene, FEP (Teflon). Samples of polyimide and FEP were exposed to (0.5-1.0) keV Ar ions at ion current densities of (1.0-1.8) mA/cm<sup>2</sup> for various exposure times. Changes in the optical and electrical properties of the samples were used to characterize the exposure. Spectral reflectance and transmittance measurements were made between 0.33 and 2.16  $\mu$ m using an integrating sphere after each exposure. From these measurements, values of solar absorptance were obtained. Total emittance measurements were also recorded for some samples. Surface resistivity was used to determine changes in the electrical conductivity of the etched samples. A scanning electron microscope was used to record surface structure after exposure. Presented in the paper are spectral optical data, resistivity measurements, calculated absorptance and emittance measurements along with photomicrographs of the surface structure for the various exposures to Ar ions.

### INTRODUCTION

Polyimide (Kapton) and fluorinated ethylene propylene, FEP (Teflon), are two polymers that transmit visible radiation when used as thin sheets but have a very high value of resistivity ( $>10^{16}$  ohm/sq). These materials are presently being used in both ground and space applications. Their use in space as thermal control surfaces,<sup>1</sup> encapsulants for solar cells,<sup>2</sup> reflectors for concentrator arrays and solar sail material<sup>3</sup> introduces the problem of spacecraft charging. Electrical charge buildup on external spacecraft insulating surfaces can be dissipated by the use of thin conducting films on the insulator. However, the

application of thin conducting films can be costly, they alter the optical properties of the materials, add weight, and create new problems such as stability of the coating's optical properties when exposed to the space environment. An alternate approach is to ion beam texture such surfaces to modify the surface chemistry and morphology to obtain: (1) higher surface conductivities, (2) improved or unique optical properties, and (3) a textured substrate to enhance thin film adherence.

This paper describes the results of ion beam texturing polyimide and FEP by use of a 30-cm diameter electron bombardment argon ion source.<sup>4</sup> An evaluation of ion beam textured polyimide and FEP surfaces was performed for various beam energies, current densities, and exposure times. Measurements of the optical properties and sheet resistance were made after each exposure. It was hoped that polyimide and FEP would attain surface resistivities of  $<10^5$  ohms per square and that the optical properties would be altered to make the textured surfaces useful as radiative surfaces or as thermal control surfaces. The results of these studies are presented herein.

#### APPARATUS AND PROCEDURE

The ion source design, developed from electric propulsion technology,<sup>5</sup> uses a hollow cathode to provide the ionizing current for the beam source. Beam extraction is accomplished by a dished, two grid ion optics system.<sup>4</sup> Neutralization of the ion beam was achieved by secondary electrons released by ion bombardment of the vacuum facility walls.

The vacuum facility 1.5 meters in diameter and 7.3 meters long is sufficiently large to minimize backspattered facility material from contaminating the experiments and maintained a pressure of  $4 \times 10^{-5}$  torr during ion source operation.

The ion source is capable of operating at beam energies between 500 and 1500 eV. The beam current can be adjusted between 200 mA and 2.0 A. The samples ( $8 \times 8$  cm) to be textured were located normal to the beam at a location 20 cm from the ion source. The current density at this location was  $1.8 \text{ mA/cm}^2$ .

for polyimide targets,  $1 \text{ mA/cm}^2$  for FEP targets and was uniform over the target area. Target texture was simply produced by ion beam etching for exposure times varying in duration from 10 to 115 minutes.

The polymers picked to be textured include: 8- $\mu\text{m}$  thick polyimide (Kapton); aluminized polyimide (Kapton), 127- $\mu\text{m}$  thick polyimide coated on one side with 0.1  $\mu\text{m}$  Al to obtain a high solar reflectivity; and silvered fluorinated ethylene propylene, FEP (Teflon), 127  $\mu\text{m}$  thick coated with 0.15  $\mu\text{m}$  of silver and backed with two layers of polyimide tape.

Spectral transmittance  $[\tau(\lambda)]$  and reflectance  $[\rho(\lambda)]$  measurements were made between 0.33 and 2.16  $\mu\text{m}$  using the integrating sphere described in reference 6. The samples were measured before and after a given exposure to the ion beam. The technique used to obtain the data was to break up the solar energy distribution curve at air mass zero into 2 percent energy increments.<sup>6</sup> Total solar reflectance at air mass zero,  $\rho_s$ , is defined as the solar energy (specular and diffuse components) reflected by a surface, divided by the total energy incident in the surface. A similar definition and technique was used to obtain the total transmittance ( $\tau_s$ ) of a sample. Once  $\tau_s$  and  $\rho_s$  are measured the corresponding total solar absorptance ( $\alpha_s$ ) at air mass zero can be calculated by use of equation (1)

$$\alpha_s = 1 - \rho_s - \tau_s \quad (1)$$

For an opaque sample, a measurement of the solar reflectance,  $\rho_s$ , subtracted from one will yield the solar absorptance.

The technique used in the integrating sphere to obtain  $\rho(\lambda)$  for a transparent material was to first measure  $\tau(\lambda)$  and then make a measurement of  $[\tau(\lambda) + \rho(\lambda)]$ . This method is described in reference 6 and can be used to obtain values of solar absorptance ( $\alpha_s$ ).

Values of the spectral reflectance  $\rho(\lambda)$  of the opaque materials in the infrared region (1 - 15.5)  $\mu\text{m}$  were obtained using a heated blackbody (hohlraum) reflectometer that is described in reference 7.



The sheet resistance of the samples were measured by placing the samples in a holder between two strips of indium. Using the dimensions of the sample, values of sheet resistance expressed in ohms per square (ohm/sq) were obtained.

## RESULTS AND DISCUSSION

### Polyimide

Uncoated polyimide 8  $\mu\text{m}$  thick was the first sample exposed to the Ar ion beam. By varying the beam energy, current density, and sample position, it was demonstrated that the polyimide samples could be ion beam textured at a beam energy of 1 keV and a current density of 1.8 mA/cm<sup>2</sup>. This beam intensity was sufficient to texture the polyimide surface in a reasonable time and was of low enough beam power density so that the polyimide samples maintained their physical integrity. Table I presents the results of exposing polyimide samples for various times, which varied from 10 to 115 minutes. The values presented are the total transmittance, total reflectance, total solar absorptance and sheet resistance for each sample after each texture exposure time.

The resulting textured surfaces were examined using a scanning electron microscope. Figures 1(a), (b), and (c) show the surfaces after ion beam texturing for 10, 30, and 115 minutes. Increasing the exposure time causes changes in the surface morphology. At 115 minutes spires 5  $\mu\text{m}$  high appear in the surface.

Figure 2 shows the effects of the exposure to 1 keV Ar ions on the spectral transmittance. Texturing polyimide for only 10 minutes causes a large decrease in spectral transmittance at all wavelengths, the largest reductions taking place below 1.0  $\mu\text{m}$ . Increasing the exposure time up to 60 minutes continues to decrease the transmittance at all wavelengths. The 115-minute exposure caused the already thin polyimide to become thinner, hence an increase in the spectral transmittance was observed. Complete penetration of the polyimide occurs after ion etching for a period of approximately 180 minutes.

Listed in table I and plotted in figure 3 are the total solar transmittance, solar absorptance, solar reflectance, and the sheet resistance for a given ion

beam exposure time. The minimum sheet resistance and lowest value of total transmittance occurred when the polyimide was exposed to the beam for 60 minutes. A sheet resistance of 10 000 ohms per square, desirable for space applications, was attained after a 30-minute ion beam exposure. The decrease in sheet resistance from  $>10^7$  to 10 000 ohms per square was possibly due to a chemical change in the surface structure of the polyimide, since other measurements rule out any contamination effects of the tank walls or ion source. It is felt that because of the chemical nature of the polyimide molecules (strong intermolecular and weak intramolecular bonding), ion beam texturing of the surface may have released some carbon atoms, thus causing a conductivity.

As shown in figure 3, the reduction in total transmittance due to beam exposure time was accompanied by an increase in solar absorptance with very little changes in the initially small reflectance. The 115-minute ion beam exposure time caused a reversal in the trend of all the measured parameters and was essentially too long an exposure for this thin a sheet of polyimide. The significant increase in sheet resistance at the 115-minute exposure might possibly be due to the very rough surface structure or partial sputter through in the valleys of microstructures. No attempt was made to measure the infrared reflectance using the hohlraum reflectometer for the transparent polyimide.

#### Aluminized Polyimide

After successfully texturing plain 8  $\mu\text{m}$  polyimide, aluminized polyimide topologically representative of a solar reflector or a solar sail was exposed on the polyimide side to 1 keV, 1.8 mA/cm<sup>2</sup> Ar beam ion. Figure 4 shows a scanning electron photomicrograph of the polyimide surface after 10 minutes of texturing by the ion beam. The surface morphology is the same as for the 8  $\mu\text{m}$  polyimide after a 10-minute exposure under similar conditions (see fig. 1(a)). Shown in figure 5 is the spectral reflectance between 0.33 and 15.5  $\mu\text{m}$  of the aluminized polyimide surface for untextured, 10 and 31 minutes of texturing. Ten minutes of texturing is enough time to cause large reductions in the spectral reflectance below 2  $\mu\text{m}$ . Increasing the exposure time to 31 minutes, yields

further, but smaller reductions. At wavelengths greater than  $2\text{ }\mu\text{m}$  the changes in reflectance are wavelength and exposure dependent with no clear trends. Figure 6 is a plot of solar reflectance, solar absorptance, sheet resistance, and thermal emittance calculated at 550 K for the textured aluminized polyimide surfaces. This figure shows that the large changes in these parameters take place after 10 minutes of texturing. A sheet resistance of 10 000 ohms per square is reached in only 10 minutes of texturing. This value of sheet resistance is attained in a shorter period of time than for the  $8\text{ }\mu\text{m}$  polyimide (30 min, see fig. 3). The SEM photomicrograph of the 10-minute textured surfaces of both samples look alike, and even though the absolute sheet resistances are different, significant conductivity was achieved. Use of aluminized polyimide as a solar reflector in space missions at 0.3 AU (10 suns) requires a thermal emittance of at least 0.40 to keep the aluminized polyimide at a safe temperature level, 550 K. By ion beam texturing the Kapton surface for 31 minutes a thermal emittance of 0.765 was attained at this temperature. If 550 K is a safe upper limit operating temperature for the aluminized polyimide than with an emittance of 0.765 on the textured polyimide side the material could tolerate operation at 0.20 AU (25 suns). An additional benefit of using the textured material would be a surface resistivity of 6000 ohms per square.

#### Silvered FEP

127- $\mu\text{m}$  thick FEP (Teflon) coated on the back side with  $6.15\text{ }\mu\text{m}$  Ag was exposed on the FEP side to the Ar ion beam. This coated FEP is a thermal control surface for spacecraft and has proper optical properties, but does not have a surface conductivity necessary to reduce the effects of spacecraft charging. Attempts to texture FEP at the same energy level and beam current density as polyimide caused thermal decomposition of the FEP. A reduced beam current density of  $1\text{ mA/cm}^2$  and a beam energy of 0.5 keV was found to be more appropriate to ion beam texture FEP with Ar ions. Shown in figure 7 is a scanning electron photomicrograph taken at  $\times 3000$  magnification of the ion beam textured FEP after a 27-minute exposure. Its surface morphology is



unlike that of the polyimide but consistent with results obtained in references 8 and 9. Sheet resistance measurements indicated that ion beam texturing the FEP surface did not decrease the surface resistivity (still  $>10^7$  ohm/sq). Figure 8 presents the spectral reflectance of untextured and textured FEP. At wavelengths below  $4\text{ }\mu\text{m}$  there is a small reduction in the spectral reflectance. No significant trend takes place at wavelengths greater than  $4\text{ }\mu\text{m}$ . The solar absorptance changed from 0.087 to 0.122 as shown in table I. The thermal emittance calculated at 300 K (nominal operating temperature of this thermal control coating on a spacecraft) changed from 0.807 to 0.670. This leads to a higher  $\alpha/\epsilon$  ratio, thus increasing the surface temperature. Although the ion beam texturing of FEP does not lower the ratio of  $\alpha/\epsilon$ , or produce a conducting surface, the surface texturing may allow better adherence of metallic films.

Metalizing the textured FEP surface with gold produces a relatively efficient solar absorber (fig. 8, table I). This type of surface, with further optimization, might find terrestrial application for heating and cooling systems. For example, this FEP sheet might be ion beam textured and metalized with aluminum to produce a solar selective surface. FEP may be ion machined at rates up to  $200\text{ }\mu\text{m/hr}$  and ion beam processing may be cost effective relative to electroplated systems. The easily textured FEP surfaces appear matt white. Subsequent sputter deposition of the gold film results in a microscopically rough metal film having a grey-black matt appearance similar to textured bulk metals.<sup>10</sup> However the cones are far more elastic and mechanical damage resistant than cones in metal substrates. Casual handling of the surface did not appear to alter its optical appearance.

#### CONCLUDING REMARKS

Polyimide (Kapton) and FEP (Teflon) can be textured using an electron-bombardment Argon ion source at low enough power levels so as to not cause thermal changes in their physical properties.

Polyimide develops a conducting surface when ion beam textured and attains a surface resistivity of 10 000 ohms per square with a 10-minute expo-

sure. Ion beam texturing of the Kapton caused large changes in the transmittance and solar absorptance, but only slight changes in reflectance.

The aluminized polyimide surface (solar reflector material) when exposed for 31 minutes to 1 keV Ar ions attained a thermal emittance at 550 K of 0.765. This emittance allows the solar reflector material a capability of operating at 0.20 AU (25 suns). Ion beam texturing of the polyimide eliminates the need of coating the Kapton with a high emittance, conductive material. Texturing of the polyimide side of reflector material also eliminates the weight penalty associated with a thin film coating.

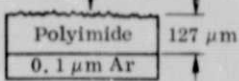
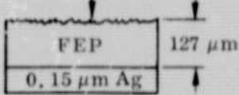
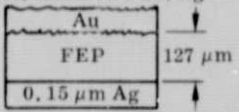
FEP does not become conducting when textured; however, the surface texturing may allow better adherence of subsequent sputtered metallic films, thus resulting in a high absorptance surface.

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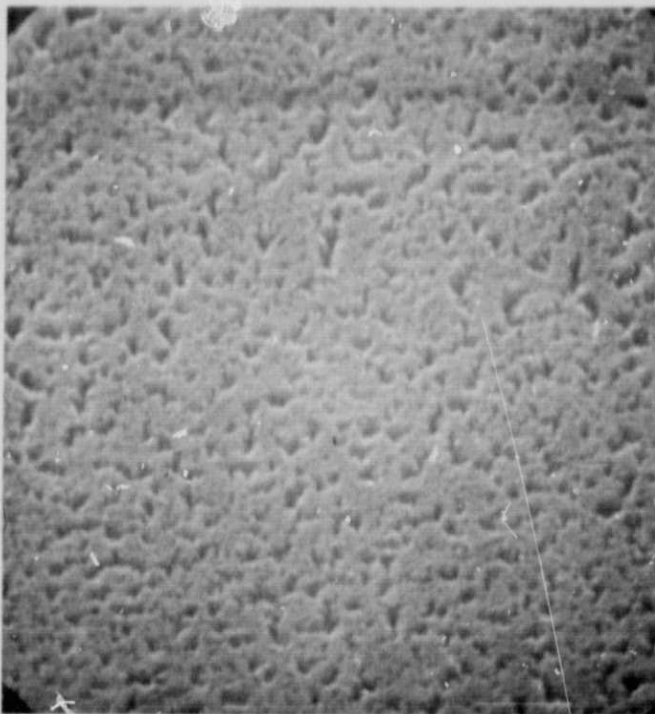
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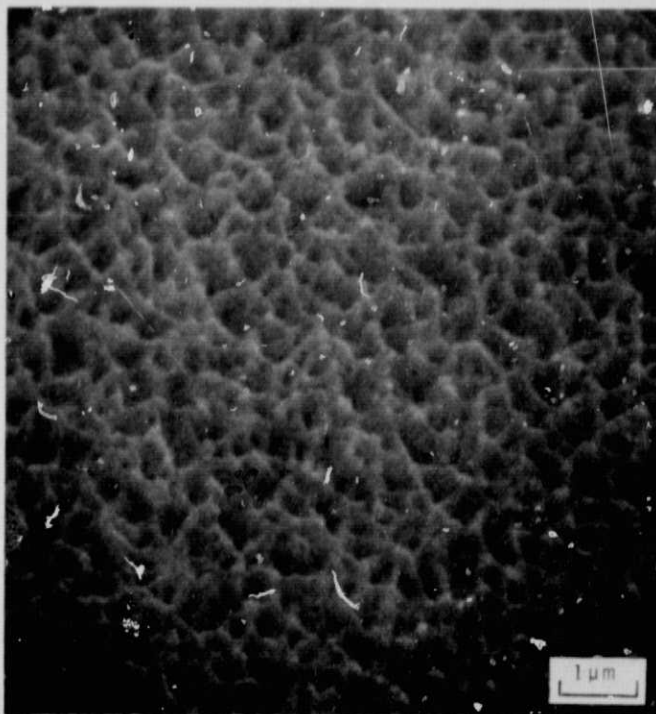
TABLE I - OPTICAL PROPERTIES AND SHEET RESISTANCE OF ION TEXTURED SURFACES

Sample	Texture exposure time, min	Total solar transmittance, $\tau_s$	Total solar reflectance, $\rho_s$	Total solar absorptance	Total infrared reflectance, $\rho$	Thermal emittance, $\epsilon_T$	Sheet resistance of polymer surface, ohm/sq
Polyimide (8 $\mu\text{m}$ )	Untextured	0.724	0.102	0.174			$\left( \begin{smallmatrix} >10^7 \\ a_{10^{18}} \end{smallmatrix} \right)$
	10	.324	.016	.660			18 000
	30	.224	.006	.770			10 000
	60	.193	.017	.790			6 000
	115	.217	.003	.780			21 000
Aluminized polyimide (exposed surface)	Untextured	-----	0.501	0.499	At 550 K 0.262	At 550 K 0.738	$\left( \begin{smallmatrix} >10^7 \\ a_{10^{18}} \end{smallmatrix} \right)$
	10	-----	.106	.894	.329	.671	10 000
	31	-----	.677	.923	.235	.765	6 000
Silvered FEP (exposed surface)	Untextured	-----	0.913	0.087	At 300 K 0.193	At 300 K 0.807	$\left( \begin{smallmatrix} 15^7 \\ a_{10^{16}} \end{smallmatrix} \right)$
	27	-----	.878	.122	.330	.670	$>10^7$
Au/textured FEP/Ag	27	-----	0.118	0.882	0.292	0.708	-----
							

<sup>a</sup>Values found in the literature.ORIGINAL PAGE IS  
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(a) 10 MINUTE EXPOSURE.

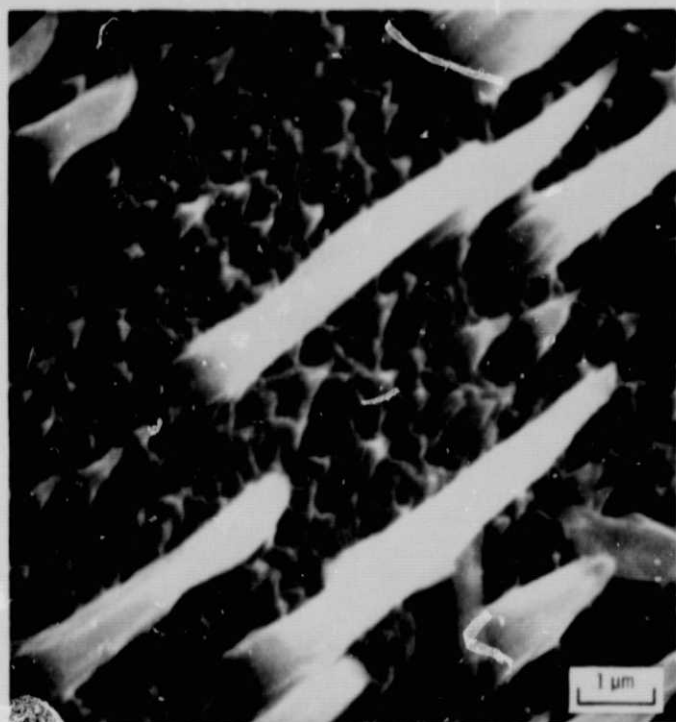


(b) 30 MINUTE EXPOSURE.

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Figure 1. - Scanning electron photomicrographs of ion beam textured 8  $\mu\text{m}$  thick polyimide exposed to 1 KeV argon ions at 1.8  $\text{ma}/\text{cm}^2$ ; 35° tilt. X10 000.





(c) 115 MINUTE EXPOSURE.

Figure 1. - Concluded.

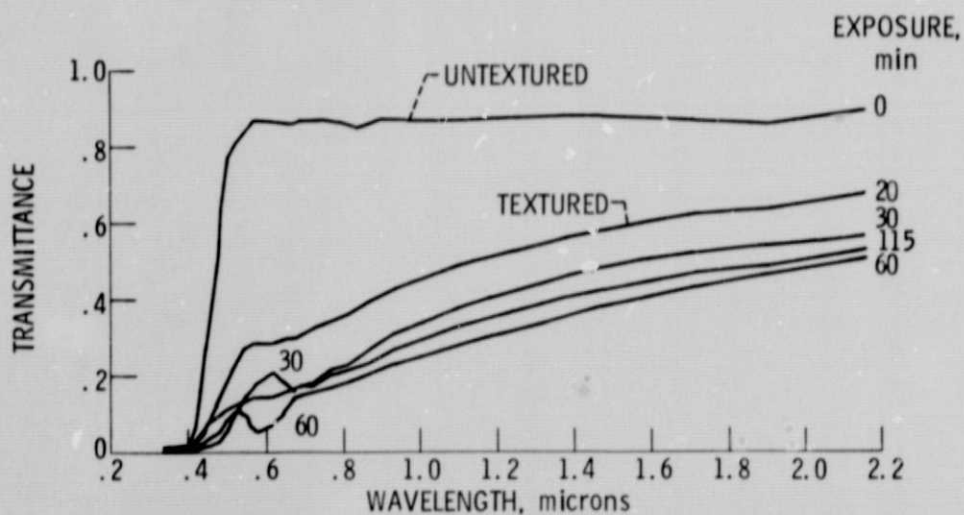


Figure 2. - Spectral transmittance of 8  $\mu$ m thick polyimide before and after exposure to 1 keV Ar ions at 1.8 mA/cm<sup>2</sup>.

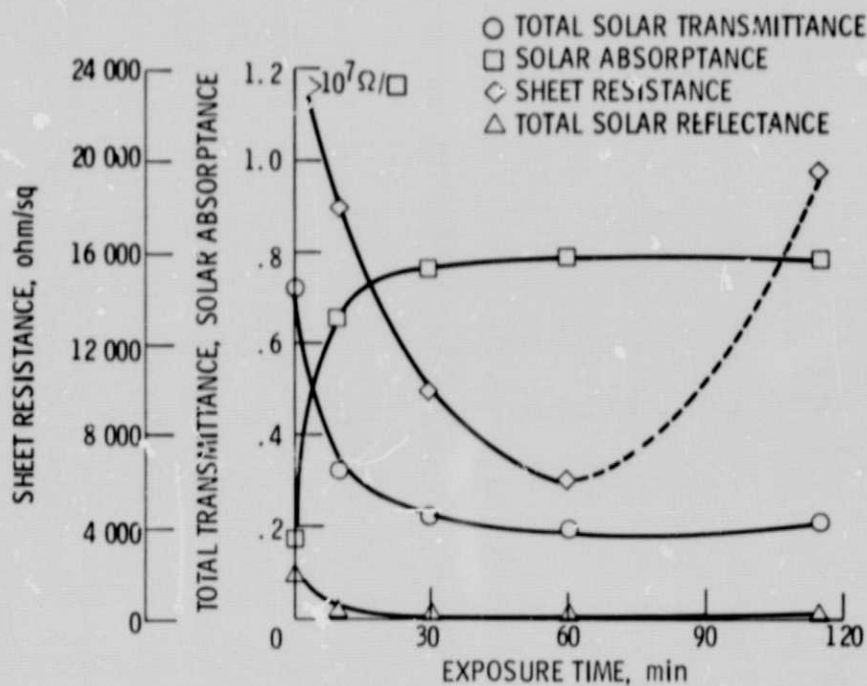


Figure 3 - Total transmittance, solar absorptance, and sheet resistance versus exposure time for 8  $\mu\text{m}$  polyimide exposed to 1 keV, 1.8 mA/cm<sup>2</sup> Ar ions.

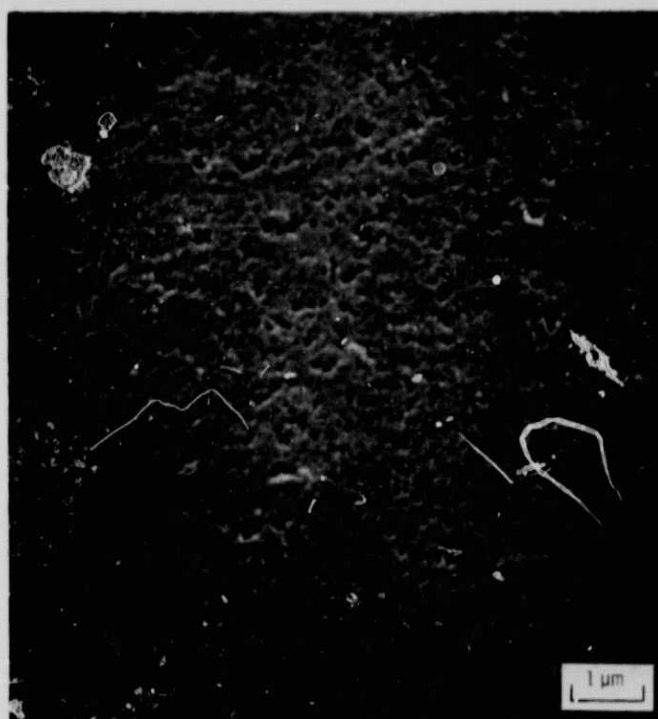


Figure 4. - Scanning electron photomicrograph of ion beam textured 127  $\mu\text{m}$  aluminized polyimide exposed to 1 KeV argon ions at 1.8 ma/cm<sup>2</sup> for 10 minutes. X10 000.

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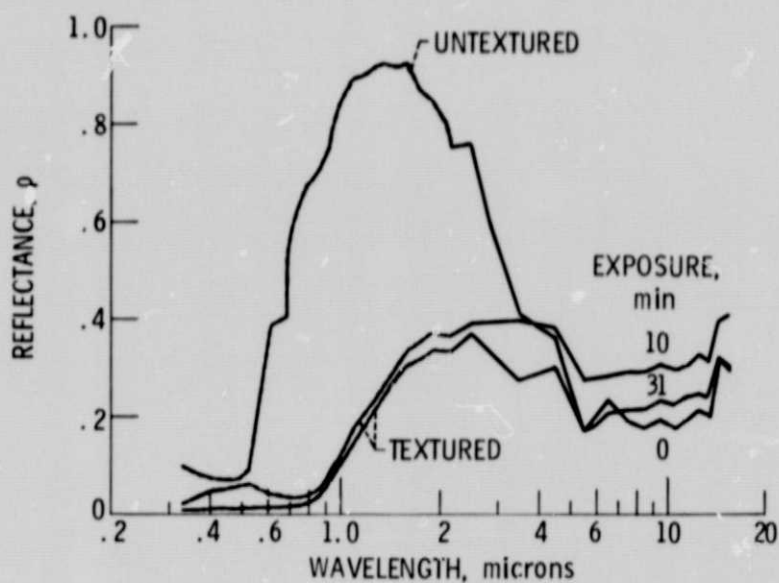


Figure 5. - Spectral reflectance of 127  $\mu\text{m}$  aluminized polyimide exposed to 1 keV Ar ions.

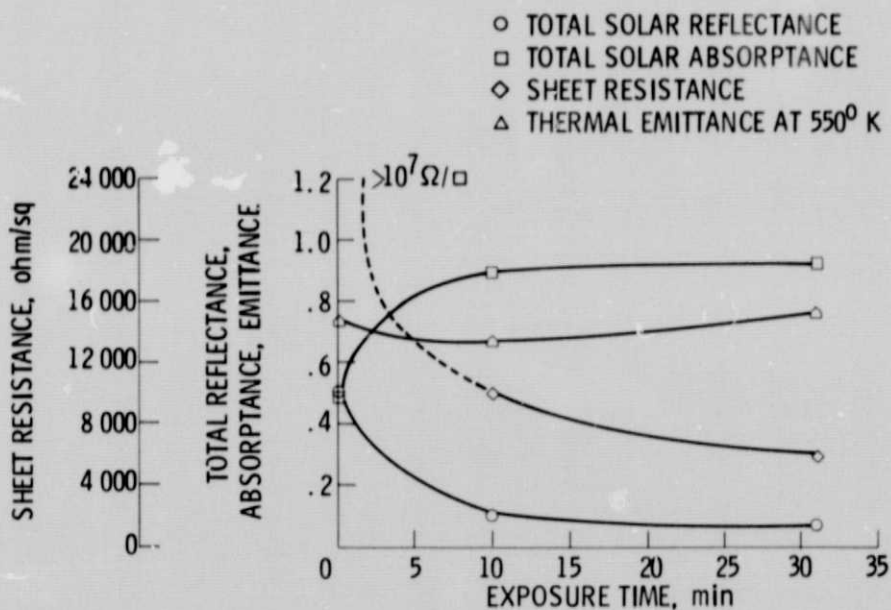


Figure 6. - Solar absorptance, emittance, solar reflectance and sheet resistance versus exposure time for 127  $\mu\text{m}$  aluminized polyimide, exposed to 1 keV, 1.8 mA/cm<sup>2</sup> Ar ions.

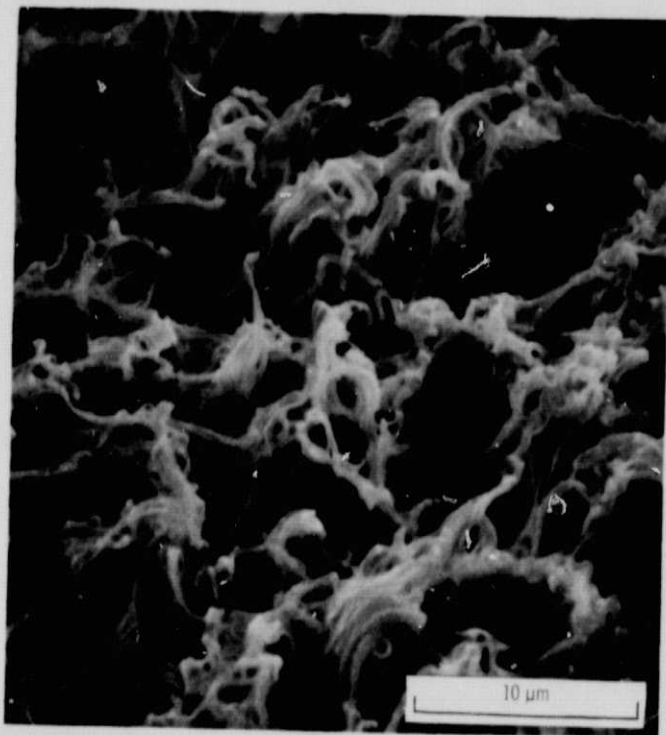
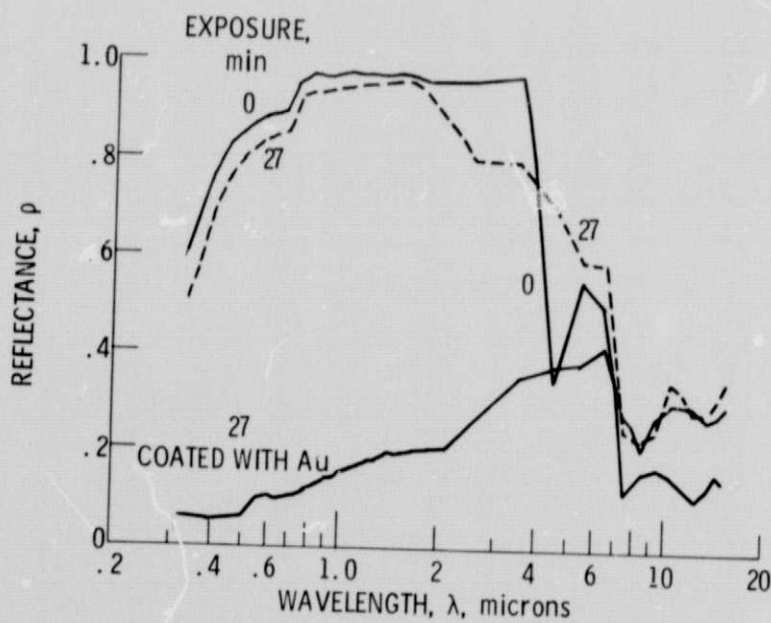


Figure 7. - Scanning electron photomicrograph of ion beam textured FEP exposed to 0.5 KeV argon ions at 1 ma/cm<sup>2</sup>; 40° tilt. X3000.



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Figure 8. - Spectral reflectance of 127 μm silvered FEP.