

# polymer papers

## ESCA study of several fluorocarbon polymers exposed to atomic oxygen in low Earth orbit or within or downstream from a radio-frequency oxygen plasma

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The ESCA (electron spectroscopy for chemical analysis) spectra of films of poly(vinyl fluoride) (Tedlar), tetrafluoroethylene-hexafluoropropylene copolymer (in the form of a Teflon FEP coating on Kapton H, i.e. Kapton F) and polytetrafluoroethylene (Teflon or Teflon TFE) exposed to atomic oxygen ( $O(^3P)$ ) – either in low Earth orbit (LEO) on the STS-8 Space Shuttle or within or downstream from a radio-frequency oxygen plasma – were compared. The major difference in surface chemistry of Tedlar induced by the various exposures to  $O(^3P)$  was a much larger uptake of oxygen when etched either in or out of the glow of an  $O_2$  plasma than when etched in LEO. In contrast, Kapton F exhibited very little surface oxidation during any of the three different exposures to  $O(^3P)$ , while Teflon was scarcely oxidized.

(Keywords: ESCA; poly(vinyl fluoride); Kapton F; polytetrafluoroethylene; atomic oxygen; etching; space environment; radio-frequency oxygen plasma)

### INTRODUCTION

Recently, we compared the ESCA (electron spectroscopy for chemical analysis) spectra of Kapton polyimide film (Kapton H) exposed to atomic oxygen ( $O(^3P)$ ) either in low Earth orbit (LEO) on the STS-8 Space Shuttle or within or downstream from a radio-frequency (r.f.)  $O_2$  plasma<sup>1</sup>. The major difference in surface-chemical changes produced on exposure to  $O(^3P)$  in space or in ground-based r.f.  $O_2$  discharge apparatus – both of which caused surface recession (etching)<sup>2</sup> – was a much larger uptake of oxygen by Kapton H when etched in or out of the glow of an  $O_2$  plasma than when etched in LEO. This difference was attributed to the presence of molecular oxygen in the plasma reactor and its virtual absence in LEO. In the r.f. reactor,  $O_2$  is believed to react with polymer radicals generated in the Kapton H macromolecule as it etches, becoming incorporated in the etched polymer, and thereby yielding a higher steady-state 'surface oxidation' than is obtained in LEO. Although the O atoms in a low-pressure, r.f.  $O_2$  discharge apparatus operating near ambient temperature have translational energy of  $\approx 0.04$  eV, while the O atoms in LEO have collisional energy of  $\approx 5$  eV (owing to the  $8 \text{ km s}^{-1}$  velocity of the Space Shuttle)<sup>3,4</sup>, this difference in  $O(^3P)$  impact energy is not considered to affect the oxygen uptake at the polymer surface, but it does have a strong effect on the absolute etch rates<sup>2</sup> of Kapton H.

The availability of several fluorocarbon polymer films that were returned from the STS-8 Shuttle flight<sup>3</sup>, namely poly(vinyl fluoride) (Tedlar), tetrafluoroethylene-hexafluoropropylene copolymer (in the form of a Teflon FEP

coating on Kapton H, i.e. Kapton F) and polytetrafluoroethylene (Teflon or Teflon TFE), prompted us to determine, by means of ESCA, if these LEO-exposed polymers likewise exhibited a lower oxygen uptake than the corresponding polymers exposed to  $O(^3P)$  in an r.f.  $O_2$  discharge apparatus. Our interest is also drawn to these fluorocarbon polymers since, in the only publication other than Reference 1 which deals with ESCA spectra of polymer films recovered from the STS-8 flight, Worley *et al.*<sup>5</sup> reported on an ESCA analysis of white Tedlar (i.e. Tedlar containing inorganic pigment) and Kapton F, as well as Kapton H (equivalent to the Kapton discussed in our previous paper<sup>1</sup>). These authors<sup>5</sup> presented in their Figure 2 what they regarded as  $C_{1s}$  spectra of white Tedlar, before and after exposure to O atoms during the STS-8 mission, but this paper will show that these spectra are not representative of the Tedlar macromolecule, with its  $-\text{CH}_2\text{CHF}-$  monomer unit. Moreover, they made two claims concerning ESCA spectral shifts that appeared to be incorrect: first, 'the ... two  $C_{1s}$  bands [representing] the two inequivalent C atoms in ... Tedlar ... shift to higher [our italics] binding energy upon exposure to space ... demonstrat[ing] that the surface was oxidized during flight, probably by atomic oxygen'; and second 'the  $C_{1s}$  bands for Kapton F [whose structure contains  $-\text{CF}_3$  and  $\text{>CF}-$  in addition to  $-\text{CF}_2-$  units] ... both [!] shifted to lower binding energy for the exposed shuttle sample vs the unexposed control, [indicating] that fluorine atoms are removed from the sample in space due to bombardment by atomic oxygen'. As indicated in this work, Tedlar does undergo surface oxidation, and loss of fluorine, upon exposure to  $O(^3P)$  whether in LEO or in r.f.  $O_2$  discharge apparatus, but Kapton F upon similar exposures is only slightly

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oxidized and experiences negligible loss of fluorine. However, there are no  $C_{1s}$  spectral shifts attending the oxidation of Tedlar, only the development of an additional  $C_{1s}$  peak representing a carbon atom attached to an oxygen atom; nor are there any significant shifts in the case of Kapton F.

## EXPERIMENTAL

Disks stamped out of 25  $\mu\text{m}$  thick Tedlar, Kapton F and Teflon films (the first two from Du Pont; the third from Chemical Fabrics, Torolon Division; all films used as received) were individually exposed at ambient temperature for various periods of time to  $O(^3P)$  downstream from an  $O_2$  plasma, using the flow-tube discharge apparatus described previously<sup>2</sup>, or in the glow of a low-pressure  $O_2$  plasma reactor (Plasma Science PS0500). Samples of the same kinds of polymer film (12.5–25  $\mu\text{m}$  thick) exposed in LEO for  $\approx 40$  h at 121°C on the STS-8 Space Shuttle<sup>3,4</sup> were made available for this study. ESCA analysis of the polymer films before and/or after  $O(^3P)$ -induced etching was performed on a Surface Science Instruments SSX-101 spectrometer, using monochromatic Al  $K\alpha$  X-rays. The binding energies ( $BE$ ) of the ESCA peaks for each fluorocarbon polymer were determined by referencing to the  $F_{1s}$  peak, whose  $BE$  was first determined by thinly staining the polymer surface with hydrocarbon and then assigning the value of 284.6 eV to the  $CH_2 C_{1s}$  peak. The  $O(^3P)$ -exposed films were found to be stable to the atmosphere, so no special handling was required in the time between exposure to O atoms and eventual ESCA analysis.

## RESULTS AND DISCUSSION

### Tedlar

Figure 1 shows the  $C_{1s}$  ESCA spectra of Tedlar film before and after exposure to  $O(^3P)$  in LEO, or downstream from or within the glow of an r.f. oxygen plasma. The ESCA data are summarized in Table 1, where the estimated accuracy of each deconvoluted peak's contribution to the surface atomic percentage composition is  $\pm 5\%$  of its given value; the estimated accuracy of each F/C ratio is also  $\pm 5\%$ . Since the  $O_{1s}$  and  $F_{1s}$  spectra corresponding to the  $C_{1s}$  spectra of Figures 1a–d each possessed a single, essentially symmetric peak in the binding energy ranges indicated in Table 1, there is no need to show them here. The two prominent  $C_{1s}$  peaks at 284.8 and 287.0 eV in the

spectrum of the unexposed Tedlar film, which are of approximately equal intensity and which remain unshifted in the spectra of the  $O(^3P)$ -exposed films, are assigned to  $-CH_2-$  and  $-CHF-$ , respectively. The very weak signal at 289.5 eV in Figure 1a may be associated in part with the slight oxygen content of the unreacted Tedlar (0.3 at% O in Table 1) and in part with some unspecified structural irregularity at the surface. The 289.5 eV peak, which disappears as Tedlar is etched, is insignificant with respect to the surface chemical changes observed in this work. A small oxygen content in untreated Tedlar was also noted by Corbin *et al.*<sup>6</sup>, who presented an ESCA spectrum similar to Figure 1a, having the same two prominent peaks (at  $\approx 284.9$  and 287.3 eV), but with an F/C ratio (0.31) that was, surprisingly, well below that (0.49) obtained here. The spectra of the  $O(^3P)$ -exposed Tedlar films all show the development of a new peak at 288.9–289.0 eV, which increases in intensity from 1.4 to 4.2 at% C from Figure 1b to Figure 1d and which is assigned to a carbon attached to an oxygen, presumably  $>C=O$ . There is a parallel, progressive increase in intensity of the  $O_{1s}$  peak at 532.0–532.4 eV ( $>C=O$ ;  $>C-O-$ )<sup>7</sup>, with oxygen contents increasing from 6.7 to 16.4 at% O for Figures 1b–d (see Table 1). As observed earlier with Kapton H, there is a much larger oxygen uptake in Tedlar etched in an r.f.  $O_2$  discharge than in LEO, and we again account for this difference by invoking the reaction scheme<sup>1</sup> involving the participation of molecular oxygen present in the r.f. reactor but absent in LEO.

Accompanying the oxidative etching of Tedlar is a substantial loss of fluorine atoms and a slight loss of carbon atoms: in the case of Figure 1d, these losses amount to 32.7–20.3, or 12.4 at% F, and to 67.0–63.3, or 3.7 at% C, respectively; together, the losses of F and C correspond to the 16.1 at% O gain of oxygen atoms (see Table 1). The loss of fluorine is reflected in the decrease of the F/C ratio from the initial value of 0.49 (theoretical: 0.50) to 0.39 for the LEO-exposed film, and to 0.34 or 0.32 for the films exposed downstream from or within the glow of an  $O_2$  plasma, respectively. Interestingly, the shapes and locations of the  $F_{1s}$  peaks at 685.9–686.1 eV ( $>CF-$ ) in the spectra of Tedlar before and after various exposures to  $O(^3P)$  are not perceptibly affected by the surface oxidation. Since the intensities of the  $O_{1s}$  peaks (at 532.0–532.4 eV) greatly exceed those of the  $C_{1s}$  peaks (at 288.9–289.0 eV), most of the oxidation must involve formation of  $>C-O-$  groups: such groups, which have  $C_{1s}$  peaks around

Table 1 ESCA data for Tedlar before and after exposure to  $O(^3P)$

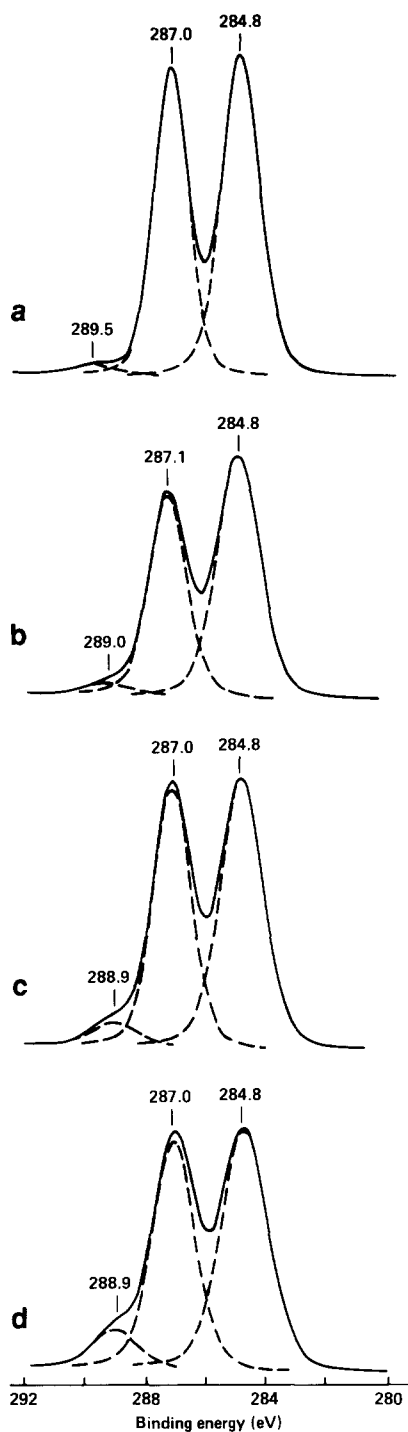
Peaks	$BE$ (eV)	Composition (at%)			
		Unexposed <sup>a</sup>	LEO <sup>b</sup>	Out of glow <sup>c</sup>	In glow <sup>d</sup>
$C_{1s}$	284.8	35.9	38.6	35.0	32.0
	287.0–287.1	30.2	27.3	29.0	27.1
	288.9–289.0	—	1.4	2.8	4.2
	289.5	0.9	—	—	—
$O_{1s}$	532.0–532.4	0.3	6.7	10.2	16.4
$F_{1s}$	685.9–686.1	32.7	26.0	23.0	20.3

<sup>a</sup> F/C ratio = 0.49; theoretical: 0.50 (Figure 1a)

<sup>b</sup> F/C ratio = 0.39 (Figure 1b)

<sup>c</sup> F/C ratio = 0.34 (Figure 1c)

<sup>d</sup> F/C ratio = 0.32 (Figure 1d)



**Figure 1**  $C_{1s}$  ESCA spectra of Tedlar: (a) typical initial, unexposed film; (b) film recovered from the STS-8 Space Shuttle flight, involving exposure for  $\approx 40$  h to  $O(^3P)$  in LEO; (c) film after exposure for 15 min to  $O(^3P)$  downstream from the r.f.  $O_2$  plasma; (d) film after exposure for 10 min 'in the glow' of an  $O_2$  plasma. The broken curves correspond to deconvolutions of the solid curves

286.7–287.5 eV<sup>7</sup>. would be obscured by the dominant  $-CHF-$  peak at 287.0–287.1 eV.

In view of the above results, the ESCA spectra presented by Worley *et al.*<sup>5</sup> can only be regarded as those of highly contaminated Tedlar. We believe that their use of a gold wire to hold the ESCA samples in place and to serve as an 'internal calibrant' contributed to distortion and artificial displacement of the two characteristic  $C_{1s}$  peaks associated with the  $-CH_2-$  and  $-CHF-$  groups, and hence to incorrect conclusions regarding shifted peaks. Thus, their control spectrum,

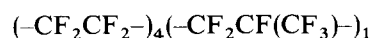
besides displaying a strongly overlapping peak at 285.4 eV which they assigned to carbon contamination on the gold wire, showed ill-defined peaks at 287.2 and 290.0 eV (cf. 284.8 and 287.0 eV in Figure 1a), for a BE difference of 1.8 eV (cf. 2.2 eV in this work). The spectrum of their LEO-exposed Tedlar exhibited, in addition to the very strong 'C/Au' peak at 285.4 eV, a strong peak at 289.4 and a weak peak at 291.5 eV (cf. peaks of similar intensity at 284.8 and 287.1 eV in Figure 1b), for a BE difference of 2.1 eV (cf. 2.3 eV here). While the BE differences in Figure 2 of Reference 5 are roughly comparable to those in our Figure 1, the presence of the gold metal in contact with the Tedlar film could have been responsible for the spurious shifts reported by Worley *et al.*<sup>5</sup>.

Although not shown here, ESCA spectra for a series of Tedlar films etched for 5–60 min downstream from (or for 2–30 min within the glow of) an r.f.  $O_2$  plasma were superimposable on those illustrated in Figure 1c (or 1d), and analysis gave approximately the same C, O and F contents as those given in column 5 (or 6) of Table 1. Again, in common with Kapton H discussed previously<sup>1</sup>, the shapes of the ESCA spectra and the surface atomic compositions for Tedlar are substantially invariant with extent of etching in the r.f. discharge apparatus, whether in or out of the glow of the  $O_2$  plasma. This indicates that, for Tedlar as well, there is a dynamic competition between degradation (or fragmentation) and oxidation, such that the etching front maintains a steady-state composition. In addition, the comments made in our previous paper<sup>1</sup> that the different reactive environments within and downstream from an  $O_2$  plasma had a negligible effect on the surface composition of Kapton H etched by  $O(^3P)$ , albeit an important catalytic or synergistic effect on etch rates, presumably apply also to Tedlar, and need not be repeated here. Likewise, we assume, as we have done with Kapton H, that the 5 eV O atoms in LEO react chemically with Tedlar in much the same way as the 0.04 eV O atoms in the r.f. discharge, but the respective Tedlar etch rates can vary considerably<sup>2</sup>.

Before leaving the subject of the Tedlar spectra, it should be noted that ESCA is not able to distinguish between head-to-tail and head-to-head additions of  $-CH_2CHF-$  units<sup>8</sup>, which are present in Tedlar in the molar ratio<sup>9</sup> of  $\approx 10:1$ .

#### Kapton F

Figure 2 shows the  $C_{1s}$  ESCA spectra of Kapton F before and after the same types of exposure to  $O(^3P)$  as discussed above for Tedlar, and the data are summarized in Table 2. Again, there was no need to show the  $O_{1s}$  and  $F_{1s}$  spectra: the  $O_{1s}$  spectra corresponding to Figures 2b–d had signals ( $\text{>C=O}$ ;  $\text{>C-O-}$ ) that were very weak and broad, while the  $F_{1s}$  spectra corresponding to Figures 2a–d each comprised a single, symmetric peak ( $\text{>CF-}$ ); the relevant BE values are given in Table 2. The major  $C_{1s}$  peak at 291.9 eV and the two minor flanking peaks of nearly equal intensity at 290.4 and 293.8 eV in the spectrum of the unexposed Kapton F film represent  $-CF_2-$ ,  $\text{>CF-}$  and  $-CF_3$  groups, respectively. The data for Figure 2a provide for the following stoichiometry for the tetrafluoroethylene-hexafluoropropylene copolymer structure of the Teflon FEP coating on Kapton H, i.e. Kapton F:



As may be seen in Figure 2, the three  $C_{1s}$  peaks characteristic of the initial Kapton F film remain virtually unshifted as the films are subjected to  $O(^3P)$  attack. Moreover, in contrast to Tedlar, Kapton F experienced only slight oxidation on exposure to  $O(^3P)$ , no doubt due to the high oxidative resistance of its perfluorinated structure. Kapton F, again unlike Tedlar, showed a small, progressive increase of surface oxidation with time of exposure in the glow (but not downstream from the glow) of the  $O_2$  plasma, with oxygen uptakes of 0.8, 1.1 and 1.4 at% O after exposures of 2, 5 and 10 min, respectively. That the LEO-exposed Kapton F film has the same oxygen uptake as the film exposed downstream from the  $O_2$  plasma (both 0.4 at% O; see Table 2) is fortuitous. The presence of hydrocarbon contamination in the former film (evidenced by the extraneous  $C_{1s}$  peak at 284.8 eV, due to  $>CH-$ , in Figure 2b) probably caused that film to be more oxidized in LEO than a pristine Kapton F film similarly exposed. (In fact, the problem of trace surface contaminants in many polymer films recovered from the STS-8 mission limited the variety of polymers we were able to examine by ESCA.) Thus, the trend of increasing oxygen uptake observed in Tedlar in Figures 1b-d apparently carries over to Figures 2b-d as well, but with substantially lower levels. Since the F/C ratios in Table 2 are all close to 2.0, we observe that there is virtually no loss of F atoms in Kapton F on exposure to  $O(^3P)$ ; this contradicts one of the claims by Worley *et al.*<sup>5</sup>, who neglected to show the  $C_{1s}$  spectrum of their LEO-exposed Kapton F.

Gjerde *et al.*<sup>10</sup> observed by means of ESCA that samples of 'single-sided aluminized' FEP (exposed surface having essentially the same structure as the exposed surface of Kapton F) reached oxygen contents of 2.5-4.9 at% O on exposure for 30 min to a low-pressure,  $O_2$  plasma reactor. That their FEP developed higher oxygen uptakes than did our plasma-exposed Kapton F (1.4 at% O) may likewise be due to hydrocarbon contamination in their films. The contamination, expressed as a ratio of intensities of the  $C_{1s}$   $>CH-$  and  $>CF-$  peaks, was 0.02 in their control and ranged from 0.08 to 0.15 in their exposed FEP samples. However, in sharp contrast to our findings with plasma-exposed Kapton F, the plasma-exposed FEP of Gjerde *et al.*<sup>10</sup> showed F/C ratios that dropped from an initial value of 2.0 to as low as 1.3. Their work was part of an effort to rationalize the unexpected degradation of Teflon retrieved from the Solar Maximum Satellite (SMS, a spacecraft which orbited the Earth at altitudes of 450-530 km compared with 225 km for STS-8, with samples exposed for 50 months on SMS and only 40 h on STS-8), a degradation that conflicted with prior indications<sup>3,4</sup> from STS-8 that Teflon was especially resistant to LEO. This apparent conflict may not be real, however, since Liang *et al.*<sup>11</sup>, who studied the ESCA spectra of a Teflon tape sample recovered from SMS, reported that the tape was not a pure fluorocarbon, but rather a copolymer of fluorocarbon and hydrocarbon, thus accounting for its extensive oxidative degradation on exposure to atomic oxygen in space.

#### Teflon

Polytetrafluoroethylene (Teflon, or Teflon TFE) exhibited little or no oxygen uptake on exposure to  $O(^3P)$  whether in LEO or in or out of the glow of an r.f.  $O_2$  plasma. Since the  $C_{1s}$  and  $F_{1s}$  spectra for the exposed

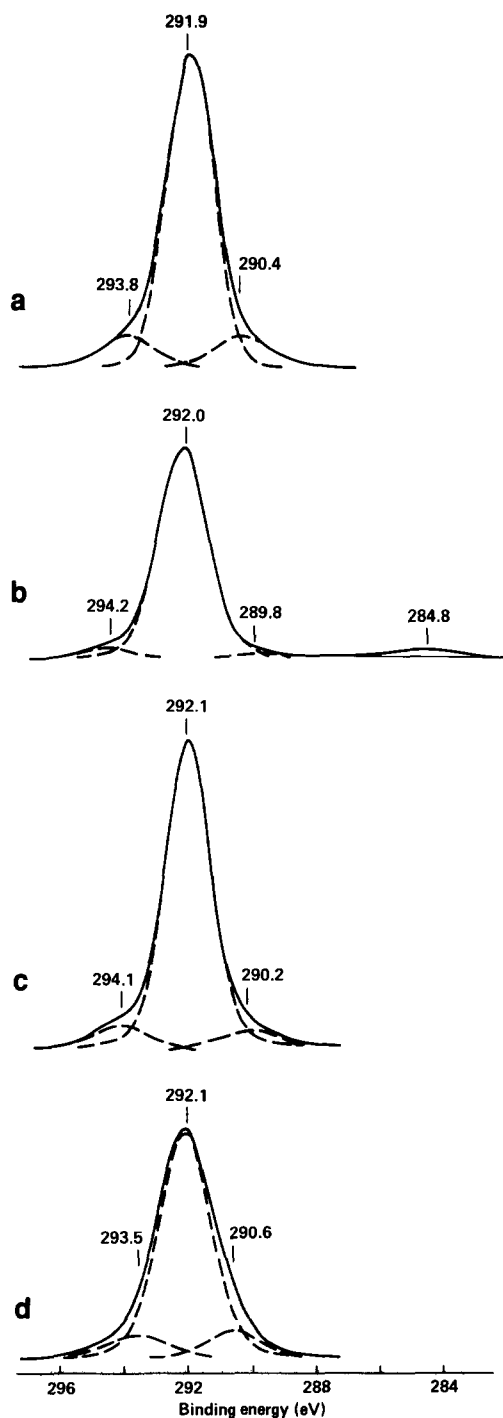


Figure 2  $C_{1s}$  ESCA spectra of Kapton F: (a), (b) and (d) refer to the same types of exposures as in Figure 1, but (c) represents a film after exposure for 1 h (instead of 15 min) to  $O(^3P)$  downstream from the r.f.  $O_2$  plasma. The broken curves correspond to deconvolutions of the solid curves

films were virtually the same as the control spectra, each exhibiting single peaks at 292.2 ( $-CF_2-$ ) or 689.2 eV ( $-CF_2-$ ), respectively, they are omitted here. Likewise, there is no need to exhibit the very weak peak(s) at around 532-535 eV ( $>C=O$ ) in the  $O_{1s}$  spectra of the exposed Teflon films. Two Teflon samples recovered from the STS-8 mission were examined: the exposed side of one film exhibited an oxygen content of 0.58 at% O and a hydrocarbon (contamination)  $C_{1s}$  peak at  $\approx 285$  eV representing 1.3 at% C, while the exposed side of the other film showed 0.90 at% O and a hydrocarbon  $C_{1s}$  peak representing 2.8 at% C. Apparently, there is a correlation between the O uptake and the level of

**Table 2** ESCA data for Kapton F before and after exposure to O(<sup>3</sup>P)

Peaks	BE (eV)	Composition (at%)			
		Unexposed <sup>a</sup>	LEO <sup>b</sup>	Out of glow <sup>c</sup>	In glow <sup>d</sup>
C <sub>1s</sub>	284.8	—	1.6	—	—
	289.8–290.6	3.1	0.3	1.6	3.0
	291.9–292.1	27.4	29.6	29.7	25.9
	293.5–294.2	3.0	1.3	2.3	3.0
O <sub>1s</sub>	532.5–535.1	—	0.4	0.4	1.4
F <sub>1s</sub>	689.2	66.5	66.8	66.0	66.7

<sup>a</sup>F/C ratio = 1.99; theoretical: 2.00 (Figure 2a)<sup>b</sup>F/C ratio = 2.04 (Figure 2b)<sup>c</sup>F/C ratio = 1.96 (Figure 2c)<sup>d</sup>F/C ratio = 2.09 (Figure 2d)

hydrocarbon contamination for Teflon films exposed to LEO on the STS-8, and we may speculate that the  $-\text{CF}_2\text{CF}_2-$  structure *per se* undergoes negligible oxygen uptake on exposure to O(<sup>3</sup>P) in LEO. We reported previously that the ESCA spectrum of a Teflon film etched to the extent of 0.4  $\mu\text{m}$  (downstream from the plasma of a single-sample r.f. reactor<sup>7</sup>) showed 0.47 at% O; in this work, the ESCA spectrum of another Teflon film etched to the extent of 0.04  $\mu\text{m}$  downstream from the plasma of a multi-sample r.f. reactor<sup>2</sup> showed <0.05 at% O. Finally, Teflon films exposed for 10, 20 and 30 min to O(<sup>3</sup>P) 'in the glow' of an r.f. O<sub>2</sub> plasma yielded oxygen uptakes of 0.11, 0.15 and 0.21 at% O, respectively. These results suggest that, in contrast to Tedlar but in common with Kapton F, Teflon shows a very small increase of surface oxidation with time of exposure to O(<sup>3</sup>P) in an r.f. O<sub>2</sub> discharge. However, in all the Teflon ESCA spectra examined, the F/C ratios were virtually unchanged from that of the control (2.0).

To conclude, Teflon (or Teflon TFE) and Kapton F (or Teflon FEP), because of their perfluorinated polymer structures, are very resistant to O(<sup>3</sup>P)-induced surface oxidation, whereas Tedlar with three hydrogen atoms and one fluorine atom in its monomer unit ( $-\text{CH}_2\text{CHF}-$ ) is highly vulnerable to such oxidation. An ESCA examination of the effects of O(<sup>3</sup>P) on other partially fluorinated polymers, such as poly(vinylidene fluoride), along with that of the non-fluorinated polymer polyethylene, would be of interest. Although this paper has not dealt with the relative etch rates for Tedlar, Kapton F and Teflon on exposure to O(<sup>3</sup>P) in LEO or within or downstream from an r.f. O<sub>2</sub> plasma, a survey of such data is given in Reference 2.

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