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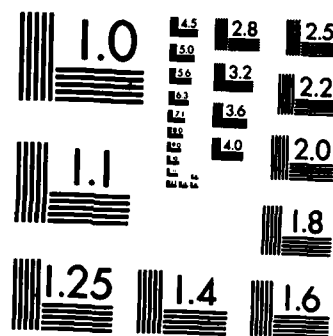
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Electroactive Polymers as Antistatic Materials

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*Surface Chemistry Branch
Chemistry Division*

February 7, 1983



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ELECTROACTIVE POLYMERS AS ANTISTATIC MATERIALS

INTRODUCTION

Precision instrument bearings and other delicate instrument components, such as electronic devices, are extremely sensitive to atmospheric particulate contamination. To prevent the accumulation of electrostatic charges during transport or storage, these components are often packaged in antistatic polyethylene or nylon containers. As shown, however, in earlier studies at this Laboratory (1,2), long-chain surface seeking surfactants, currently used as antistatic agents, produce adverse effects after extended contact with bearing steel surfaces and their lubricants: migration of the antistatic agent from the polymer surface to the lubricant and ultimately to the bearing surface causes physical or chemical changes in the lubricant and/or nonwettability of the bearing by the lubricant. Concern for improvement, therefore, elicited a search for alternate packaging materials whose conductive properties do not depend upon the incorporation of surface-seeking additives.

This investigation reports on electroactive polymers (EAPs) as a novel approach to conductive packaging materials employed for miniature precision bearings and other contaminant-sensitive devices. The concept of employing EAPs as antistatic materials is attractive because, assuming they themselves cause no contamination problems, no additional agents are present to migrate from the film surface to the packaged contents. Specially prepared EAPs as well as a commercial copolymer film were either chemically doped or ion-implanted with selected ions to optimize their surface conductivity and oxidation stability. Improvement of their physical properties, such as flexibility, was also addressed. Several newly available commercial antistatic packaging materials are also included in this study.

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The modified EAPs and commercial materials were placed in long term contact with lubricated and unlubricated bearing steels, after which the surfaces were examined for adverse effects. Specific attention was directed to a) any transfer from film to the steel substrate causing the steel surface to be nonwetable, b) any transfer from film to the respective lubricant resulting in altered physical or chemical characteristics of the latter, and c) physical properties and surface resistivities of the films.

MATERIALS

A. Films

Polymers examined were polyacetylene, $(CH)_x$, prepared at the Naval Research Laboratory, polyparaphenylene sulfide (PPS), and a copolymer of ethylene and methacrylic acid (COPOL); the latter two were obtained from commercial suppliers. Since unmodified $(CH)_x$ was susceptible to oxidation when exposed to air, samples were either chemically doped with 2% I^- or PF_6^- , or ion implanted with F^+ to improve their stability. To maintain flexibility during air exposure, $(CH)_x$ was deposited on polypropylene (PP) film prior to F^+ implantation. PPS and COPOL, although flexible and stable, were implanted with F^+ to decrease their surface resistivities.

Three flexible polyethylene-based films, and a more rigid gold-coated polyester film, all commercially available antistatic or conductive materials, were also examined. Composition and selected properties of the films are summarized in Table I. All properties of the commercial films were supplied by the vendors.

B. Substrates and Lubricants

Substrates were flats of 440C steels, representative of precision miniature bearing steels (3), metallurgically polished and passivated with sodium dichromate and nitric acid. To ensure surface-chemically clean surfaces, they were ultrasonically cleaned with acetone and fluorinated solvent immediately prior to use.

The four oils employed in this study are listed in Table II. They include: a base stock nonpolar synthetic hydrocarbon derived from the polymerization of α -olefins; a formulated version of the above synthetic hydrocarbon; a formulated synthetic hydrocarbon from a different base stock; and a formulated mixture of a polyolester and a diester (Military Specification MIL-L-81846). The formulations are designed for use with precision miniature bearings. All formulations contained small amounts of antioxidants and other additives.

EXPERIMENTAL TECHNIQUES

A. Preparation and Modification of EAPs

Polyacetylene was synthesized by a modified Shirakawa technique (4). Polyacetylene on oriented polypropylene was prepared by coating the substrate film on one side with an appropriate catalyst and then adding the acetylene.

Chemical doping with iodine was performed by contacting I_2 vapor with the $(CH)_x$ for about 15 hours. For PF_6^- doping, $NOPF_6$ was dissolved in nitromethane and the resulting solution was introduced to the $(CH)_x$. All reactions were carried out under vacuum.

The implantations were performed with a modified VARIAN/EXTRION ion implanter (5) capable of generating 25-200 KeV ions. The ions used were 25 KeV F^+ generated by fragmentation of BF_3 . To reduce effects caused by local

heating, the ion beam was maintained at 1-10 μ A while simultaneously cooling the samples. Rastering the beam across the sample ensured a homogeneous distribution of ions laterally across the surface. The samples were implanted to a total fluence of 1×10^{17} F⁺/cm². The energy and fluences used for these implants resulted in a band of implanted ions ~ 400 Å wide centered at ~ 300 Å below the surface, achieving a ~ 20 -50 atomic percent F⁺ content in the implanted region.

B. Long-term Compatibility with Unlubricated and Lubricated Bearing Steel Surfaces

Unlubricated and lubricated bearings as well as electronic devices are often stored in protective polymer packaging for extended time periods. To determine whether such long-term storage results in any interaction of the films with the bearing steels and/or the lubricants, clean surfaces and surfaces covered with a layer of each lubricant (approximately 0.5 mm thick) were placed in intimate contact with the respective films, care being taken to exclude air. After storage in darkness at $23 \pm 2^\circ\text{C}$ and $50 \pm 10\%$ RH for 3 to 5 months, films and substrates were separated and inspected visually for physical changes. Subsequently, the liquids remaining on the lubricant-covered surfaces were analyzed by reflectance Fourier Transform IR (FTIR); the unlubricated steel surfaces were examined by FTIR, X-ray photoelectron spectroscopy (XPS), a surface analysis technique sampling depths to 30 Å, and by contact angle (θ) measurements for wettability. Surface resistivity of the experimental ion implanted films was measured by a method similar to that contained in Military Specification MIL-P-82646 (6,7).

RESULTS

A. Surface Resistivity

The maximum surface resistivity permissible for antistatic packaging materials is 10^{12} Ω /sq. All films examined in this study, including the unmodified $(CH)_x$, exhibited values in the acceptable range (Table I). Surface resistivities of the F^+ implanted specimens displayed excellent values and compared favorably with the commercial metal-coated laminates.

B. Visual

Table III summarizes the effects of extended contact of the EAPs and polymer films with the unlubricated bearing steels as well as the lubricants spread on the steel surfaces. Visual examination showed that the undoped $(CH)_x$ film adhered strongly to the steel surfaces so that removal could be achieved only by prying off discrete small fragments of the embrittled film. Because of its porous structure, the $(CH)_x$ film completely absorbed every lubricant layer spread on the substrate with the exception of SHC-S which partially remained on the steel surface as discrete liquid droplets of unchanged color and viscosity. Contact of the I^- doped $(CH)_x$ film with either lubricated or unlubricated steel caused the appearance of a dark brown precipitate tenaciously adhering to the metal and removable only by metallurgical polishing; the lubricants, however, remained unchanged, and the films acquired some iridescence. Contact with the PF_6^- doped $(CH)_x$ did alter the film appearance, but did result in a marked viscosity increase of the contacted lubricants and an opaque adherent residue covering the steel substrates.

Implantation of the $(CH)_x$ film with F^+ ions greatly minimized such adverse interactions with lubricants and steels; all remained unaffected

after prolonged contact. Porosity of the $(CH)_x$ polymer which permitted seepage of the lubricants through the film was eliminated by preparing the $(CH)_x$ on PP backing prior to F^+ implantation; the film then became impervious to liquid penetration, assumed the flexibility of PP and remained inert to the steel substrate and lubricants. Equally promising results were obtained with F^+ ion-implanted PPS and COPOL.

None of the commercial materials showed any perceptible interactions. Of the commercially available materials, MYL + Au showed the least interaction with substrate and lubricants; all laminated films transferred some contaminants in varying degree to the clean steel surface.

C. Infrared Spectroscopy

After visual examination, the unlubricated steel surfaces were analyzed by reflectance FTIR for any residue resulting from film contact. Each spectrum was referenced against that of a clean steel surface to determine whether additional absorption bands were present, (Fig. 1). Features in the region of C = O bonds (Fig. 1a) indicate transfer of oxidized hydrocarbons from $(CH)_x$. The $(CH)_x + PF_6$ film produced fairly heavy transfers, shown by the presence of several additional absorption bands, including one at 800 cm^{-1} for the PF bond (Fig. 1b), whereas the spectrum after contact with $(CH)_x + F$ (Fig. 1c) shows no residual matter. A commercial PE-based film (Fig. 1d) exhibited some features in the hydrocarbon region. Such analyses for each system provided the data for the interactions listed in Table III under the FTIR heading.

The compositions of the lubricants remaining on the steel substrates were also analyzed by FTIR for possible component transfer. Each spectrum was compared with that of the pure lubricant spread on the steel surface (Fig. 2). When the spectrum of SHC-F after contact with $(CH)_x + PF_6$ (Fig. 2b) was compared with that of the pure SHC-F on steel (Fig. 2a),

additional features in the wavenumber range of 3400, 1650, 1170 and 850 cm^{-1} were observed, clearly establishing interaction of the film with the lubricant. However, the spectrum of SHC-F after contact with $(\text{CH})_x + \text{F}$ (Fig. 2c) was identical to that of the pure SHC-F, an indication that the lubricant remained unaffected.

D. Electron Spectroscopy

The unlubricated steel surfaces were also examined by XPS for contamination from the contacting films. Table IV lists data of the most prevalent elements on the bearing steel surfaces, both freshly cleaned and after contact with several representative films. The intensities and the line positions serve as indicators of quantity and chemical bonding. Predominant elements were the steel constituents Fe and O. A binding energy (BE) for O at 530 eV is indicative of iron oxide (8) and at 532-533 eV of organic C-bonded O such as carbonyls, alcohols or esters (9). A definite shift of the predominant O peak to a higher BE was observed after contact with the unmodified $(\text{CH})_x$ film, which was not too surprising, given the ready oxidizability of $(\text{CH})_x$. A BE of 285 eV for C indicates an aliphatic C-C linkage and is, therefore, characteristic of an almost ubiquitous atmospheric hydrocarbon contamination overlayer, whereas a BE of about 289 eV is characteristic of C bonded to O (10). Such shifts toward higher BE for C in conjunction with the shifted O signals suggest the presence of organic contamination other than atmospheric. In addition to Fe, O, and C, large signals of F and traces of P were observed in the steel surface after contact with $(\text{CH})_x + \text{PF}_6$. Signals of ionic F^+ and traces of F bonded to C were also evident from contact with $(\text{PPS}) + \text{F}$, signifying some interaction between $(\text{PPS}) + \text{F}$ and steel. With the exception of the $(\text{CH})_x + \text{PF}_6$ specimen, the essentially unvarying C intensities indicate only traces of contaminants from the films.

E. Wettability

Wettability of the unlubricated steel surfaces was determined by contact angle measurements. On a freshly cleaned metal or metal oxide surface, water and $C_{14}H_{30}$ spread spontaneously ($\theta=0^\circ$), and CH_2I_2 exhibits $\theta < 20^\circ$ (11). Contact angles larger than those values are indicative of foreign material covering the surface. Table V shows the effect on wettability from contact with some of the films studied. Values similar to those of $(CH)_x$ and several of the other films are characteristic of a layer of atmospheric hydrocarbon contamination, an indication that these materials did not appreciably affect the wettability of the steel surface. The larger values observed after $(CH)_x + F$ on PP contact suggest contamination resulting from O-containing compounds such as esters (12). Although F was detected by XPS from $(PPS) + F$ contact, it did not affect the wetting of the steel surface, whereas the values from $PE + Al$ contact suggest some organic contamination. The erratic behavior on the $(CH)_x + PF_6$ contacted surface was in keeping with the erratic results obtained by the other analyses. There was generally good correlation between the XPS and the wettability results.

SUMMARY AND CONCLUSIONS

Flexible antistatic film packaging is, in a variety of cases, a requirement for components which must be protected against spark discharge or particulate contamination. Present commercial antistatic packaging for the most part meets these requirements. However, where contamination from the antistatic agents themselves causes problems, new approaches to such packaging are useful. EAPs, because they do not contain an active mobile phase, are one such approach. Because not all properties of unmodified EAPs are suitable for packaging materials, this investigation sought to impart improvements by selective chemical doping and ion implantation. Implanted EAPs were found in many instances to display improved stability,

conductivity, flexibility and heat sealability (on backing if necessary). In those cases where contamination from the film cannot be tolerated (lubricated bearings, etc.) the implanted EAPs show promise. Further research, both with regard to the preparation of the EAPs and their implantation would probably result in even greater improvements than those reported here. It must be pointed out, however, that present implant technology and facilities are not yet adequate to produce commercial quantities of film, nor to compete with available antistatic materials with regard to cost. The potential of these films, however, is such as to justify their further study.

ACKNOWLEDGMENTS

The authors want to thank our colleague, Robert Mowery, for obtaining the FTIR spectra, and Gary O. Head of Lear Siegler, Inc. for performing the surface resistivity measurements on the electroactive polymers and copolymers.

Table I
PHYSICAL PROPERTIES OF FILM MATERIALS

Composition	Code	Flexibility	Thickness (mil)	Surface Resistivity (Ω/sq)
$(\text{CH})_x$	$(\text{CH})_x$	flexible	~ 4	3×10^{11}
$(\text{CH})_x \text{I}^-$ doped	$(\text{CH})_x + \text{I}$	fairly flex.	~ 4	-
$(\text{CH})_x \text{PF}_6^-$ doped	$(\text{CH})_x + \text{PF}_6$	stiff	~ 4	-
$(\text{CH})_x \text{F}^+$ implanted	$(\text{CH})_x + \text{F}$	fairly flex.	~ 5	2×10^7
$(\text{CH})_x \text{F}^+$ implanted, on polypropylene	$(\text{CH})_x + \text{F}(\text{PP})$	flexible	3	5×10^5
$(\text{PPS}) \text{F}^+$ implanted	$(\text{PPS}) + \text{F}$	flexible	3	1×10^5
Copolymer ethylene/methacrylic acid, F^+ implanted	$\text{COPOL} + \text{F}$	flexible	5	7×10^7
Polyethylene with slip agent	PE	flexible	4	3×10^9
Polyethylene with antistat + mylar + Al-Ti + anti-abrasive	PE-Al-Ti	flexible	3	$< 10^{11}$
Polyethylene with antistat + Al + urethane	PE-Al	flexible	3	$< 10^{12}$ (int) $< 10^6$ (ext)
Mylar + Au + ceramic	MYL + Au	bendable	5	1.6×10

Table II

LUBRICANTS

Type	Code
Synthetic Hydrocarbon, base stock	SHC-B
Synthetic Hydrocarbon, formulated ^{a)}	SHC-F
Synthetic Hydrocarbon, formulated ^{b)}	SHC-S
Polyolester - Diester, formulated	SPDE

a) Base stock is SHC-B

b) Different base stock than SHC-B

Table III

EFFECT ON BEARING STEELS AND LUBRICANTS AFTER
EXTENDED CONTACT WITH FILMS

Film	Lubr.	Visual			FTIR
		Film	Lubricant	Steel	
(CH) _x	None	brittle	-	adherent film	unaffected
	SHC-B	brittle	disappeared	adherent film	-
	SHC-F	brittle	disappeared	adherent film	-
	SHC-S	brittle	diminished	oily droplets	lubr.
	SPDE	brittle	disappeared	dry	unaffected clean steel
(CH) _x + I	None	brown ppte	-	brown ppte	unaffected
	SHC-B	iridescent	unaffected	brown ppte + lubr.	lubr.
	SHC-F	unchanged	unaffected	brown + lubr.	unaffected
	SHC-S	iridescent	unaffected	brown + lubr.	unaffected
	SPDE	iridescent	disappeared	brown dry	unaffected clean steel
(CH) _x + PF ₆	None	iridescent	-	opaque adherent	interaction
	SHC-B	unchanged	thickened	opaque adherent	interaction
	SHC-F	unchanged	thickened	opaque adherent	interaction
	SHC-S	unchanged	thickened	opaque adherent	interaction
	SPDE	unchanged	thickened	opaque + lubr	interaction
(CH) _x + F	None	unchanged	-	unaffected	unaffected
	SHC-B	lubr seepage	unaffected	unaffected	lubr.
	SHC-F	lubr seepage	unaffected	unaffected	unaffected
	SHC-S	lubr seepage	unaffected	unaffected	unaffected
	SPDE	lubr seepage	unaffected	unaffected	lubr. unaffected lubr.

Table III (Cont'd)

Film	Lubr.	Visual			FTIR
		Film	Lubricant	Steel	
(CH) _x + F (PP)	None	slightly darkened	-	unaffected	-
	SHC-F	darkened	unaffected	unaffected	lubr. unaffected
	SPDE	darkened	unaffected	unaffected	lubr. unaffected
(PPS) + F	None	unaffected	-	unaffected	-
	SHC-F	unaffected	unaffected	unaffected	lubr. unaffected
	SPDE	unaffected	unaffected	unaffected	lubr. unaffected
COPOL + F	None	unaffected	-	unaffected	-
	SHC-F	unaffected	unaffected	unaffected	-
	SPDE	unaffected	unaffected	unaffected	-
PE	None	unaffected	-	smudge	HC contamin. lubr.
	SHC-B	unaffected	unaffected	unaffected	unaffected
	SHC-F	unaffected	unaffected	unaffected	lubr. unaffected
PE-A1-Ti	None	unaffected	unaffected	unaffected	unaffected
	None	unaffected	-	unaffected	unaffected
	None	unaffected	-	unaffected	unaffected
MYL + Au	None	unaffected	-	unaffected	unaffected
	SHC-B	unaffected	unaffected	unaffected	lubr. unaffected
	SPDE	unaffected	unaffected	unaffected	unaffected
					unaffected

Table IV

XPS RELATIVE ELEMENTAL INTENSITIES OF BEARING STEEL SURFACE
AFTER CONTACT WITH FILMS
(Normalized to Fe-10)

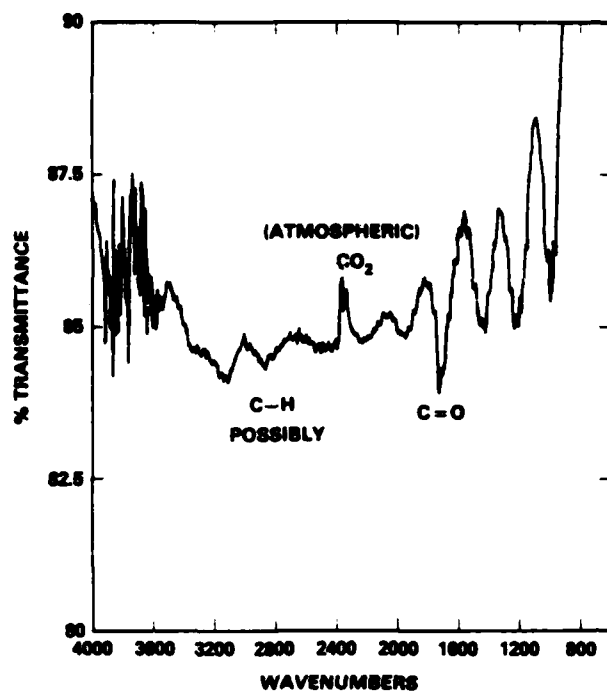
Film	Intensities at Binding Energies (eV)						
	O(ox) (530)	O-C (532)	C-C (285)	C-O (289)	F (685)	F-C (688)	P (133)
None	23.1	-	3.0	-	-	-	-
(CH) _x	9.0	17.8	3.0	3.0	-	-	-
(CH) _x + PF ₆	10.0	trace	2.5	0.3	15.5	-	0.4
(CH) _x + F (PP)	26.2	trace	3.8	4.0	-	-	-
(PPS) + F	25.6	-	4.2	-	1.3	0.8	-
PE - Al	21.7	trace	3.3	1.1	-	-	-
PE-Al-Ti	23.3	trace	4.5	1.1	-	-	-

Table V

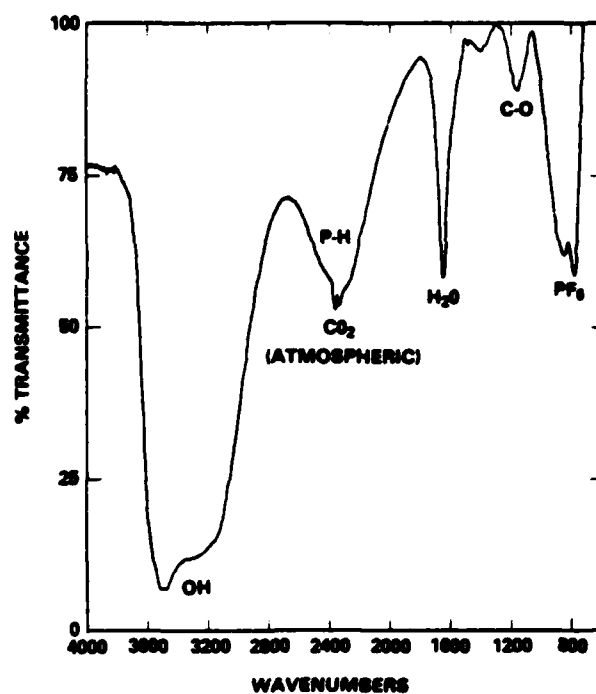
WETTABILITY OF STEEL SURFACE AFTER CONTACT WITH FILMS

Film	Contact Angle (°)		
	H ₂ O	CH ₂ I ₂	C ₁₄ H ₃₀
(CH) _x	52	40	spreads
(CH) _x + PF ₆	30 ^a	39 ^b	spreads ^c
(CH) _x + F	50	39	spreads
(CH) _x + F (PP)	78	46	spreads
(PPS) + F	55	35	spreads
PE - Al	66	44	spreads
PE - Al - Ti	54	40	spreads

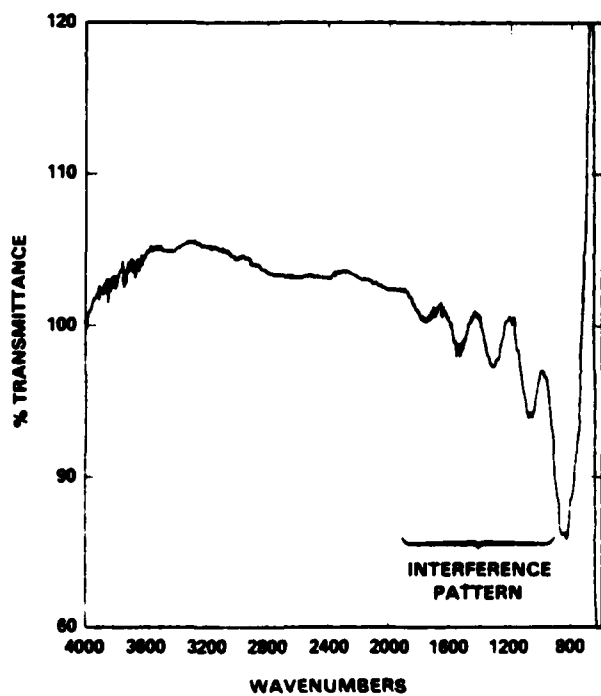
^adrop interacts^bdrop sinks into residue^cdrop dissolves residue



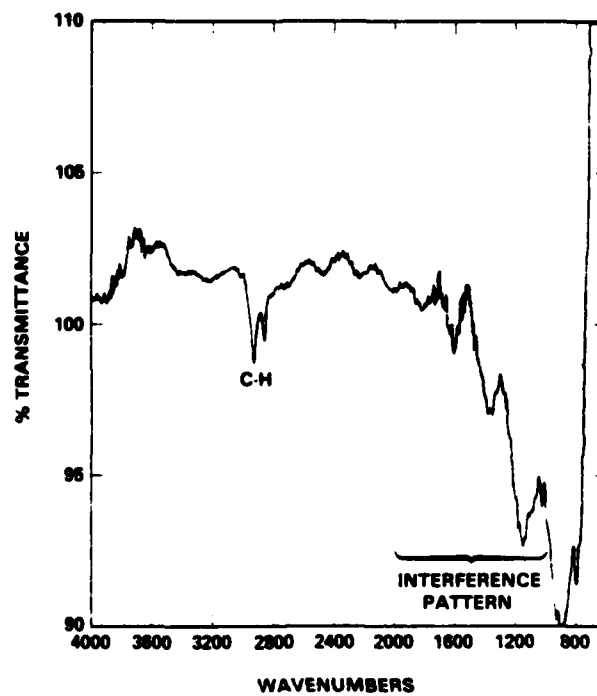
a) $(CH)_x$



b) $(CH)_x + PF_6$

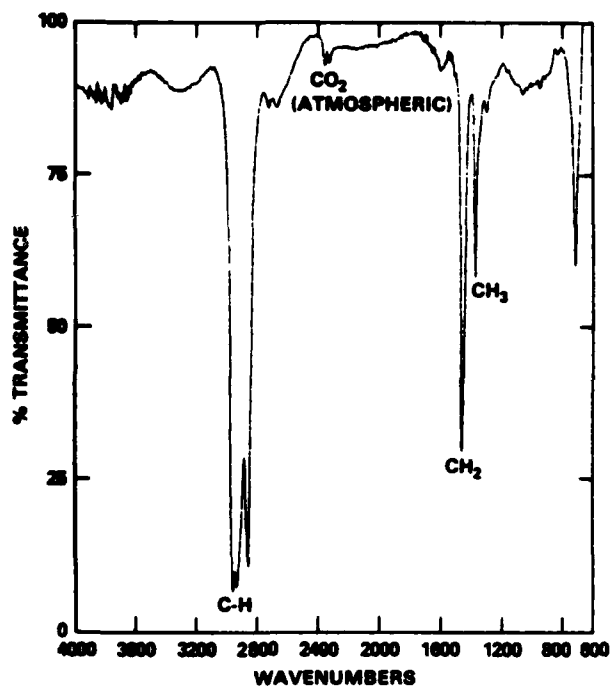


c) $(CH)_x + F$

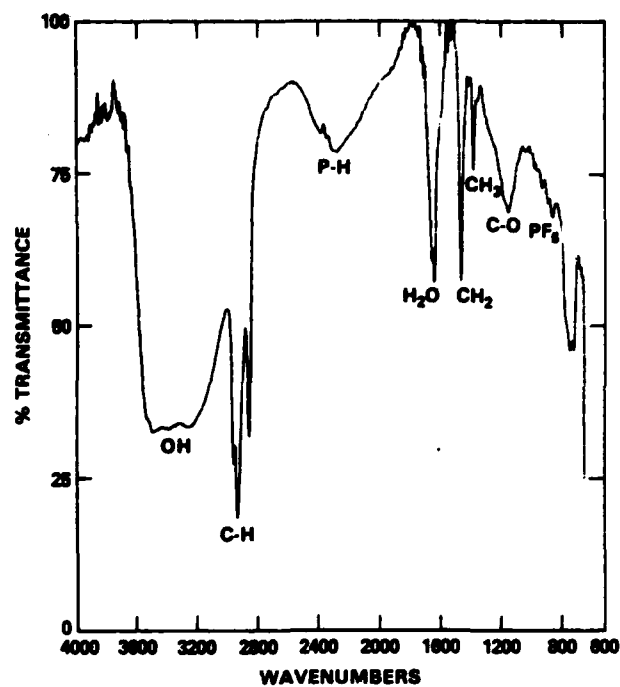


d) PE

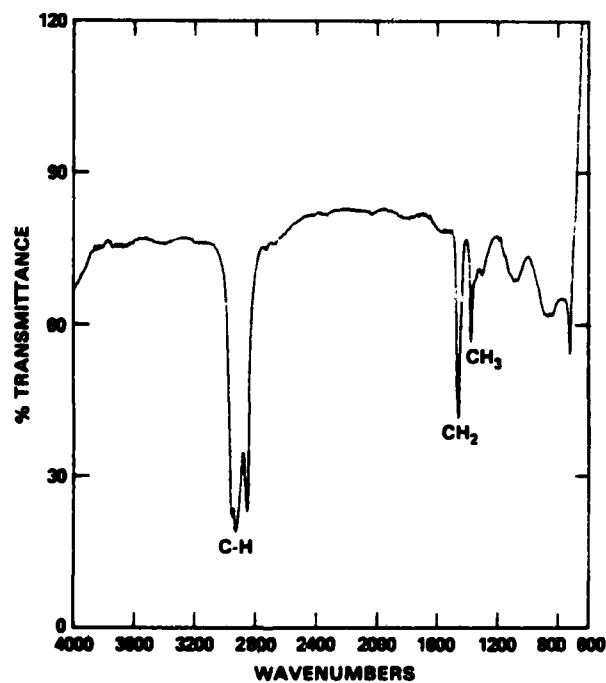
Fig. 1: Contamination transfer from films to clean unlubricated 440C steel surface after 5 months contact as monitored by FTIR



a) SHC-F



b) SHC-F after contact with
 $(CH)_x + PF_6$



c) SHC-F after contact with
 $(CH)_x + F$

Fig. 2: Contamination transfer from films to lubricant after 5 months contact as monitored by FTIR

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