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Polytetrafluoroethylene Transfer
Film Studied With X-Ray
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Summary

Polytetrafluoroethylene (PTFE) was rubbed against nickel in ultrahigh vacuum at loads up to 3.9 newton (400 g weight) and speeds up to 94 millimeters per second. The transfer film formed on the nickel was analyzed using X-ray photoelectron spectroscopy.

It was found that the film was indistinguishable from bulk PTFE except for the possible presence of a small amount (<1%) of NiF₂. The transfer film was found to be about 1 molecule (0.5 nm) thick under all conditions; but at speeds above 10 millimeters per second, there was evidence of bulk transfer in the form of fragments as well. The thickness measurements required a choice among conflicting published values of the inelastic mean free path for electrons in polymers. The values chosen gave internally consistent results.

Introduction

There is ample evidence that the desirable friction properties of polytetrafluoroethylene (PTFE) depend on the development of a transfer film of PTFE on the material against which it rubs (refs. 1 and 2). This film has been the subject of several studies (refs. 1 to 7), but fundamental questions are still unanswered: (1) Is the transfer film developed by sliding, in fact, PTFE? (2) What is the thickness of the film? (3) What interaction between film and substrate is relevant to the adhesion of the film to the substrate? The present work was undertaken to answer these questions.

The work of Pooley and Tabor (ref. 2) and Makinson and Tabor (ref. 1) established some basic features of the transfer process. At low speed (~1 mm/sec) the transferred material consists initially of lumps and fragments of PTFE. After sliding for some time, however, a transfer film consisting of sheets or streaks of PTFE develops. From electron microscope observations, this film is estimated by Pooley and Tabor to be less than 10 nanometers thick. At higher sliding speeds (>100 mm/sec) the transferred PTFE always contains fragments. The same behavior was observed on glass and metal provided the surface was smooth (<0.1 μm mean asperity height).

Pepper studied the transfer of PTFE to S-Monel using Auger electron spectroscopy (AES) (ref. 7). From the attenuation by the film of the Auger

electrons from the substrate, he estimated that the film was 2 to 4 molecules thick. Electron induced desorption of the fluorine atoms complicated interpretation of the results.

Cadman, et al. (refs. 4 to 6) have studied the PTFE transfer film on a variety of metals using X-ray photoelectron spectroscopy (XPS). The film is stable during XPS analysis, and XPS has surface sensitivity similar to AES. The conditions of rubbing are not specified in the experiments of Cadman, et al. and were apparently uncontrolled to some degree. Both thin layers and bulk transfer were observed. Since the intensity of electron emission from the thin film component of the transfer film varied with the angle of emission, it was concluded that the thickness of this component was on the order of the inelastic mean free path (IMFP) of the electrons. In their studies, a weak feature in the XPS spectrum from fluorine was attributed to metal fluorides. This feature was found when PTFE was rubbed in air on stainless steel and nickel. When the PTFE transfer film was scraped away with a razor blade, the feature remained. It was concluded by Cadman, et al. that fluorides exist in the interfacial region between the substrate and the transfer film.

The purpose of the present work was to study the transfer of PTFE to metal under controlled sliding conditions in ultrahigh vacuum. Both nickel and S-Monel flats were used as substrate metals although most of the results reported here will be for nickel. Sliding speeds ranged from 0.94 to 62.8 millimeters per second with normal loads from 0.19 to 3.9 newtons on a PTFE rider with a 4.8-milimeter-radius hemispherical surface.

Experimental Apparatus

The XPS system used and the principles of XPS have been described previously (refs. 8 and 9). XPS analyzes the energies of electrons emitted by a sample when it is irradiated by a beam of X-rays. Since the energies of these electrons are less than about 1200 electron volts, their IMFP in metals is less than 2 nanometers and depends on the electron energy. The magnitude of the IMFP in polymers and its energy dependence are matters of some importance for this work. They will be discussed later. In XPS attention is focused on electrons emitted with energies characteristic of the atoms from which they come. These are photoelectrons and Auger electrons. In both cases, the details of the spectrum will depend

on the chemical state of the atoms producing it. Furthermore, the intensity of photoelectrons detected from a particular type of atom depends on the concentration of that type of atom. The technique can thus be made quantitative. Finally, in extensive studies of PTFE (ref. 10) there has been no evidence that the polymer is altered in any way by the X-ray flux it is exposed to. In the present experiment, MgK_{α} radiation was used. A region on the specimen about 2 millimeters in diameter was analyzed.

The rubbing apparatus was a conventional pin-on-disk device incorporated into the vacuum chamber of the XPS instrument. The arrangement is shown schematically in figure 1. The disk was positioned in such a way that the XPS analyzer accepted electrons from a spot about 3 millimeters in diameter located 180 degrees from the spot where the PTFE rider touched the disk. The disk was rotated by a variable speed drive so that the relative speed of disk and rider ranged from 0.94 to 62.8 millimeters per second. The PTFE rider was a 4.8-millimeter radius sphere mounted on an arm which entered the vacuum system through a welded metal bellows and was pivoted outside the system. The pivots allowed the rider to be dead-weight loaded against the disk. Loads ranged from 0.19 to 3.9 newtons (19 to 400 g weight).

An ion gun was directed at the disk in such a way that it could sputter clean an area 6 by 6 millimeters in front of the analyzer. Argon ions of 5 keV energy were used for ion bombardment. The vacuum system was at 6.7×10^{-3} N/m² (5.0×10^{-5} torr) of argon during sputtering. Otherwise, the pressure was below 1.3×10^{-7} N/m² (1.0×10^{-9} torr).

Materials

The rider was fabricated from a rod of high-purity, high-density, research-grade PTFE. The end was machined to a hemisphere of radius 4.8 millimeters and abraded with 600-grit SiC paper. XPS analysis of PTFE prepared this way showed the surface to be clean within the limits of detection of the instrument.

The nickel disk was 63.5 millimeters in diameter and was cast from nominally high-purity nickel. After sputter cleaning, XPS analysis showed no detectable contaminants. The flat surface of the disk was lapped flat and finished on 600-grit SiC paper, 6- and 3-micrometer diamond paste, and finally with 1-micrometer aluminum oxide in distilled water.

Procedure

After the disk and rider were mounted in the vacuum chamber, the system was evacuated and baked at 200° to 250° C for 12 hours. All

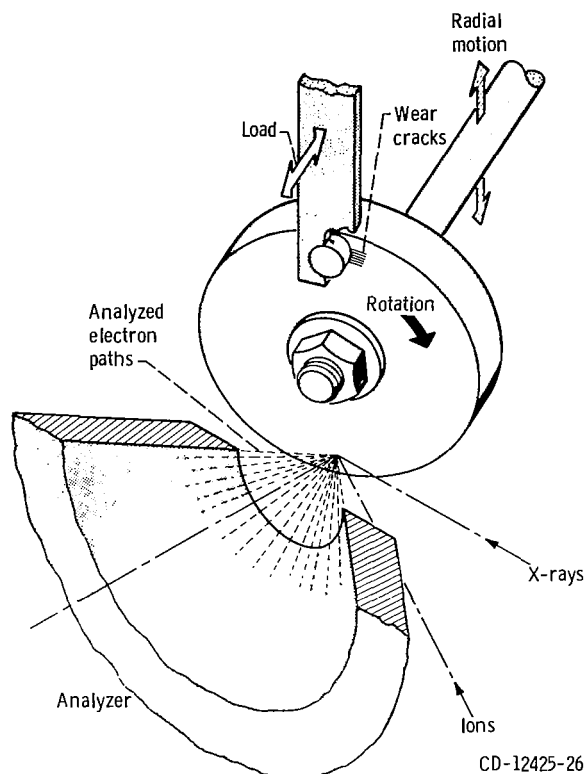


Figure 1. - Schematic diagram of apparatus for XPS analysis of PTFE transfer.

experiments were performed after the disk had cooled to between 24° and 27° C as indicated by a thermocouple spot welded to it.

With the ion gun, an area of the disk was sputter etched repeatedly until XPS analysis showed no detectable oxygen or carbon. If the rubbing surface was to be native oxide, the vacuum system was back filled with dry air, then evacuated again. Otherwise, the disk was used immediately after sputter etching.

The disk was rotated 180 degrees and moved 2.5 millimeters radially (refer to fig. 1). The rider was brought into contact with the disk at the desired load, and the motor drive was engaged to rotate the disk at the desired speed until the rider had moved about 5 millimeters along the disk. The rider was then lifted, the motor drive disengaged, the disk rotated back to its starting position and advanced 0.125 millimeter radially. The sliding procedure was repeated in this way until the disk had been advanced 5 millimeters radially. The result was a series of 40 concentric arc-shaped wear tracks about 5 millimeters long separated by 0.125 millimeter.

The disk was then rotated 180 degrees again and positioned for analysis of the wear tracks. It was also possible to position the rider tip for analysis if

desired. In some experiments oxygen was then adsorbed on the disk by using a leak valve to admit oxygen to the vacuum system at 1.33×10^{-4} N/m² (1.0×10^{-6} torr) for 150 seconds (150 Langmuir exposure).

Results and Discussion

Run-in of Rider

In experiments at 2.0-newton (210-g-weight) load and 0.94-millimeter-per-second sliding speed, it was found that the XPS spectrum was not repeatable until several sputter cleaning and rubbing sequences had been performed. Specifically, the F(1s) to C(1s) peak intensity ratio varied between 10.5 and 12.0. After two to five cleaning and rubbing sequences, the F(1s)/C(1s) ratio became repeatable at the higher value. No shift in the binding energy of the F(1s) or C(1s) peaks accompanied these variations. As will be explained next, the varying ratio is consistent with the transfer of random fragments while the higher final ratio is characteristic of a very thin film. This result agrees with the observations by Tabor, et al. (refs. 1 and 2) that fragmentary transfer occurred during the initial sliding of PTFE, but that once the PTFE rider was run-in, the transfer film was thin and uniform. This behavior was identical on nickel and S-Monel.

The rider was examined by XPS before and after the run-in. No changes in the C or F spectra were detected. The rider run-in on nickel showed Ni in the XPS spectrum. The transferred Ni fragments could also be observed optically after removing the rider from the apparatus (fig. 2). The rider run-in on S-Monel showed no such transfer of metal. Since all other results were the same on both metals, the transfer of nickel is not important to the results presented here. All results presented hereinafter will refer to experiments performed on the nickel disk after appropriate run-in.

entire spectrum so that differences between the binding energies of spectral features are unchanged. Wagner (ref. 11) has pointed out that the difference in energy between the Auger peaks and the photoelectron peaks of an element will change if the chemical state of the element is changed. This difference has been tabulated for many fluorine compounds including PTFE (ref. 12).

Table I shows the result of measuring this difference on bulk PTFE and on a transfer film formed at a 2.0-newton (212-g-weight) load and 0.94-millimeter-per-second sliding speed. The literature values for PTFE and NiF₂ are shown for comparison. The carbon Auger peak is too broad for



Figure 2. - Wear scar on PTFE rider rubbed on clean nickel in vacuum. Load, 2 newtons; sliding speed, 0.94 millimeter per second; temperature, 24^o to 27^o C. 75X. (Note nickel fragments.)

TABLE I.—RELATIVE ENERGIES OF F(1s), F AUGER, AND C(1s) ENERGIES IN THE XPS SPECTRUM OF PTFE

	F Auger—F(1s), eV	C(1s)—F(1s), eV
Bulk PTFE	87.7 ± 0.2	397.6 ± 0.2
PTFE transfer on Ni	87.7 ± 0.2	397.2 ± 0.2
Bulk PTFE (ref. 12)	87.6 ± 0.1	-----
NiF ₂ (ref. 12)	86.9 ± 0.1	-----

Chemistry of the Transfer Film and Interface

The chemistry of the transfer film will be examined in three ways: (1) the binding energies of the C(1s), F(1s) and F Auger peaks will be compared with those from bulk PTFE, (2) the structure of the Auger spectrum from the film and bulk PTFE will be compared, and (3) structure in the F(1s) photoelectron peak will be identified.

(1) Because PTFE is an insulator and charges under the X-ray beam, apparent binding energies are not significant. However, charging will shift the

this type of analysis, but the binding energy of the carbon photoelectron peak relative to the fluorine photoelectron peak can be measured. Table I also shows this measurement.

(2) Weissmann has shown that, for oxygen, the structure of the KLL Auger spectrum depends on the chemical state of the oxygen atom (ref. 13), because the intensity of features involving the 2p and 1s electron shells will depend on the degree of charge transferred to the 2p shell in the chemical bond. The feature involving only electrons in the 2s and 1s levels, however, will have constant intensity since both these levels are filled and no charge transfer occurs. The same argument should hold for fluorine.

Figure 3 shows the fluorine KLL Auger spectrum observed for both bulk PTFE and a transfer film. Feature 1 involves the 2s and 1s electrons, while feature 2 involves the 2p and 1s electrons and would be expected to change size with changes in chemical bond. Thus a change in the ratio of feature 2 to feature 1 is indicative of a change in the chemical state of the film. Although the features are not sharp and feature 1 is particularly difficult to measure, there is no difference in the spectra beyond the measurement uncertainties.

(3) Both the foregoing results are consistent with transfer film of unmodified PTFE which does not interact chemically with the nickel surface. It is possible, however, that an interaction occurs at a few isolated sites, rather than generally over the interface. This would be revealed by a chemically shifted small peak in the F(1s) or C(1s) spectrum. Such a peak would be difficult to observe in the nickel spectrum because the strong nickel peaks have associated structure which would produce a masking effect. Just such a weak peak was observed in the F(1s) spectrum by Cadman, et al. (ref. 6). It has also been observed in this work (fig. 4).

In Figure 4 the horizontal scale has been shifted to correct for sample charging so that the peak due to PTFE falls at the accepted value of 690.2 electron volts (ref. 12). The small peak at lower binding energy is then consistent with values in the literature for NiF_2 (ref. 12). While an exact measurement of the relative sizes of these peaks is futile, the 684.9 eV (NiF_2) peak is clearly less than 1 percent of the 690.2 (PTFE) peak. As will be shown later, the film is at most a few molecules thick, so that the relative sizes of these peaks imply that only an occasional fluorine has reacted chemically with the nickel substrate. Since fluorine is monovalent, it seems unlikely that it can participate directly in a strong bond between the metal and the PTFE. Rather its single bond must be formed either with a metal or with a carbon atom. The presence of the nickel fluoride peak may, however, be indirect evidence of a metal carbon

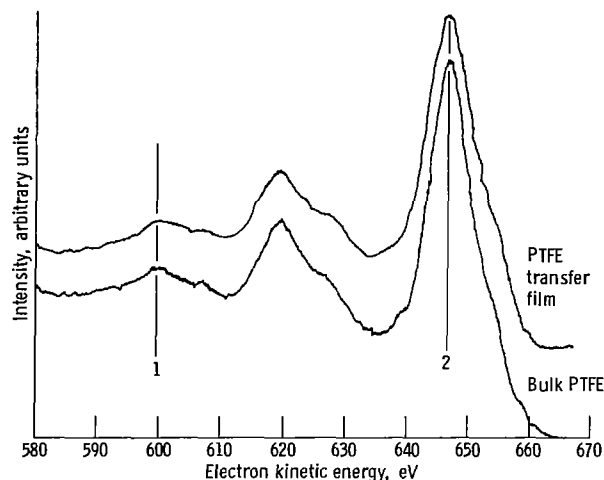


Figure 3. - Fluorine auger electron spectra from PTFE transfer film (on clean nickel in vacuum) and from bulk PTFE. Load, 2 newtons; sliding speed, 0.94 millimeter per second. (To account for charging, the upper peak is shifted horizontally so that the spectra coincide.)

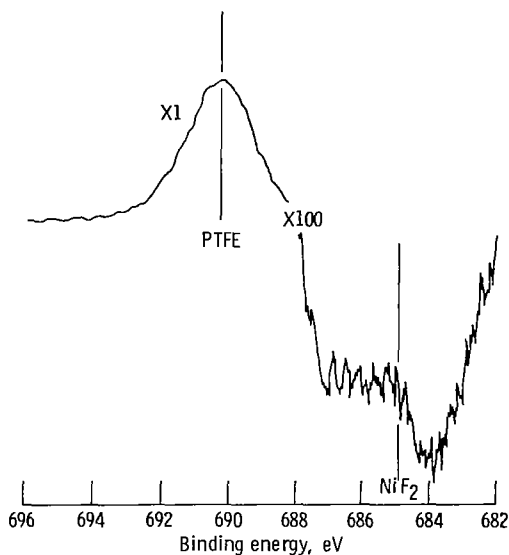


Figure 4. - F(1s) XPS peak from PTFE transfer film applied to clean nickel in vacuum. Load, 2 newtons; sliding speed, 0.94 millimeter per second.

bond. Since, in the process of bonding to the metal, a carbon atom would lose one fluorine, that fluorine could then bond to a metal atom. If there is, indeed, an occasional metal carbon bond, a small feature should appear in the C(1s) spectrum much like the nickel fluoride peak in the F(1s) spectrum. Unfortunately the sensitivity of the technique for carbon is only one-tenth that for fluorine. Therefore no such peak was observable, even if it was present.

Thickness of Transfer Film

The ratio of the intensities of the F(1s) and C(1s) peaks in the XPS spectrum from bulk PTFE is 9.8. The same ratio from transfer films (after running-in the rider) varied from 11.4 to 12.9. The ratio could be different from the bulk ratio if the film were chemically different from the bulk, but the preceding discussion indicates that it is not chemically different. The other explanation for the difference is that the film thickness is comparable to the IMFP of the photoelectrons. In this section the thickness of the PTFE transfer film will be determined by two independent methods. First, the F(1s)/C(1s) intensity ratio will be used. Then, the attenuation of the Ni(2p) peak from the substrate will be used. Both techniques require knowledge of the IMFP of electrons in polymers for which conflicting data have been published. The present results will be shown to permit a choice to be made between the two conflicting results.

F(1s)/C(1s) ratio.—Because the IMFP for the C(1s) electrons, λ_C , is greater than that for the F(1s) electrons, λ_F , the sampling depth for carbon will be greater than for fluorine on a thick sample. Consequently, as the thickness of the sample decreases the C(1s) intensity will decrease most rapidly, and the ratio of the F(1s) to C(1s) intensities will increase. The exact form of this decrease can be found as follows.

Consider a layer of thickness dt at a depth t in a film of PTFE of thickness x . The F(1s) intensity emitted from that layer at an angle θ from the normal to the surface and detected by the analyzer is

$$dI_F = K_F e^{-(t/\lambda_F \cos \theta)} dt \quad (1)$$

where K_F is a constant including the concentration of fluorine atoms, the photoelectric cross section for the F(1s) level, and the transmission of the analyzer at the energy of the F(1s) electron. The IMFP of the F(1s) electron is λ_F . The total intensity from the film is obtained by integrating

$$I_F = \int_0^x K_F e^{-(t/\lambda_F \cos \theta)} dt \\ = \lambda_F K_F \cos \theta \left[1 - e^{-(x/\lambda_F \cos \theta)} \right] \quad (2)$$

The intensity from a bulk specimen is obtained by letting $x \rightarrow \infty$; then,

$$I_F^B = \lambda_F K_F \cos \theta \quad (3)$$

Thus, the intensity of emission for the film relative to the bulk is

$$I_F = I_F^B \left[1 - e^{-(x/\lambda_F \cos \theta)} \right] \quad (4)$$

For the emission from the C(1s) level, the same argument gives

$$I_C = I_C^B \left[1 - e^{-(x/\lambda_C \cos \theta)} \right] \quad (5)$$

Thus, the F(1s)/C(1s) intensity ratio is

$$\frac{I_F}{I_C} = \frac{I_F^B}{I_C^B} \left[\frac{1 - e^{-(x/\lambda_F \cos \theta)}}{1 - e^{-(x/\lambda_C \cos \theta)}} \right] \quad (6)$$

For the experiments described here, $(I_F^B/I_C^B) = 9.8$. The geometry of the analyzer and the specimen is such that the average value of θ is 42° . If the values of λ_F and λ_C are known, then x can be determined from the measured values of I_F/I_C . However, the value of the IMFP in polymers is a subject of some controversy (refs. 14 and 15).

Table II shows published values for λ_C and λ_F . The larger value of λ_F is calculated from the corresponding value of λ_C using the energy dependence used by Evans, et al. (ref. 16). The smaller values are from Clark, et al. (ref. 17). Figure 5 is a plot of equation (6) for the two choices of λ_F and λ_C . The range of I_F/I_C measured in this study is also shown. The thickness is shown in number of molecular layers (ref. 18) as well as in nanometers. The ratio I_F/I_C was measured for PTFE transfer films applied at loads up to

TABLE II.—PUBLISHED VALUES OF THE
IMFP FOR C(1s) AND F(1s) ELECTRONS
IN FLUORO-CARBON POLYMERS

Source	λ_C , nm	λ_F , nm	Material
Ref. 16	6	^a 4.6	PTFE
Ref. 17	1	.7	Vinylidene-fluoride

^aDetermined from λ_C using $\lambda \propto E^{0.5}$.

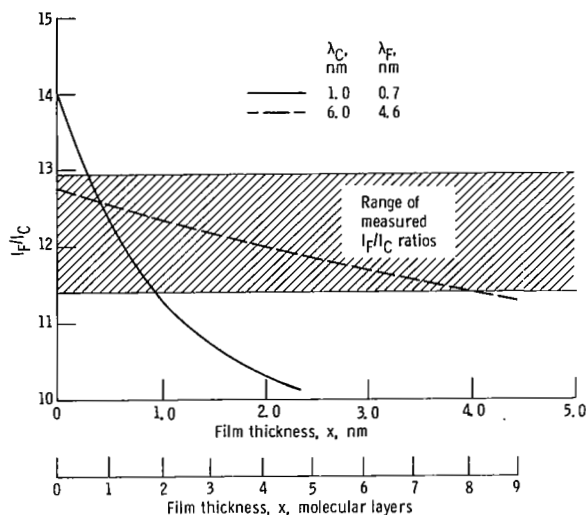


Figure 5. - Plot of $\frac{I_F}{I_C} = 9.8 \frac{1 - \exp\left(-\frac{x}{\lambda_F \cos \theta}\right)}{1 - \exp\left(-\frac{x}{\lambda_C \cos \theta}\right)}$.

4 newtons. The average film thickness x was then determined from the curves of figure 5. The solid circles are the result of using the larger (Evans) values of λ_F and λ_C , that is, the dashed curve in figure 5. The one negative value has no physical significance, but can be attributed to experimental uncertainty. The open circles are obtained from the smaller (Clark) values, that is, from the full curve in figure 5.

Attenuation of nickel spectrum.—The attenuation of the nickel spectrum by the PTFE film can be used as an independent measure of the film thickness. Once again, knowledge of the IMFP is required. Furthermore, the possibility of incomplete coverage of the nickel by the film must be considered. Letting I_1 be the intensity of a particular nickel peak from the clean surface, I_2 the intensity after applying the

PTFE film, and f the fraction of the surface covered by the film,

$$I_2 = (1 - f)I_1 + fI_1 e^{-(x/\lambda \cos \theta)} \quad (7)$$

Rearranging and taking the logarithm of both sides give

$$x = \lambda \cos \theta \ln \left[\frac{f}{(I_2/I_1) + f - 1} \right] \quad (8)$$

Once again θ is 42° . The value of λ is the IMFP in PTFE of electrons with the energy of the particular nickel peak being considered.

Since PTFE, cleaned in air, shows no oxygen upon examination with XPS, it is clear that oxygen does not absorb on PTFE. On the other hand, a reproducible oxygen signal is observed after exposure of clean nickel to 150 Langmuir of oxygen. Thus, the amount of oxygen adsorption by the nickel can be used as a measure of the coverage of PTFE, f . If I_0 is the oxygen intensity from cleaned nickel exposed to oxygen, and I'_0 is the oxygen intensity from cleaned nickel exposed to oxygen after the application of a PTFE transfer film, then

$$f = 1 - \frac{I'_0}{I_0} \quad (9)$$

This method of measurement is inherently less precise than the method using the I_F/I_C ratio, because it involves the measurement of both nickel peak ratios and oxygen peak ratio. Furthermore, the oxygen peak and the Ni(3p) peak were weak adding to the measurement problem. Finally, the appropriate background level above which the nickel intensities were measured was ambiguous because of the structure in the nickel spectral features. On the other hand, the two nickel peaks give two independent measurements for a given transfer film. By averaging these two measurements, an improved estimate can be obtained.

The attenuation of both the Ni(2p) and Ni(3p) peaks were measured for PTFE applied at several loads. In each case f was measured by oxygen exposure. Values of λ were obtained from reference 17 by interpolating between measured values: $\lambda_{Ni(2p)} = 0.7$ nanometer, $\lambda_{Ni(3p)} = 1.8$ nanometers.

Then, using equations (8) and (9), the average film thickness x was calculated. The results are plotted as the open squares in figure 6. The solid squares in figure 6 are the result of the same measurement using values of $\lambda_{Ni(2p)}$ and $\lambda_{Ni(3p)}$ calculated from λ_C in the first line of table II by assuming $\lambda \propto E^{0.5}$ according to Evans, et al.

Discussion of thickness results.—It is clear from figure 6 that these results are consistent with Pooley and Tabor's measurement of less than 10 nanometers, whichever choice of IMFP is made. The largest measured value is, in fact, about 4 nanometers or eight molecular layers. The actual transfer film may not, of course, be of uniform thickness. What is measured here is an average thickness over the analyzed area (excluding possibly uncovered regions of zero thickness).

It is also evident from figure 6 that agreement between the two methods of measurement is much better when the IMFP's due to Clark are used (open symbols), than when those due to Evans are used (solid symbols). Furthermore, use of Clark's values entails no assumption about the energy dependence of the IMFP, since they were measured over a wide range of energies. Use of the Evans values, on the other hand, requires calculation of IMFP's from the one measured value of λ_C and the assumption that λ is proportional to $E^{0.5}$. For both these reasons the Clark values seem to be most probably correct.

On the assumption that Clark's values of IMFP are correct, the average thickness of the PTFE transfer film is about one molecular layer. There is a tendency for the thickness to increase with load which is especially evident in the I_F/I_C results. However, it is just these results, at the small values of x , that are most influenced by the energy dependence of the IMFP. Since the uncertainties in the IMFP's are of the order of 30 percent (ref. 17), the trend with load cannot be taken too seriously.

To determine whether or not the transfer film thickness increased in repetitive passes, the rubbing experiment at 2 newton load was repeated 10 times. The I_F/I_C ratio was measured at intervals during the tests. It remained constant after the first pass indicating that no additional PTFE was deposited after the initial transfer.

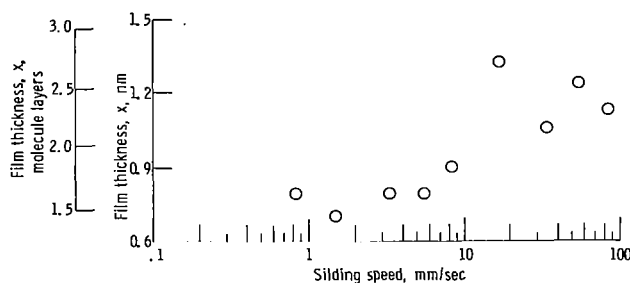


Figure 7. - Average thickness of PTFE transfer film on nickel at various sliding speeds in vacuum. Load, 2 newtons; temperature, 24° to 27° C.

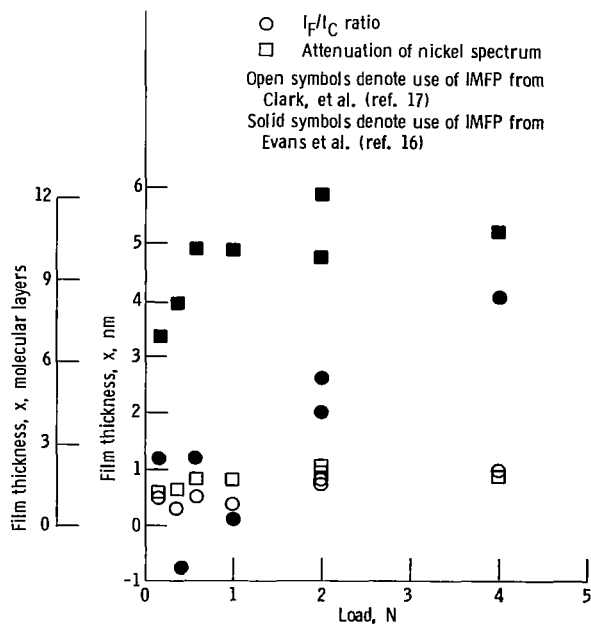


Figure 6. - Average thickness of PTFE transfer film on nickel at various loads in vacuum. Sliding speed, 0.94 millimeter per second; temperature, 24° to 27° C.

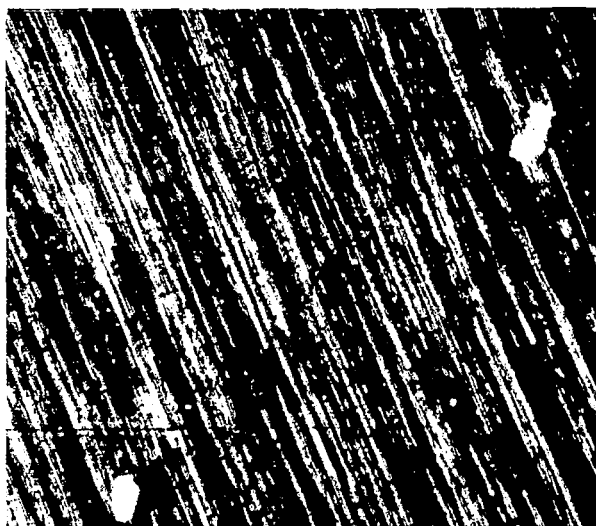


Figure 8. - Photomicrograph of clean nickel surface rubbed with PTFE in vacuum. Load, 2 newtons; sliding speed, 94 millimeters per second; temperature, 24° to 27° C. 200X.

The effect of sliding speed on the thickness of the transfer film was observed by measuring I_F/I_C for films prepared at 2 newton load and sliding speeds from 0.94 to 94.0 millimeters per second. The result is shown in figure 7. Above 10 millimeters per second the average film thickness is increased and is more irregular than at lower speed. A photomicrograph (fig. 8) of the transfer film shows scattered small bits of PTFE on a generally uniform surface. The erratic results of the higher speed tests in figure 7 could be due to occasional inclusion of microscopically thick bits of transfer material in the analyzed area, while a molecularly thin film still covers the area overall. Such a view is consistent with the observation by Tabor (ref. 1) of transferred fragments during high speed sliding.

Conclusions

Transfer films of PTFE have been formed on nickel under controlled sliding conditions and analyzed with XPS; the following conclusions have been made:

1. The transfer film is chemically identical to bulk PTFE.

2. The only evidence of chemical interaction with the nickel is an extremely small XPS peak attributable to NiF_2 . This may be indirect evidence of a bond between the substrate and a very few sites on the PTFE molecule.

3. The film is unchanged by repetitive passes over the same area.

The following conclusions depend on the values of IMFP chosen for the calculations. The values obtained by Clark and Shuttleworth in the Journal of Polymer Science gave the most self-consistent results, and are taken to be correct. If they are not, the film thicknesses may be as great as eight molecular layers, but the conclusions are otherwise correct.

4. At speeds below 10 millimeters per second and loads up to 4 newtons, the transfer film is about one molecular layer thick, on the average.

5. Above 10 millimeters per second, the monomolecular film persists but is accompanied by the random transfer of fragments of bulk material.

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Cleveland, Ohio, April 21, 1980,
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16. Abstract <p>Polytetrafluoroethylene (PTFE) was rubbed against nickel in ultrahigh vacuum at loads up to 3.9 N (400 g weight) and speeds up to 94 mm/sec. The transfer film formed on the nickel was analyzed using X-ray photoelectron spectroscopy. It was found that the film was indistinguishable from bulk PTFE except for the possible presence of a small amount (<1%) of NiF₂. The transfer film was found to be about 1 molecule (0.5 nm) thick under all conditions; but at speeds above 10 mm/sec, there was evidence of bulk transfer in the form of fragments as well. The thickness measurements required a choice among conflicting published values of the inelastic mean free path for electrons in polymers. The values chosen gave internally consistent results.</p>			
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