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# Atomic Oxygen Textured Polymers

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# ATOMIC OXYGEN TEXTURED POLYMERS

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## ABSTRACT

Atomic oxygen can be used to microscopically alter the surface morphology of polymeric materials in space or in ground laboratory facilities. For polymeric materials whose sole oxidation products are volatile species, directed atomic oxygen reactions produce surfaces of microscopic cones. However, isotropic atomic oxygen exposure results in polymer surfaces covered with lower aspect ratio sharp-edged craters. Isotropic atomic oxygen plasma exposure of polymers typically causes a significant decrease in water contact angle as well as altered coefficient of static friction. Such surface alterations may be of benefit for industrial and biomedical applications. The results of atomic oxygen plasma exposure of thirty-three (33) different polymers are presented, including typical morphology changes, effects on water contact angle, and coefficient of static friction.

## INTRODUCTION

Early Space Shuttle missions provided the opportunity to observe results of in-space atomic oxygen attack on hydrocarbon polymer materials.<sup>(Ref.1)</sup> Directed atomic oxygen in space has been shown to produce microscopic cone-like surface textures on all materials whose sole oxidation products are volatile species.<sup>(Ref.2)</sup> Figure 1 shows a typical high aspect ratio cone-like surface morphology resulting from high fluence directed atomic oxygen exposure in space for chlorotrifluoroethylene exposed to directed atomic oxygen on the Long Duration Exposure Facility. Similar morphology polymer surfaces have been produced in ground laboratory atomic oxygen exposure facilities using directed beams of either thermal energy (<0.1 eV) or several eV atomic oxygen. Such surfaces result in significant reductions in specular reflectance with an accompanying increase in diffuse reflectance.

Although directed atomic oxygen exposure of polymers tends to produce high aspect ratio surface microstructures, isotropic arrival resulting from oxygen plasma exposure of polymers also produces surface micro-roughness but at a lower aspect ratio level. Such plasmas can be created by using radio frequency discharges at low gas pressures (typically the order of  $10^{-1}$  torr) of oxygen, air or other reactive species. Low pressure RF plasma ashers which operate at 13.56 MHz have been used for many years for determination of the ash content of foods and the

removal of photoresist from semiconductor wafers <sup>(Ref. 3-5)</sup>. RF plasma ashers used to produce atomic oxygen are relatively inexpensive and effective in producing surfaces whose optical, tribological, water-contact angle and bonding properties can be altered for potential industrial and biomedical applications similar to surfaces which are microscopically textured by ion beam sputtering. <sup>(Ref.6,7)</sup>

The objective of the research presented in this paper was to characterize the effects of isotropic atomic oxygen plasma exposure to polymers in terms of surface morphology, water-contact angle, and coefficient of static friction for a wide variety of polymeric materials typically used in industry and biomedical applications.

## APPARATUS AND PROCEDURE

The RF plasma ashers used for this investigation were 100 watt, 13.56 MHz SPI RF plasma ashers with a cylindrical exposure chamber operated on air at  $\sim 100$  m torr (Fig. 2).

To enable a comparison between pristine and plasma exposed polymers it was necessary to determine the amount of plasma exposure used so all materials would be documented at the same atomic oxygen fluence. For comparison purposes, atomic oxygen fluence was measured using polyimide Kapton witness coupons to calculate Kapton effective fluences based on an erosion yield of  $3.0 \times 10^{-24}$  cm<sup>3</sup>/atom for polyimide Kapton. The polyimide Kapton atomic oxygen fluence measurement technique was used because it has been accepted as a standardized recommended practice for asher plasma exposure of polymers <sup>(Ref. 8)</sup>.

The static coefficient of friction between atomic oxygen textured polymers sliding against identical atomic oxygen textured polymers as well as against polished 300 series stainless steel was measured using an inclined plane. The plane was slowly elevated to measure the angle at which sliding initiated. The static coefficient of friction was then computed as the tangent of the angle of inclination when sliding initiated. The coefficient of static friction was characterized for natural rubber and cellulose acetate to determine the level of fluence to be selected for treated surfaces. These two materials were chosen to represent both low modulus and high modulus polymers.

The water contact angle on atomic oxygen textured polymers was measured using a Contact Angle Measuring System Model G1, manufactured by Kernco Instruments. Distilled water droplets were used for these tests. To assess the atomic oxygen fluence necessary to produce a water contact angle which was unchanging with further atomic oxygen exposure, plots of water contact angle as a function of atomic oxygen fluence were produced for polyimide Kapton, polystyrene and natural rubber to consider a range of elastic moduli to be considered.

## RESULTS AND DISCUSSION

Based on the results of atomic oxygen exposure of a wide variety of polymers it appears that all polymers whose reaction products produce solely volatile species result in microscopic crater-like surfaces whose ridges and associated chemistry play an important role in tribological and wetting properties.

## Morphology Changes

Exposure of carbon-based polymers to isotropic thermal energy atomic oxygen resulted in microscopic cratering of the polymer surfaces as shown in Figures 3 through 5. As can be seen, the sharp edged craters can be easily misinterpreted as microscopic mounds. However, detailed microscopy examinations have validated that the surface is, in fact, crater-like. The surface morphology which developed for silicone polymers was markedly different because not all oxidation products are volatile. This results in an oxidized silicone surface which is essentially a silica surface. The silica surface may or may not be cracked depending on the specific silicone and degree of atomic oxygen exposure. (Ref. 9). It appeared that all materials whose oxidation products were solely volatile species develop microscopic crater-like surfaces upon isotropic atomic oxygen plasma exposure. Such cratered surfaces should have altered properties in terms of static and dynamic friction, wetting properties and bonding properties.

## Coefficient of Static Friction

The coefficient of static friction of atomic oxygen textured polymer surfaces sliding on identical atomic oxygen textured surfaces was in general found to decrease with increasing atomic oxygen fluence for low modulus polymers but increased for higher modulus polymers. As can be seen in Figure 6, the coefficient of friction of natural rubber decreased by 42% upon exposure to the Kapton effective atomic oxygen fluence of  $10^{21}$  atoms/cm<sup>2</sup>, whereas for cellulose acetate the coefficient of static friction increased by 290% for the same fluence.

The results also indicate that very little change in coefficient of static friction occurs after the surface texture is exposed to a fluence of  $10^{21}$  atoms/cm<sup>2</sup>. For this reason, Kapton effective atomic oxygen fluence of  $10^{21}$  atoms/cm<sup>2</sup> was selected for the exposure of a wide variety of polymeric materials as shown in Table I. As shown in Table I, all polymers increased in static coefficient of friction as a result of exposure to atomic oxygen fluence of  $10^{21}$  atoms/cm<sup>2</sup>, except for polymers which had a high pristine coefficient of friction as a result of static adhesion, low plastic modulus polymers (natural rubber and cast urethane with shore hardness of 60), and silicones. It is believed that the reduction in coefficient of friction for silicone rubber as a result of atomic oxygen exposure is due to surface conversion to silica, which has a lower coefficient of friction. In addition, atomic oxygen exposed silicones tend to develop a flat but cracked silica surface rather than rough crater rims which are left-standing for hydrocarbon polymers.

The coefficient of static friction for polished 300 series stainless steel on pristine and atomic oxygen exposed polymer surfaces is shown in Table II. As can be seen in Table II, the coefficient of static friction of polished stainless steel on polymer surfaces is in general slightly increased for most, but not all, polymers as a result of atomic oxygen exposure.

## Water Contact Angle

The effect of atomic oxygen plasma exposure on water contact angle for various polymers was examined as a function of fluence as shown in Figure 7 for polyimide Kapton, polystyrene and natural rubber. As can be seen in Figure 7, a significant reduction in water contact angle occurs even for atomic oxygen fluences below  $10^{19}$  atoms/cm<sup>2</sup>. For atomic oxygen fluences greater than  $10^{20}$  atoms/cm<sup>2</sup> relatively little further changes in contact angle occur. For this reason an effective atomic oxygen fluence of  $10^{20}$  was chosen to assess the effect of water contact

angle between pristine and atomic oxygen exposed polymers. The results of this investigation are shown in Table III. As can be seen from Table III, all polymers with the exception of FEP Teflon showed reduction in water contact angle indicating a tendency to wet as a result of atomic oxygen exposure. Many polymers exhibited wetting or near wetting behavior as a result of exposure. The increased wetting characteristics may be a result of morphology changes as well as incorporation of oxygen functionalities on the surfaces of the exposed polymers.

## SUMMARY

The effect of thermal energy ( $<0.1$  eV) isotropic atomic oxygen arrival on surfaces was investigated for a wide variety of polymeric materials. The resulting changes in coefficient of static friction (for like-on-like and smooth stainless steel) and water contact angle were measured. Coefficient of friction of atomic oxygen exposed materials was found to slightly increase for high modulus materials and decrease for elastomeric materials. Changes in coefficient of friction appeared to maximize once a Kapton effective atomic oxygen fluence of  $10^{21}$  atoms/cm<sup>2</sup> was reached. The water contact angle for most polymers was found to significantly decrease as a function of atomic oxygen fluence. Although significant reductions in contact angle occurred for only  $10^{19}$  atoms/cm<sup>2</sup> fluence, relatively little change in water contact angle occurred at atomic oxygen fluences greater than  $10^{20}$  atoms/cm<sup>2</sup>. All polymers, with the exception of FEP Teflon showed small to significant reductions in contact angle indicating significant increases in wetting as a result of atomic oxygen exposure.

## ACKNOWLEDGEMENTS

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TABLE I. Coefficient of static friction between identical surfaces of pristine and atomic oxygen textured polymeric materials

MATERIAL & SUPPLIER WHEN KNOWN	UNTREATED	ATOMIC OXYGEN EXPOSED*
ABS [Styrene-acrylonitrile matrix and styrene-butadiene rubber]	.50 ± .03	.73 ± .01
Cellulose Acetate	.47 ± .03	.57 ± .02
Celtec Sintra	.47 ± .02	.70 ± .01
Delrin Polyoxymethylene [Combination of teflon fibers uniformly dispersed in delrin acetal resin]	.23 ± .02	.76 ± .02
Pyrolytic Graphite	.48 ± .01	.55 ± .02
Nylon Synthetic polyamide	.32 ± .02	.76 ± .02
PEEK Polyetherether ketone Curbell Plastic	.30 ± .02	.62 ± .02
Polycarbonate	.41 ± .02	.83 ± .02
Clear Polyester Polyester Transilwrap	**	1.02 ± .02
Industrial Grade Polyester PETG Transilwrap	.60 ± .03	.97 ± .01
High Density Polyethylene HMW-Pipe Grade Curbell Plastics	.30 ± .01	.58 ± .02
Low Density Polyethylene Curbell Plastics	.42 ± .01	.68 ± .01

MATERIAL & SUPPLIER WHEN KNOWN	UNTREATED	ATOMIC OXYGEN EXPOSED*
Kapton Polyimide DuPont	---**	.85 ± .03
Polypropylene Co-Polymer Curbell Plastics	.37 ± .02	.56 ± .03
Polystyrene [Vinylbenzene]	.51 ± .05	.58 ± .03
Polysulfone Curbell Plastics	.55 ± .02	.73 ± .02
PVC Type 1, Grade 1 Polyvinyl chloride Transilwrap	.24 ± .02	.70 ± .01
Natural Rubber	.86 ± .10	.51 ± .03
Silicone Rubber SE6635 [phenyl vinyl methyl polysiloxane] General Electric	.82 ± .03	.50 ± .02
Silicone Rubber SE6140 [Vinyl methyl polysiloxane] General Electric	.81 ± .02	.61 ± .02
Silicone Rubber FSE 7140	.75 ± .03	.39 ± .02
Silicone Rubber DC 93 - 500 Dow Corning	1.47 ± .04	.39 ± .02
PTFE Teflon Polytetrafluoro- ethylene	.40 ± .02	.54 ± .0
FEP Teflon [Fluoronated ethylene- propylene]	.44 ± .02	.73 ± .02
Torlon Polyimide Curbell Plastics	.20 ± .01	.62 ± .04
.010" Clear TRNASVY Transilwrap	**	.79 ± .02
.060" Clear TXP Transilwrap	.54 ± .04	.78 ± .02



MATERIAL & SUPPLIER WHEN KNOWN	UNTREATED	ATOMIC OXYGEN EXPOSED*
ULTEM Polyetheramide Curbell Plastics	.30 ± .01	.46 ± .02
Cast Urethane Shore Hardness 95 Curbell Plastics	.81 ± .03	1.09 ± .02
Cast Urethane Shore Hardness 60 Curbell Plastics	.76 ± .06	.65 ± .01
.060" Clear Uvex Transilwrap	.64 ± .03	.99 ± .03
.015" Frosty Clear Calendered Vinyl Matte/Matte Transilwrap	**	**
.010" Clear Pressed & Polished Vinyl Transilwrap	**	.77 ± .01

\*Kapton effective Atomic Oxygen Fluence ~  $10^{21}$  atoms/cm<sup>2</sup>

\*\*Static charge prevented measurement

TABLE II. Coefficient of static friction for polished 300 series stainless steel sliding on untreated and atomic oxygen exposed polymers

MATERIAL & SUPPLIER WHEN KNOWN	UNTREATED	ATOMIC OXYGEN EXPOSED*
ABS [Styrene-acrylonitrile matrix and styrene-butadiene rubber]	.22 ± .02	.40 ± .03
Cellulose Acetate	.28 ± .02	.32 ± .01
Celtec Sintra	.18 ± .01	.31 ± .02
Delrin Polyoxymethylene [Combination of teflon fibers uniformly dispersed in delrin acetal resin]	.15 ± .01	.12 ± .01
Pyrolytic Graphite	.21 ± .01	.22 ± .01
Nylon Synthetic polyamide	.16 ± .02	.25 ± .01
PEEK Polyetherether ketone Curbell Plastic	.18 ± .01	.21 ± .01
Polycarbonate	.28 ± .01	.26 ± .01
Clear Polyester Polyester Transilwrap	.19 ± .01	.18 ± .01
Industrial Grade Polyester PETG Transilwrap	.24 ± .02	.28 ± .01
High Density Polyethylene HMW-Pipe Grade Curbell Plastics	.16 ± .01	.22 ± .02
Low Density Polyethylene Curbell Plastics	.24 ± .01	.32 ± .01

MATERIAL & SUPPLIER WHEN KNOWN	UNTREATED	ATOMIC OXYGEN EXPOSED*
Kapton Polyimide DuPont	.36 ± .03	.33 ± .02
Polypropylene Co-Polymer Curbell Plastics	.19 ± .01	.15 ± .01
Polystyrene [Vinylbenzene]	.35 ± .05	.36 ± .02
Polysulfone Curbell Plastics	.20 ± .02	.31 ± .02
PVC Type 1, Grade 1 Polyvinyl chloride Transilwrap	.18 ± .02	.30 ± .01
Natural Rubber	.37 ± .01	.46 ± .02
Silicone Rubber SE6635 [phenyl vinyl methyl polysiloxane] General Electric	***	.75 ± .03
Silicone Rubber SE6140 [Vinyl methyl polysiloxane] General Electric	***	**
Silicone Rubber FSE7140	***	.61 ± .03
Silicone Rubber DC93 - 500 Dow Corning	***	**
PTFE Teflon Polytetrafluoro- ethylene	.17 ± .01	.25 ± .01
FEP Teflon [Fluorinated ethylene- propylene]	.36 ± .01	.20 ± .01
Torlon Polyimide Curbell Plastics	.15 ± .02	.32 ± .02
.010" Clear TRNASVY Transilwrap	.23 ± .02	.57 ± .02

MATERIAL & SUPPLIER WHEN KNOWN	UNTREATED	ATOMIC OXYGEN EXPOSED*
.060" Clear TXP Transilwrap	.19 ± .02	.37 ± .02
ULTEM Polyetheramide Curbell Plastics	.26 ± .01	.14 ± .01
Cast Urethane Shore Hardness 95 Curbell Plastics	.28 ± .03	.39 ± .01
Cast Urethane Shore Hardness 60 Curbell Plastics	.21 ± .01	.31 ± .01
.060" Clear Uvex Transilwrap	.25 ± .03	.40 ± .01
.015" Frosty Clear Calendered Vinyl Matte/Matte Transilwrap	.17 ± .02	.20 ± .01
.010" Clear Pressed & Polished Vinyl Transilwrap	.18 ± .01	.19 ± .01

\*Kapton effective Atomic Oxygen Fluence ~  $10^{21}$  atoms/cm<sup>2</sup>

\*\*Tumbled off at a lower angle of incline than the untreated sample

\*\*\*Well over 90° incline

Table III. Water contact angle for pristine and atomic oxygen exposed polymers

MATERIAL & SUPPLIER WHEN KNOWN	UNTREATED	ATOMIC OXYGEN EXPOSED*
ABS [Styrene-acrylonitrile matrix and styrene- butadiene rubber]	84.4 ± .49°	5.4 ± .49°
Cellulose Acetate	51.8 ± 1.2°	9.7 ± .68°
Celtec Sintra	69.6 ± 1.5°	5.2 ± .40°
Delrin Polyoxymethylene [Combination of teflon fibers uniformly dispersed in delrin acetal resin]	83.4 ± 1.0°	38.8 ± .75°
Pyrolytic Graphite	110.0 ± .89°	4.4 ± .80°
Nylon Synthetic polyamide	74.2 ± .75°	3.8 ± .40°
PEEK Polyetherether ketone Curbell Plastic	83.6 ± .49°	4.6 ± .49°
Polycarbonate	74.4 ± .80°	10.2 ± .75°
Clear Polyester Polyester Transilwrap	83.8 ± .75°	25.0 ± .89°
Industrial Grade Polyester PETG Transilwrap	76.4 ± 1.0°	18.8 ± .75°
High Density Polyethylene HMW-Pipe Grade Curbell Plastic	87.6 ± 1.4°	3.6 ± .49°
Low Density Polyethylene Curbell Plastic	80.9 ± .66°	49.2 ± .75°

MATERIAL & SUPPLIER WHEN KNOWN	UNTREATED	ATOMIC OXYGEN EXPOSED*
Kapton Polyimide DuPont	62.1 ± .66°	0**
Polypropylene Co-Polymer Curbell Plastic	94.8 ± .75°	23.4 ± 1.0°
Polystyrene [Vinylbenzene]	72.0 ± 1.1°	0**
Polysulfone Curbell Plastics	85.6 ± .80°	17.2 ± .75°
PVC Type 1, Grade 1 Polyvinyl chloride Transilwrap	69.4 ± 1.6°	10.0 ± .63°
Rubber (Natural)	91.3 ± 1.8°	23.6 ± 1.1°
Silicone Rubber SE6635 [phenyl vinyl methyl polysiloxane] General Electric	92.4 ± 1.2°	91.0 ± .32°
Silicone Rubber SE6140 [Vinyl methyl polysiloxane] General Electric	94.4 ± .80°	90.8 ± 1.2°
Silicone Rubber FSE7140	95.7 ± 1.9°	67.6 ± 1.4°
Silicone Rubber DC93 - 500 Dow Corning	102.0 ± .89°	93.2 ± .75°
PTFE Teflon Polytetrafluoro- ethylene	98.6 ± 1.2°	95.0 ± .63°
FEP Teflon [Fluoronated ethylene- propylene]	78.8 ± 1.2°	96.0 ± .89°
Torlon Polyimidamide Curbell Plastics	83.2 ± 1.3°	15.2 ± .75°
.010" Clear TRNASVY Transilwrap	95.2 ± .75°	34.2 ± .98°

MATERIAL & SUPPLIER WHEN KNOWN	UNTREATED	ATOMIC OXYGEN EXPOSED*
.060" Clear TXP Transilwrap	85.4 ± .49°	28.2 ± .75°
ULTEM Polyetheramide Curbell Plastics	63.6 ± .77°	20.0 ± .89°
Cast Urethane Shore Hardness 95 Curbell Plastics	93.8 ± 1.2°	30.4 ± .49°
Cast Urethane Shore Hardness 60 Curbell Plastics	100.8 ± .75°	45.6 ± .49°
.060" Clear Uvex Transilwrap	85.8 ± .75°	3.2 ± .4°
.015" Frosty Clear Calendered Vinyl Matte/Matte Transilwrap	86.4 ± .49°	45.6 ± .49°
.010" Clear Pressed & Polished Vinyl Transilwrap	91.4 ± .49°	39.8 ± .75°

\*Kapton effective Atomic Oxygen Fluence ~ 10<sup>20</sup> atoms/cm<sup>2</sup>

\*\*very wetting

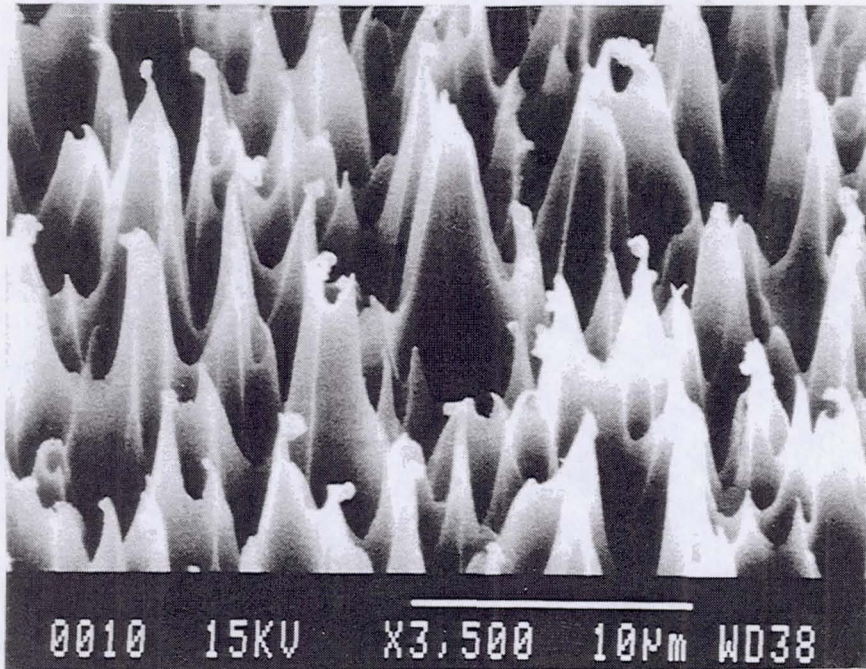


Figure 1. Scanning Electron Micrograph of the surface of chlorotrifluoroethylene exposed in space on the Long Duration Exposure Facility to a directed atomic oxygen fluence of  $5.77 \times 10^{21}$  atoms/cm<sup>2</sup>.

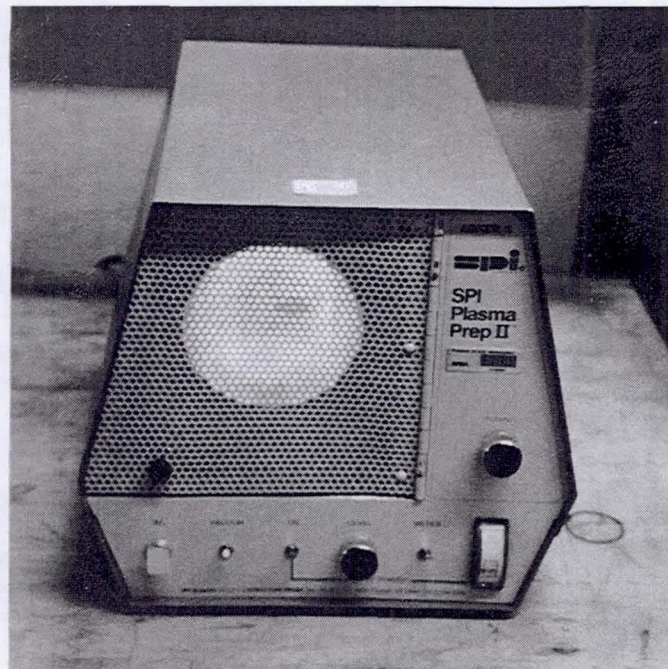
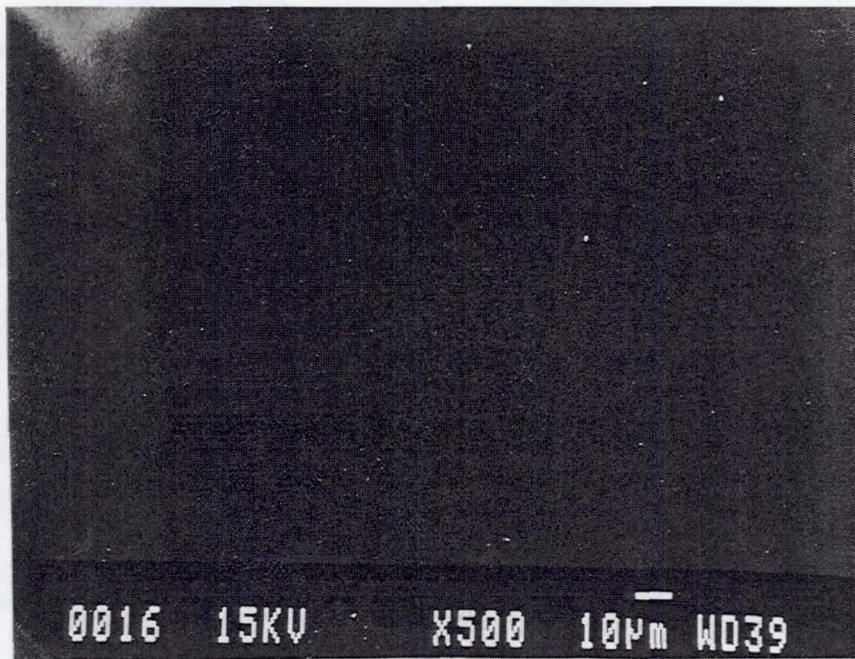
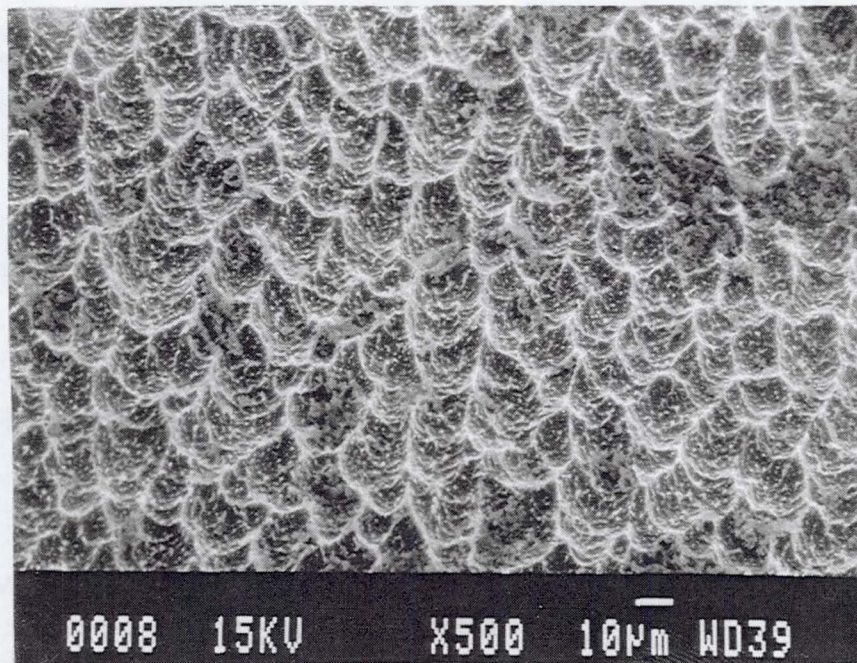


Figure 2. 100 watt, 13.56 MHz RF plasma asher operated on air.



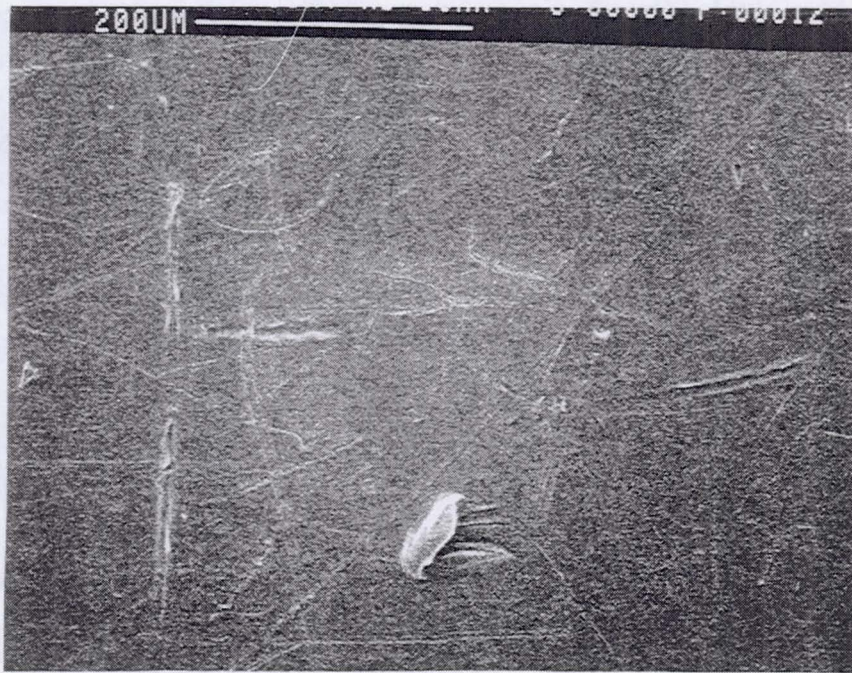


3a. Pristine



3b. After exposure to an effective atomic oxygen fluence of  $2.9 \times 10^{21}$  atoms/cm<sup>2</sup>

Figure 3. Scanning Electron Micrographs of K-max acrylic.

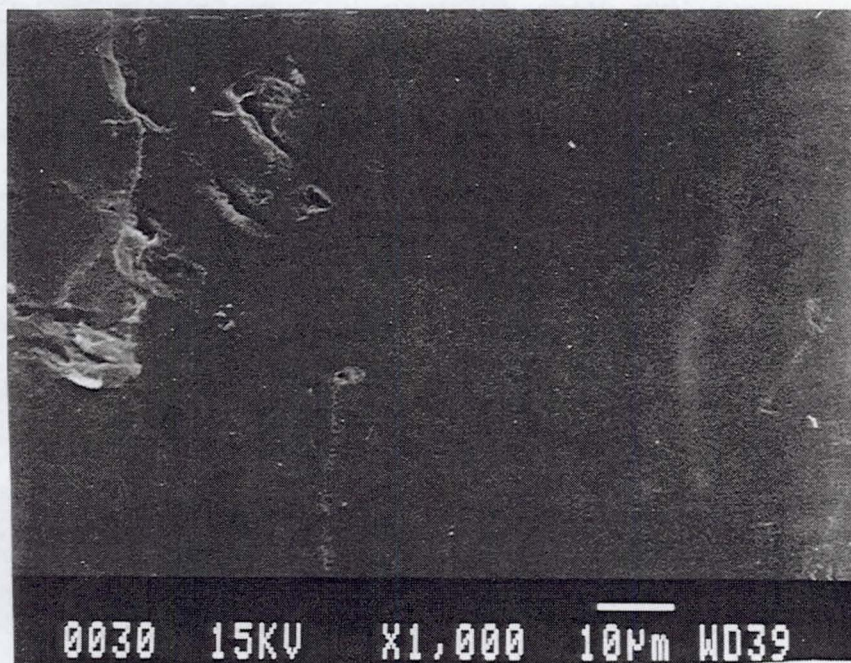


4a. Pristine

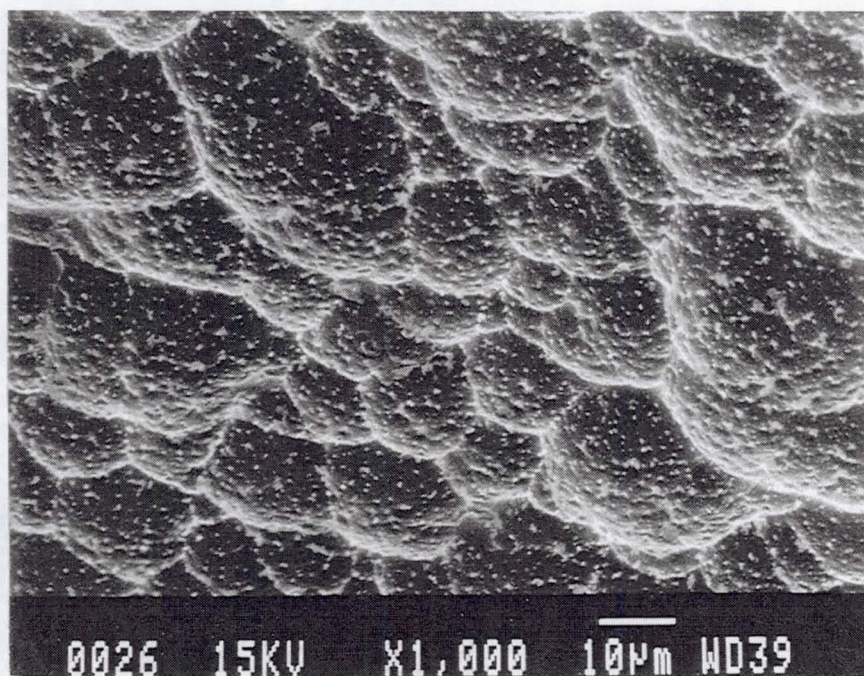


4b. After exposure to an effective atomic oxygen fluence of  $2.9 \times 10^{21}$  atoms/cm<sup>2</sup>.

Figure 4. Scanning electron micrographs of high density polyethylene



5a. Pristine



5b. After exposure to an effective atomic oxygen fluence of  $2.9 \times 10^{21}$  atoms/cm<sup>2</sup>.

Figure 5. Scanning electron micrographs of exposed polystyrene

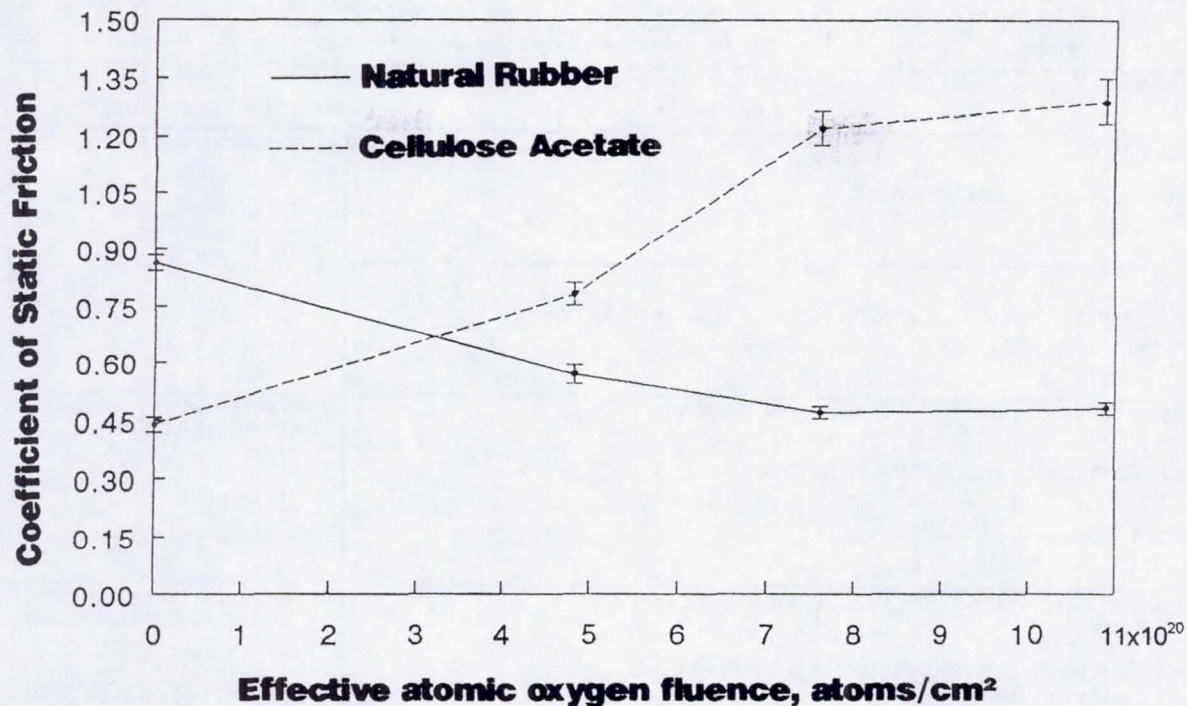


Figure 6. Coefficient of static friction between atomic oxygen textured surfaces sliding on identical atomic oxygen textured surfaces as a function of Kapton effective atomic oxygen fluence.

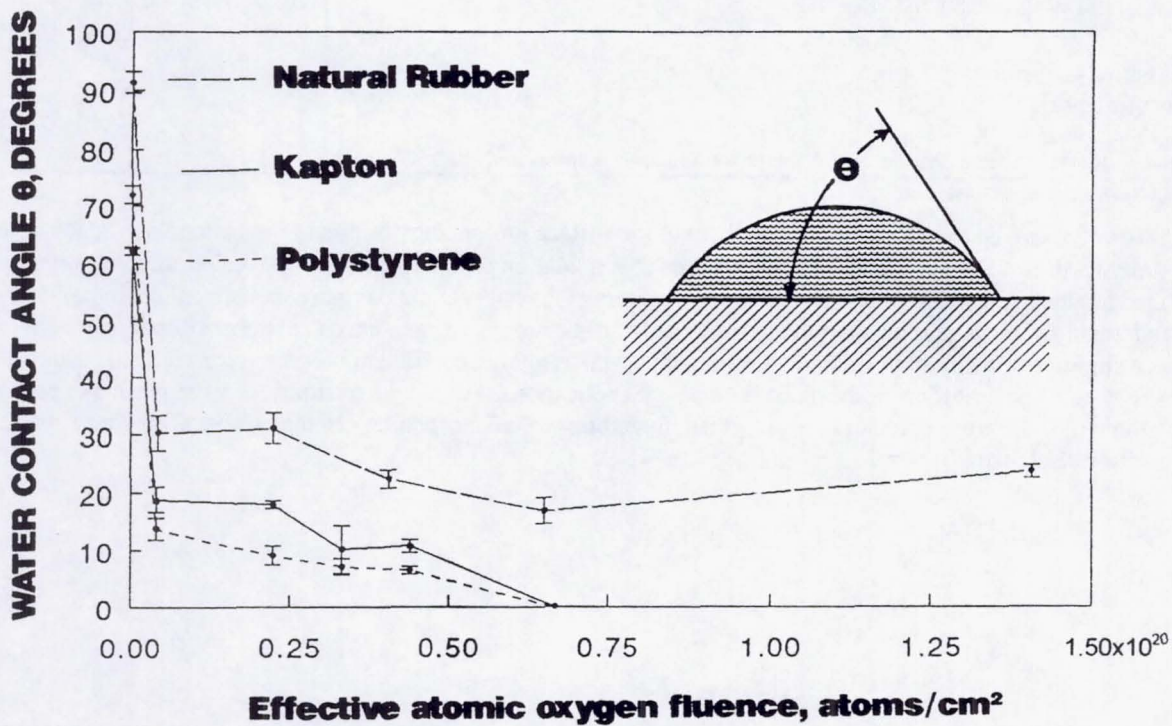


Figure 7. Water contact angle as a function of effective atomic oxygen fluence for polyimide Kapton, polystyrene and natural rubber.

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