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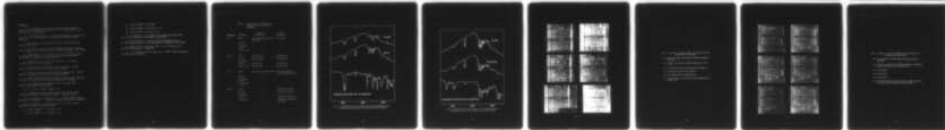
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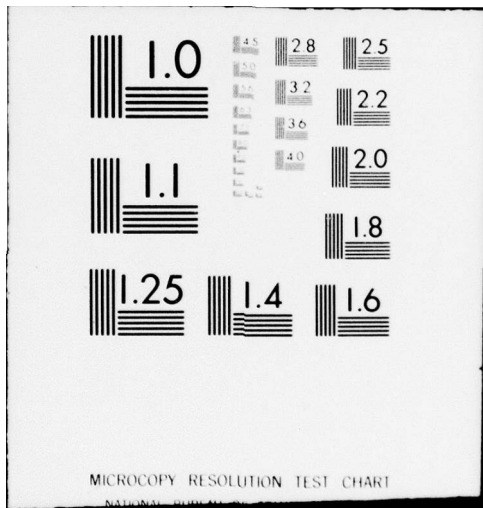
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NRL Memorandum Report 3873

Problems Encountered with Antistatic Packaging

B.J. Kinzig and H. Ravner

*Surface Chemistry Branch
Chemistry Division*

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Packaging of miniature aerospace components in antistatic polyethylene or nylon is generally considered an attractive route to eliminate major problems in their handling and storage. Although most electronic components appear compatible with antistatic packaging materials, lubricated parts, such as precision ball bearings, can apparently be adversely affected. Surfaces exposed to lubricants, for example, may become non-wetted or the lubricant become grease-like after relatively short exposure to antistatic containers. That such packaging adversely affects lubricated parts suggests (Continues)		

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20. Abstract (Continued)

potential problems involving unwanted surface-active effects for non-lubricated parts as well.

Our recent studies indicate that the surfactant material incorporated into the packaging film may be responsible for the adverse effects observed.

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PROBLEMS ENCOUNTERED WITH ANTISTATIC PACKAGING

BACKGROUND

Precision miniature ball bearings (as well as other delicate dust and moisture sensitive components) require packaging in materials that can be used in a clean room environment as well as preserve the special operational characteristics of the components throughout transport and storage. The need for clean room conditions poses special requirements for packaging materials: they must be dust-proof, moisture-proof, have a low rate of particle sloughing, be cleanable, and be inert to the cleaning solvents and lubricants. Additionally, their properties of transparency, light weight, low volume, toughness, and the ability to be heat-sealed, make several polymers (polyethylene, Mylar and nylon) materials of choice for clean packaging.

A major drawback associated with the use of these non-conducting polymers is the build-up of large electrostatic charges on their surfaces (1). This static electricity is generated when contact is made and broken with a polymer surface; thus any kind of handling or movement can be a source of charging. Once charged, the non-conducting polymer has no path for dissipation of the charge, allowing its build-up to high levels (2). These charges can tenaciously attract dust and damage contained parts by spark discharge (3).

Polyethylene and nylon film are now available with antistatic additives incorporated directly into the films (3,4,5). Packaging fabricated from these materials effectively decreases or eliminates the static electricity and the concomitant problems from dust and charging (5). These antistatic materials have gained wide acceptance, particularly in clean room operations and handling of electronic components for the aerospace industry. Bags made from antistatic films have recently been introduced for Navy precision miniature ball bearings, and are procured under current military specifications for electrostatic-free packaging materials (6-9).

Long chain surfactants are used as antistatic additives in polymer packaging films. These surface-seeking and surface-active agents are incorporated into the resin formulation during extrusion in about 0.5 percent concentration (10). A microscopic layer of the surfactant is generated on the film surface; if removed, this film regenerates at the surface from the "reservoir" in the bulk film. The surfactant has polar groups, and those which arrive at the film surface are hygroscopic,

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attracting water from the atmosphere. The polar groups and the water create an electrically conducting surface on the polymer, allowing the dissipation of static charges.

To be effective, an antistatic surfactant additive must be:

- 1) compatible with the polymer so that desirable film properties such as toughness and flexibility are not weakened;
- 2) amphipathic, having an affinity for water by containing both polar and non-polar portions;
- 3) surface-seeking when incorporated into a bulk film;
- 4) non-reactive with the polymer.

Although current additives conform to these criteria and effectively reduce surface charging, there are problems associated with their use. These may be observed in miniature bearings and other control system components in which lubrication with minimal quantities of lubricant is required. When these components come into contact with the surface active additive on the packaging films, the additive may be transferred into the lubricant or onto the metal if it has an affinity for the lubricant base stock, its formulation additives, or the metal. Precision bearings are typically lubricated with as little as 1 mg of oil. Thus a transfer to the oil of only 10 micrograms of antistatic agent (the amount calculated to be on the inside surfaces of a 2" x 2" bag) would be needed to create a contaminant concentration in the oil of one percent, a level competitive with the additives in the formulated lubricant itself. This amount of surfactant can easily alter carefully formulated lubricant properties. Antistatic additives can be transferred directly to metal bearing surfaces by rubbing contact with the package or by adsorption from the lubricant, resulting in the formation of a low energy surface film non-wettable by the lubricant. If the balls or raceway are affected, premature bearing failure due to lubricant starvation would be a likely result.

Miniature bearings are sometimes packaged in an excess of oil to be centrifuged away prior to use. This practice allows much more oil - packaging film contact, and may allow an even larger amount of antistatic additive to be leached from the package onto the metal, enhancing the possibility of lubricant starvation.

The antistatic properties of the packaging itself can be destroyed by contact and interaction with oils. Silicone oils, for example, were shown to be impossible to remove from an antistatic film surface, resulting in the permanent impairment of the antistatic properties of the films (2).

The problem of interaction of packaging materials with their contents is not new; over 30 years ago, for example, the waxed paper used as a moisture barrier wrap was traced to be a source of bearing contamination (11). In light of the present need for greater reliability of precision components in sophisticated instrumentation, the consequences of additive interactions with lubricants or metal surfaces become more significant. The study of some of the more relevant interactions involved is the subject of this investigation.

EXPERIMENTAL

Materials

Four types of packaging films, 6-mil polyethylene (PE) with and without an antistatic (AS) additive, and 2-mil nylon (NY) with and without an antistatic additive were obtained as 2" x 2" bags from Naval facilities and as sheet film from the manufacturer.

Lubricants KG-80, SRG-160, unformulated base stock of KG-80 (all "superrefined" mineral oils), and Terresso V-78 (a formulated mineral oil) were obtained from Navy sources.

Discs of 52100 steel 1" diameter x 1/8" thick were polished to a mirror finish with 0.3 μ m alumina before use.

Polyethylene powder "Microthene FN 510" was used to melt press an experimental film containing the additive normally used in antistatic nylon.

Methods

Infra-red (IR) spectra were obtained for all materials; polymer samples were studied as bulk films, and liquids as films between salt plates. Fourier transform IR with attenuated total reflection (FTIR/ATR) was employed to give enhanced information on polymer surface composition. Lubricant-antistatic additive interactions with metal surfaces were studied in thick films deposited on polished 52100 steel specimens. Long-term effects were studied by storing steel specimens in the dark, lubricated side up, in 2" x 2" polymer bags at ambient conditions (ca. 24°C and 50% R.H.). Photographic records were made of changes at lubricant-metal interfaces.

RESULTS

FTIR/ATR Spectroscopy

FTIR/ATR data were obtained on AS and non-AS PE and NY packaging film samples. Subtracting the PE spectra from that of the AS PE revealed the presence of the AS additive on the surface, Figure 1; similar results were obtained with the NY and AS NY spectra showing the AS NY additive as a surface component of the polymer film, Figure 2.

Effects of Lubricant-Covered Metal Specimens in Contact with Packaging Films

Specimens of 52100 steel with the test oils laid down as films on the polished surfaces were stored for one year in contact with the four types of polymers, as described above; the results are summarized in Table 1.

The samples in PE and NY were unaffected by the packaging; lubricant films remained fluid and wetted the metal surfaces after the full storage period. Samples exposed to 60°C for one hour after 24 hours contact were likewise unaltered.

All of the lubricant fluids studied interacted with the AS PE bags, retracting from the metal surfaces after a few days. KG-80 base stock and formulated fluid reacted more rapidly than the more viscous SRG-160. After 24 hours at room temperature, heating at 60°C for one hour showed no further change in their surface appearance. Storage of these samples at room temperature for one year, however, caused the lubricants to retract further, indicating the interaction continued during the long term storage.

Oil films on the metal surfaces in AS NY bags showed no changes either in the short term storage, or after heating for 1 hour at 60°C after 24 hours contact. Storage for one year, however, caused two effects to occur: (a) the metals became partially non-wetted, and the oil thickened, becoming sticky and grease-like, and (b) the side and bottom surfaces of the 52100 discs, which were not originally treated with oil, rusted in the AS NY bags. Rust was also observed on samples in the non-AS NY bags, but those in both types of PE were unaffected. The low moisture barrier properties of nylon film which is likely responsible for the rusting, may contra-indicate the use of nylon for packaging anticipated to be in a high r.h. environment. (One of the anomalies, to be discussed later, is that nylon, even with the AS additive, may not become antistatic until it is exposed to r.h. levels high enough so that its ineffectiveness as a moisture barrier could itself be a severe problem (2)).

Interaction of Lubricants with Antistatic Additives on Glass Slides

KG-80 and SRG-160 lubricants were applied to cleaned glass slides, forming smooth pools of liquid. The viscous liquid antistatic additive used in AS PE film was then applied in several drops to the lubricant surfaces. Photographs were taken of the slides at ca 10 minute intervals. As seen in Figure 3, there was a dramatic retraction of the lubricant from uniform films into non-wetting droplets, indicating the antistatic additive effectively displaced the oils from the surface, leaving a film of the additive on the glass. This displacement occurred in about 1 hour, after which little further change occurred. In this experiment, to illustrate in a more obvious manner the displacement

principle, the amount of AS additive in contact with the surface was probably larger than that expected to be encountered in packaging practice; the displacement was also seen on the metal specimens listed in Table 1.

A similar experiment was performed with KG-80 and SRG-160 lubricants and the AS NY additive. The additive, a waxy solid at room temperature (m.p. 46°C), was dissolved in an isopropanol/acetone mixture, and again applied dropwise. Figure 4 shows the photographic time study of this series. Precipitation of the additive caused opaque spots, as seen in the early photographs. The slide was heated gently to melt the precipitated additive; at that point, retraction of the oils occurred, again showing some displacement by the additive. The oils became grease-like and thickened as well as being displaced.

Preparation of Experimental Polyethylene Film

Polyethylene powder ("Microthene FN 510") was mixed with a ca 5 wt percent isopropanol/acetone solution of the AS additive used for NY to form a paste, and allowed to dry. The mixture was melt-pressed at 170°C and 2500 psi to a film. 52100 steel was satisfactorily wetted by both KG-80 and SRG-160 after 72 hours of contact with this new AS PE film. After 2 hours at 60°C, slight non-wetting occurred. After storage for 1 year the oils became sticky and grease-like. While this thickening could in part be due to the higher than usual concentration of additive, it nevertheless indicates the tendency of the waxy solid to thicken the oil at room temperature.

DISCUSSION

The laboratory studies described all indicate that the liquid antistatic agent used in PE packaging can readily transfer to a lubricated metal or glass surface, causing mineral oil-based lubricants to become non-spreading. The AS NY additive becomes a soft solid in contact with the lubricants studied, and is slower than the AS PE additive to displace the oil from the metal. Evidently the higher melting point and lower mobility of the AS NY additive makes it less likely to transfer to a metal surface when incorporated into either nylon or polyethylene, and/or to displace an oil from that surface. This additive is also more paraffin-like and less polar than the AS polyethylene additive and has some attraction for the polymer as well, which results in its lower mobility and lower transfer rate. We find, however, that eventually this material adsorbs on the metal surface, but not as much as the more polar and mobile surfactant. The paraffin-like mineral oils, having fewer or no polar groups to compete for the surface, are likely to be displaced by any of the antistatic agents more readily than more polar ester oils, but other categories of lubricants, e.g. esters, also appear to be able to interact with the packaging additives to some extent.

The lesser interaction of nylon antistatic additives with the lubricants studied would seemingly make the AS NY combination a sound choice. Nylon, however, is itself a poor moisture barrier, and this probably is responsible for the rusting noted earlier of initially non-lubricated portions of the 52100 specimens stored in NY bags. Further, there is some indication that a fairly large amount of water is necessary for films with AS additives to have antistatic properties. Woods (2) indicates that nylon with AS additive requires r.h. \geq 50% to be effectively non-conducting. Anderson (1) states that nylon alone picks up enough water at r.h. values \geq 40% to be antistatic, even without additives. Our studies took place at a fairly constant r.h. of 50%, where the antistatic properties should thus be operative. The high attraction for water and the potential for moisture problems inside the packaging, however, indicate caution should be used in the use of this material especially for long term storage and/or for use in high r.h. conditions.

When items are double-wrapped, i.e., an intermediate wrap is encased in an outer wrap, the charge from the inner wrap must be dissipated for the protection of the contents. Grounding techniques to assure bleed off of charge from the intimate packaging are discussed by Anderson (10). He suggests sealing a fine wire or mesh or other conducting material to extend from the antistatic inner wrap to the outside of an outer wrap which may not itself be antistatic.

Since the antistatic properties depend primarily on surface conductivity, anything that interferes with or coats over the packaging film will adversely affect those properties. Silicones and other oils have such an adverse masking effect. Antistatic polyethylene contaminated with silicone oil has been shown to develop and hold a charge of 15 KV (2), and once present, the oil cannot be readily removed.

There are a number of approaches which can be employed to create an antistatic polymer surface. While some allow for an entirely conducting polymer (12,13), most require the incorporation, or at least coating, of a polymeric or fatty acid derivative material having a fair degree of polarity into the polymer film. The activities of some additives are further enhanced by various radiation treatments (14).

Very little information on antistatic additives is reported in the U.S. literature; the bulk of information is from foreign patents usually issued on the chemical composition of the additives themselves (15-19). There is some notice taken in the open literature of the specific problem of the untoward effects of the use of antistatic additives in the packaging of foods (20,21); new regulations concerning their use are in the making (22). The toxicity of antistatic additives with respect to their use in toys as well as in food packaging has also been investigated (23).

SUMMARY AND CONCLUSIONS

We have demonstrated that a variety of liquid lubricants (base stocks and formulated mineral oils) interact with antistatic surfactant additives from packaging materials, resulting in surfaces no longer wetted by the lubricant. An additive used in PE films causes rapid non-wetting and retraction of the lubricant. The AS additive used in NY packaging films interacts more slowly, but deleterious effects were noted after one year of storage. Oils exposed to this additive become sticky or grease-like.

In the case of precision miniature bearings, antistatic additive transfer from packaging films, resultant interactions, and the concomitant lubricant starvation on contacting surfaces may result in premature bearing failure. The incorporation of an extraneous surface active agent into carefully formulated oils may change critical oil properties such as stability, viscosity, or clarity, and may also be associated with a reduction in performance. Since precision bearings are often lubricated with a very small quantity (one mg) of lubricant, it requires extremely small quantities of an extraneous additive to change perceptibly the composition and thus the properties of the lubricant. The development of antistatic packaging materials for bearings which would not interact significantly with the lubricants is thus a matter of considerable importance.

The observed interaction of presently used antistatic surfactants and lubricants suggests a number of areas for further study:

- 1) The nature of the interaction should be defined - additional studies might reveal whether simple displacement or more complex reactions are occurring on lubricated surfaces.
- 2) Surface active antistatic additives to be used for bearing packaging should minimize non-wettability of the bearing metal surfaces, and not interact adversely with the lubricants used. The practice of storing and shipping bearings in an excess of oil (to be centrifuged off later) contained in antistatic bags should be examined for possible changes in lubricant composition and effectiveness from this lubricant/surfactant contact.
- 3) The possible effects of transfer of AS additives from packaging onto non-lubricated components should be examined.
- 4) Grounding methods, and combination packages may need to be examined, for a combination having best moisture barrier as well as best antistatic properties. This is especially important with the use of nylon, and with corrodable contents such as precision bearings.

5) Further studies are needed in general, on the effects of environment such as humidity, corrosive or salty atmospheres, heat, and UV radiation to determine the effects of these variables on the packaging of critical components.

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Table I. Interaction of Lubricants on
52100 Steel with Antistatic
Packaging

<u>Bag Type</u>	<u>Lubricant</u>	<u>1 Week Interaction</u>	<u>1 Year Interaction</u>
PE	KG 80	Lubricant satisfactorily wets steel	No change
	SRG 160	"	"
	KG 80 base stock	"	"
	Teresso V78	"	"
AS PE	KG 80	Non-wetting	Non-wetting
	SRG 160	Some wetting	Non-wetting
	KG 80 base stock	Non-wetting	Non-wetting
	Teresso V78	Non-wetting	Non-wetting
NY	KG 80	Wets steel satisfactorily	Wets and rusts on non-lubricated edges
	SRG 160	"	"
	KG 80 base stock	"	"
	Teresso V78	"	"
AS NY	KG 80	"	Thickens and rusts
	SRG 160	"	Thickens and rusts
	KG 80 base stock	"	Thickens and rusts
	Teresso V78	"	Thickens, non-wetting and rusts

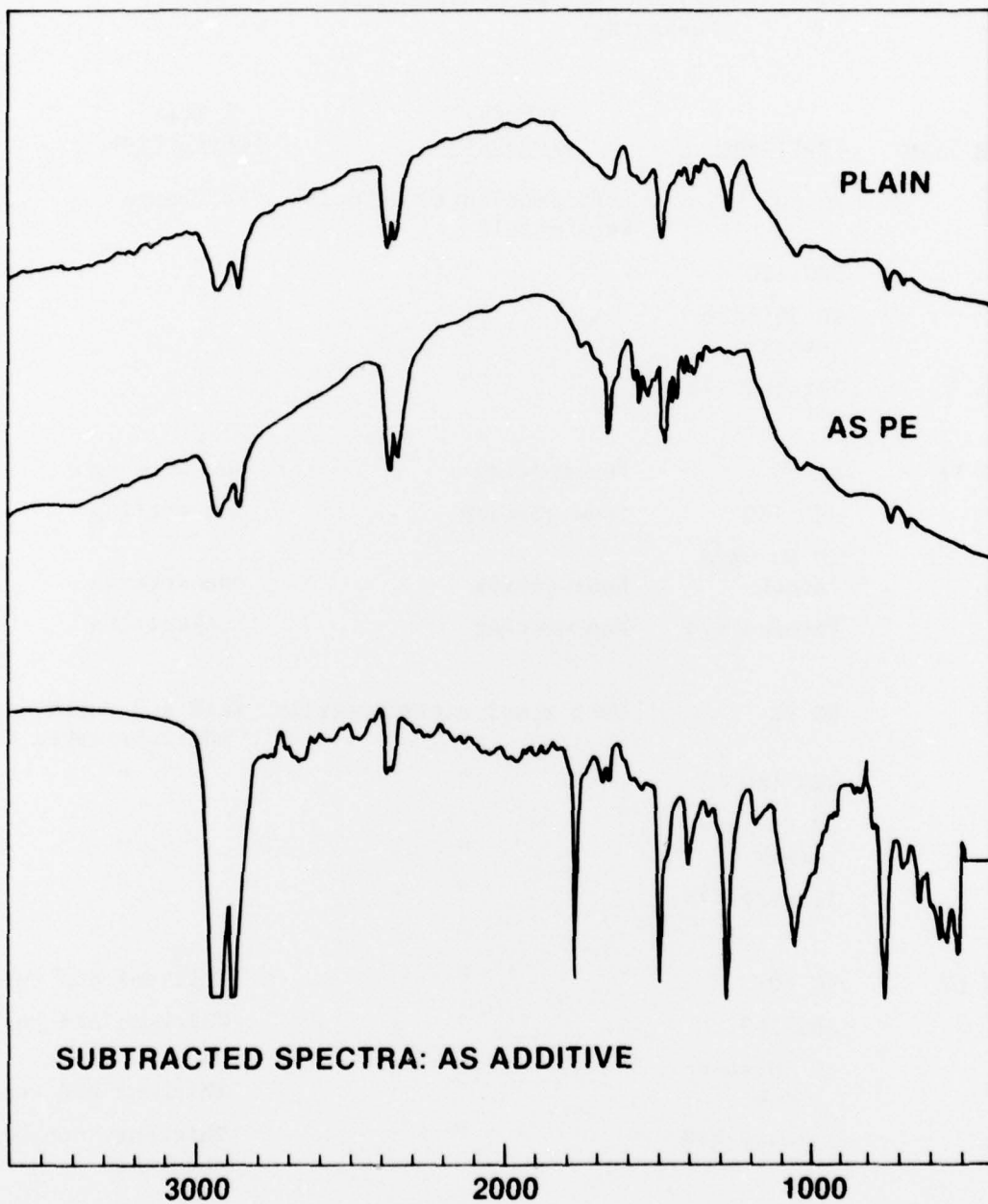


Fig. 1 — FTIR/ATR spectra of the surfaces of AS PE and ordinary PE, and the subtracted spectra showing the additive on the AS PE surface

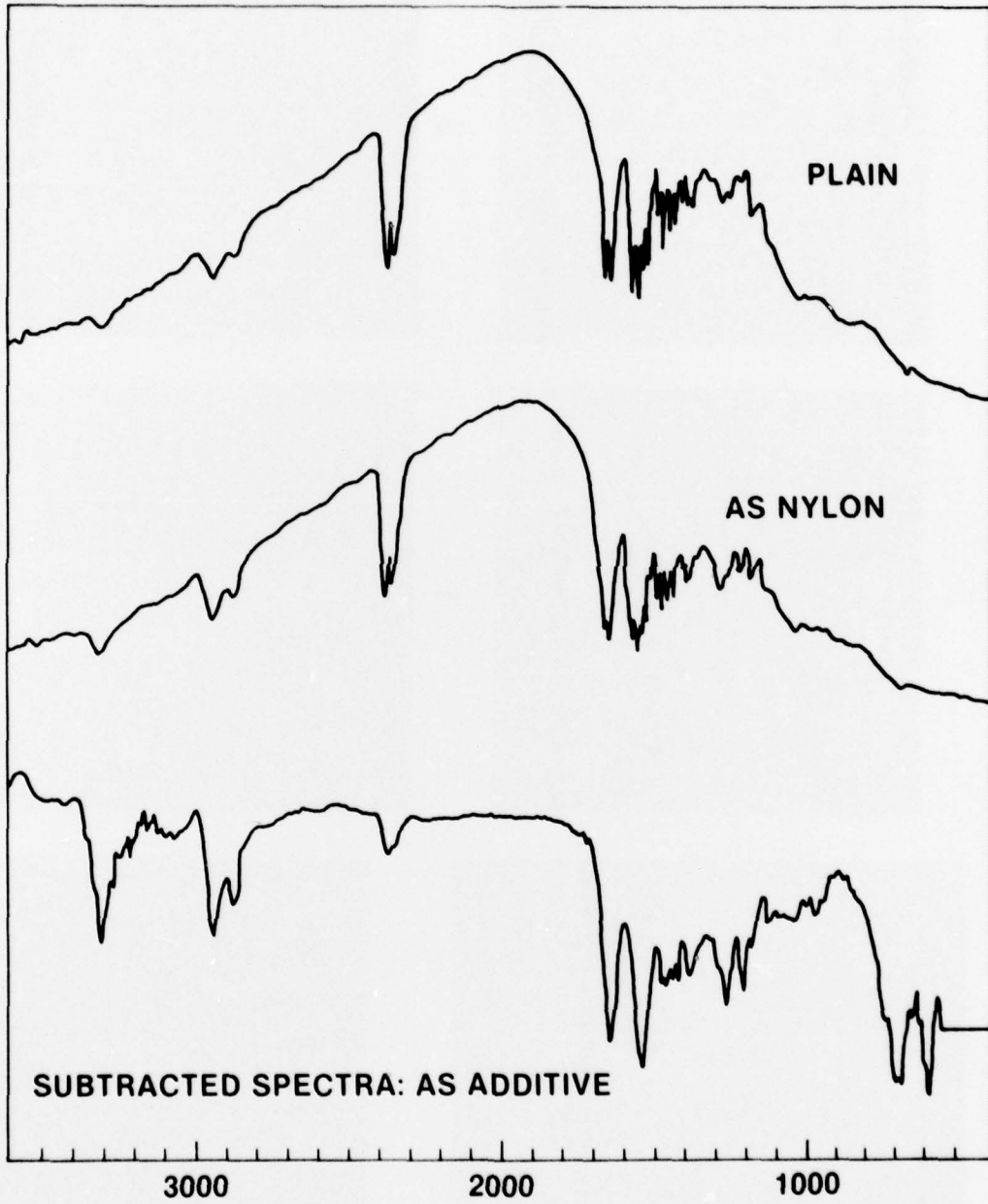


Fig. 2 — FTIR/ATR spectra of the surfaces of AS NY and NY, and the subtracted spectra showing the additive on the AS NY surface

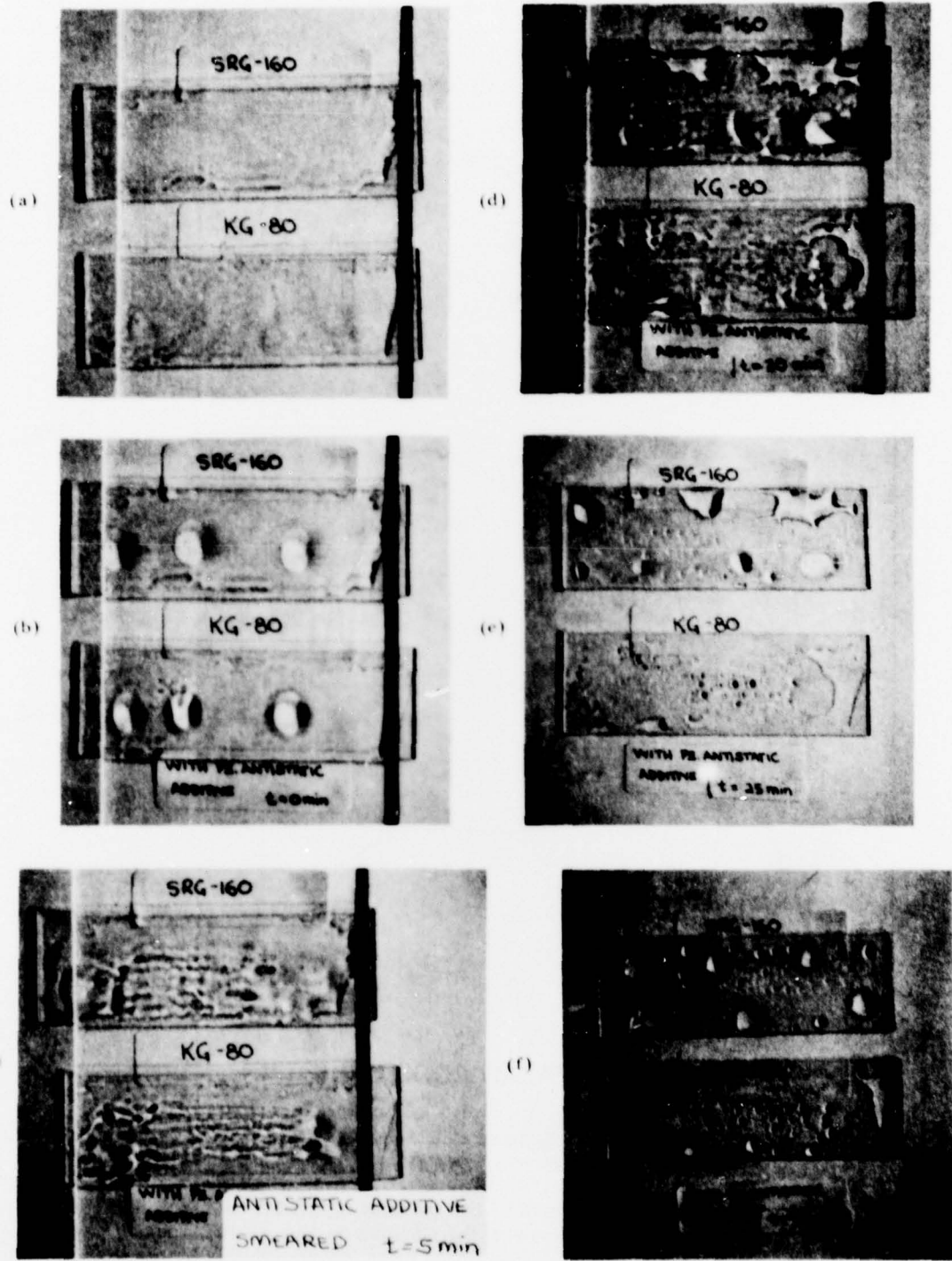


Fig. 3

Fig. 3 — Interaction of antistatic additive of AS PE with KG-80 and SRG 160 on glass slides

- (a) KG-80 and SRG-160 lubricants are spread on glass in a thick film
- (b) 3 drops of the antistatic additive were placed on oil at $t = 0$
- (c) at 5 minutes, the drops were smeared with a glass rod
- (d) at 20 minutes both oils have retracted
- (e) at 25 minutes further retraction occurs
- (f) at 70 minutes oil is in droplets on a thin base film; little change occurs

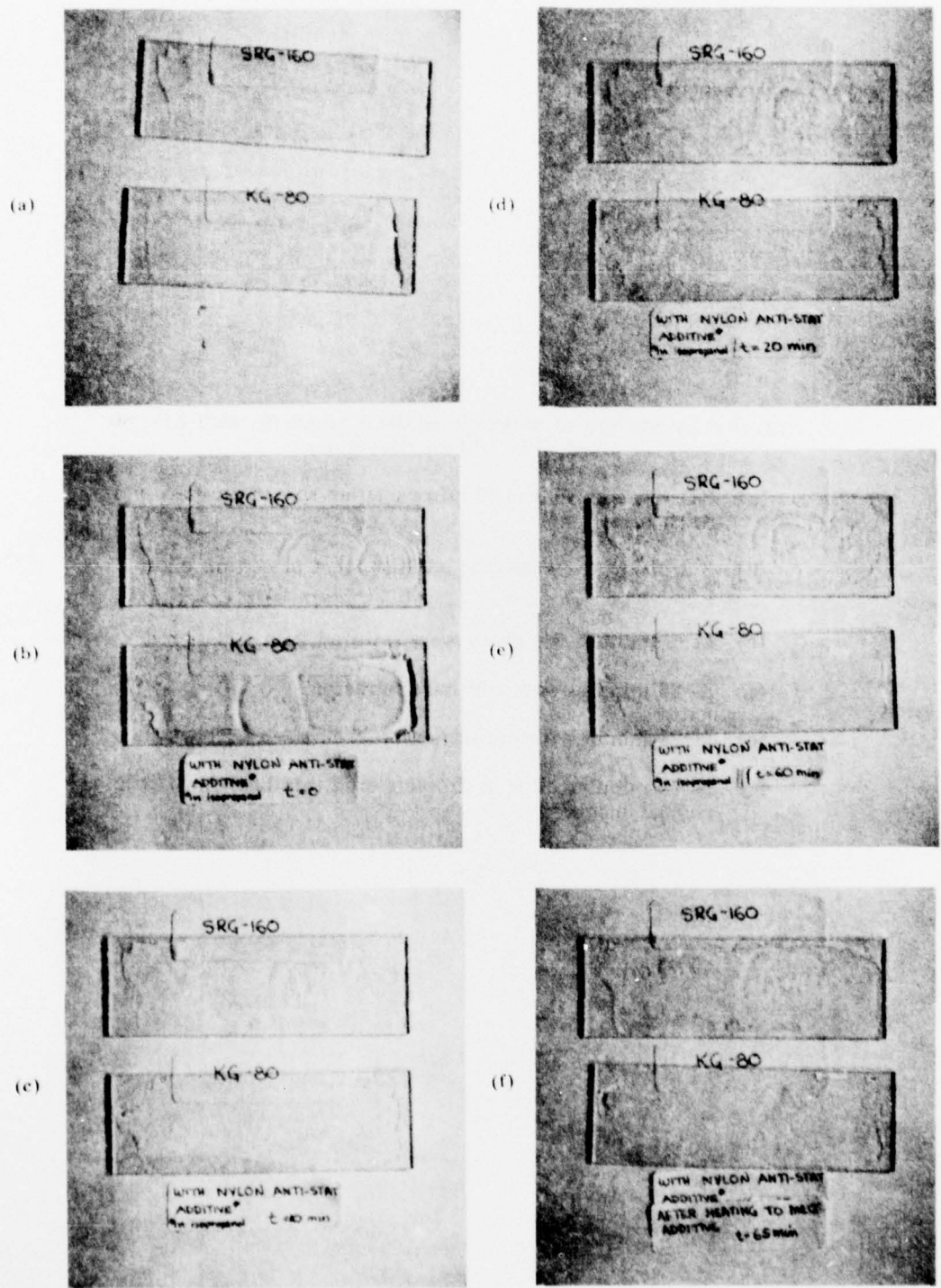


Fig. 4

Fig. 4 — Interaction of antistatic additive of AS NY with KG-80 and SRG-160 on glass slides

- (a) KG-80 and SRG-160 lubricants are spread on glass in a thick film
- (b) 3 drops of a solution of the antistatic additive in isopropanol acetone were placed on the oil surface at $t = 0$
- (c) at 10 minutes
- (d) at 20 minutes
- (e) at 60 minutes
- (f) Slides heated gently to melt the additive. Slight retraction is occurring; the oil and additive become grease-like.