

30p

FACILITY FORM 602

N65-35203

(ACCESSION NUMBER)

(THRU)

30

1

(PAGES)

(CODE)

TMX-54549

18

(NASA CR OR TMX OR AD NUMBER)

(CATEGORY)

DEGRADATION OF POLYMERIC COMPOSITIONS IN VACUUM TO  $10^{-9}$  mm Hg  
IN EVAPORATION AND SLIDING FRICTION EXPERIMENTS

By Donald H. Buckley and Robert L. Johnson 1964 30p w/o

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio

Presented at  
~~Prepared for the 20th Annual Technical Conference Society~~

of Plastics <sup>Engr.</sup> Engineers, January 28-31, 1964

Atlantic City, N. J.

Code 107-12

□ Conf

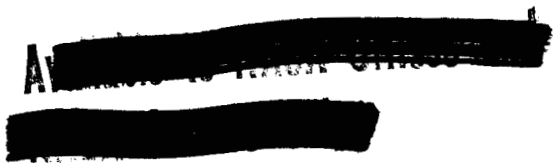
GPO PRICE \$ \_\_\_\_\_

CFSTI PRICE(S) \$ \_\_\_\_\_

Hard copy (HC) 2.00

Microfiche (MF) .50

ff 653 July 65



Unc

Prepared for the 20th Annual  
Technical Conference Society  
of Plastics Engineers  
January 28-31, 1964  
Atlantic City, N. J.  
Code 107-12

DEGRADATION OF POLYMERIC COMPOSITIONS IN VACUUM TO  $10^{-9}$  mm Hg

IN EVAPORATION AND SLIDING FRICTION EXPERIMENTS

By Donald H. Buckley and Robert L. Johnson

Lewis Research Center  
National Aeronautics and Space Administration  
Cleveland, Ohio

ABSTRACT

A 35203

Solid compositions studied included PTFE, PCFE, polyimides, both filled and unfilled, and fluorocarbon telomers. Evaporation experiments were conducted at ambient temperatures to  $1100^{\circ}$  F and pressures to  $10^{-8}$  mm Hg. Various molecular weights of different polymers were examined. Results indicate that evaporation rates in vacuum for polymers vary with molecular weight. Friction and wear experiments were conducted with a 3/16-inch-radius rider hemisphere (usually polymer) sliding on a flat disk (various materials) at speeds to 1480 feet per minute with a 1000-gram load on the rider specimen and at an ambient pressure of  $10^{-9}$  mm Hg. Fillers were found to influence markedly the wear of PTFE and PCFE in vacuum as a result of changes in heat dissipation properties, but they showed little or no influence on friction. Unfilled polyimide was found superior to unfilled PTFE and PCFE in friction, in wear, and in degradation characteristics. With the aid of a mass spectrometer, the decomposition mechanism for various PTFE compositions was observed during sliding.

AUTHOR

~~ALL INFORMATION CONTAINED HEREIN IS UNCLASSIFIED~~  
~~DATE 11-14-83 BY 1043/UC/STP~~

## INTRODUCTION

Polymer and polymer compositions have been found to be useful in a considerable number of lubrication applications. Liquid polymeric materials such as the polyalkylene glycols, the polyphenyl ethers, the silicones, and the fluorocarbon oils, are used as lubricants in areas where petroleum oils have limited usefulness. An example might be the high-temperature thermal stability of the polyphenyl ethers that makes them one of the more promising candidates as high-temperature liquid lubricants. Many of these materials, either alone in a waxlike (highly viscous) state or blended with fillers have been used as greases. Solid polymer compositions are also used in lubrication. Materials such as nylon, polytetrafluoroethylene, polychlorotrifluoroethylene, and phenolic resins have been used for some time in bearing, in seal, and in gear applications. Polymeric materials such as phenolic and epoxy compositions have been used and found to be very good binding agents for more conventional solid lubricants such as molybdenum disulfide.

Polymers have been studied both as lubricants and as self-lubricating components of lubrication systems, and they have been used under a wide variety of environments and conditions. The environments have included both low and high ambient temperatures ( $-423^{\circ}$  to  $+800^{\circ}$  F), cryogenic fluids such as liquid nitrogen, corrosive atmospheres, and radiation environments (refs. 1 to 4). The conditions investigated have included the effects of load, speed, orientation, and deformation (refs. 5 to 7).

Space vehicle and satellite systems have many operating components that require lubrication or self-lubricating components (ref. 8). Space presents a new environment for the application of polymers with lubricating capabilities. At the low ambient pressure of space ( $10^{-9}$  to  $10^{-13}$  mm Hg) and with

lack of oxygen, the lubrication of metals becomes extremely difficult. In the absence of oxygen and metal surface oxides to afford these surfaces protective films, high coefficients of friction, mass metal transfer, and complete welding of contacting metal surfaces can occur (refs. 9 and 10). The need for lubricants in the vacuum environment of space is even more critical than is found in conventional applications.

Polymers appear attractive for many space lubrication problems because of their high molecular weights (low volatility) and lubricating characteristics. Materials such as the polyphenyl ethers have very low vapor pressures in addition to having good thermal stability. Furthermore, solids such as polytetrafluoroethylene do not depend on the presence of adsorbates for their lubricating characteristics; that is, they are self-lubricating.

Although solid polymers have many characteristics that make them attractive as components for space lubrication systems, they also have limitations compared with metals. Most solid polymers suffer from poor mechanical characteristics, poor thermal conductivity, and temperature limitations. Mechanical properties, such as the compressive creep of materials like polytetrafluoroethylene, require special design considerations. With heat dissipation in vacuum being primarily by conduction and by radiation and with the extremely high local surface temperatures that can be generated as a result of frictional heating in lubrication systems, the poor thermal conductivity of polymers can create a real problem. This problem can become even more serious with the relatively low decomposition temperatures associated with most polymers (800° F or less).

In an effort to improve these properties of solid polymers, various fillers

have been added to polymers not only to improve mechanical and thermal properties but also to improve lubricating characteristics. These fillers have included metals, glasses, inorganic compounds, and solid lubricants.

The objectives of this investigation with polymers in a vacuum environment simulating space conditions were as follows: (1) to determine the volatility or evaporation rate of some solid polymer compositions at ambient temperatures to 1100° F, (2) to determine the friction characteristics and wear mechanism of various solid polymers, and (3) to determine, with the aid of a mass spectrometer, the mechanism of decomposition of these polymers in lubrication experiments.

## APPARATUS

### Evaporation Apparatus

The evaporation-rate apparatus used in this study is described in detail in reference 10 and is shown schematically in figure 1. The specimen, a 5/16-inch-diameter surface area, was suspended in a platinum pan from an electronic balance (sensitivity  $\pm 0.01$  mg) into the mouth of a wire-wound-tungsten cylindrical furnace. About 1.0 inch above the furnace was a copper condensing plate that was liquid nitrogen cooled. The liquid-nitrogen-cooled shield was provided to condense the evaporating species as completely as possible. The entire apparatus was enclosed in a glass bell jar system, and pressure within the system was measured with a nude hot-cathode ionization gage adjacent to the evaporating system.

### Friction and Wear Apparatus

The apparatus used in this investigation is described in detail in

reference 11 and is shown in figure 2. The basic elements of the apparatus were the specimens (a  $2\frac{1}{2}$ -in.-diam. flat disk and a  $3/16$ -in.-rad. rider) mounted in a vacuum chamber. The disk specimen was driven by a magnetic drive coupling. The coupling had two 20-pole magnets 0.150 inch apart with a 0.030-inch diaphragm between magnet faces. The driver magnet that was outside the vacuum chamber was coupled to a hydraulic motor. The second magnet was covered completely with a nickel-alloy housing (cutaway in fig. 2) and was mounted on one end of the shaft within the chamber. The end of the shaft that was opposite the magnet contained the disk specimen.

The rider specimen was supported in the specimen chamber by an arm that was mounted from a gimbal and sealed to the chamber with a bellows. A linkage at the end of the restraining arm, away from the rider specimen, was connected to strain-gage assembly that was used to measure frictional force. Load was applied through a dead-weight loading system.

Attached to the lower end of the specimen chamber was a 400-liter-per-second ionization pump and a mechanical forepump with liquid-nitrogen cold traps. The pressure in the chamber was measured adjacent to the specimen with a nude hot-cathode ionization gage. In the same plane as the specimens and ionization gage was a diatron-type mass spectrometer (not shown in fig. 1) for determination of gases present in the vacuum system. A 20-foot,  $3/16$ -inch-diameter stainless-steel coil was used for liquid-nitrogen and liquid-helium cryopumping of the vacuum system.

## EXPERIMENTAL PROCEDURE

### Evaporation Studies

Each sample was placed into the system, and the system was then evacuated.

When an ambient pressure of  $10^{-7}$  to  $10^{-8}$  mm Hg was reached, a 2-hour outgassing at  $200^{\circ}$  F of the specimen was initiated. The purpose of the outgassing was to remove entrained gases and water from the specimens. In some instances (e.g., PTFE) outgassing periods to 24 hours were used, and the results were compared to those obtained for the shorter time period. After completion of the outgassing phase, the sample was cooled to room temperature, and the evaporation experiments were started. At the lower ambient temperatures the specimen was held at specific temperatures for as long as 8 hours, while at higher temperatures meaningful data could be obtained in much shorter periods of time.

#### Friction and Wear Experiments

The polymer compositions used in the friction and wear experiments were machined to size. The metal disks were circumferentially finish ground to a surface roughness of 4 to 8 microinches. Before each experiment the metal disks were given the same preparatory treatment: (1) a thorough rinsing with acetone to remove oil and grease, (2) a polishing with moist levigated alumina on a soft polishing cloth, and (3) a thorough rinsing with tap water followed by distilled water. The polymer specimens were rinsed with acetone followed by ethyl alcohol. For each experiment a new set of specimens was used.

### RESULTS AND DISCUSSION

#### Evaporation Studies

One of the most important properties of a material that must be considered in the selection of lubricants for space applications is evaporation tendencies. Evaporation-rate experiments were therefore conducted with various solid polymer compositions in vacuum. These included fluorocarbon telomers, polytetrafluoroethylene, and polychlorotrifluoroethylene (ref. 12).

### Telomers

The telomers are fluorocarbon polymers that have varied end groups introduced to function in part as chain stoppers. These materials are solids and have relatively low molecular weights compared with PTFE. The evaporation rates for two of these compositions were determined in vacuum, and the results obtained are presented in figure 3. At ambient temperatures of 55° to 450° F the evaporation rate of two molecular weight telomers (2000 and 3700) could be represented by a single curve. Above 450° F the evaporation rate of the lower molecular (2000) material increased more rapidly than the higher molecular weight material.

### PTFE

The evaporation rates for three PTFE compositions were determined next in vacuum. The materials examined were an extruded PTFE composition and two molded materials, PTFE No. 1 and PTFE No. 6. The results obtained in the evaporation experiments are presented in figure 4 with the results obtained from reference 10 for an early PTFE commercial product. The results of figure 4 indicate that all three compositions examined exhibited lower evaporation rates at the higher temperatures than the early commercial PTFE composition from reference 10. This may have resulted from such factors as improved purity (lower number of contaminants to initiate decomposition mechanisms), more efficient polymerization, and/or a narrower molecular weight distribution range (higher molecular weight distribution). The extruded composition exhibited a higher evaporation rate than the two molded compositions. With both molded compositions a relatively low rate of evaporation was measured at



ambient temperatures to 800° F. Above 800° F, as indicated from the data, decomposition began to influence markedly the measured evaporation rates. The results indicate that, if a low evaporation rate is needed, the molded compositions may be more desirable.

#### PCFE

The evaporation rates for four different molecular weights of PCFE were determined in vacuum. The molecular weights examined were 76,000, 157,000, 185,000, and 196,000. In addition, a copolymer of PCFE and vinylidene fluoride was also examined. The results obtained are presented in figure 5. The evaporation rate for the 76,000 molecular weight PCFE was higher than that for the other three compositions. Very little difference in the evaporation rate could be detected for the other compositions. This may have resulted from the relatively small differences in molecular weight (157,000, 185,000, and 196,000). At 550° F the weight loss for the 76,000 molecular weight material was so rapid that significant data could not be obtained. This was observed at 600° F for the higher molecular weight species. It is at these temperatures that decomposition of the material occurs.

#### Polyimide

A relatively new development in the solid polymers is the polyimides (ref. 13). In order to determine the stability of this material in vacuum some evaporation-rate experiments were conducted with the material. The results obtained in these experiments are presented in figure 6. At ambient temperatures to 500° F the evaporation rate was less than  $10^{-10}$  grams per square centimeter per second. The weight loss was so low that no weight

change in the sample could be detected. Above 500° F the weight loss was quite high since the material begins to decompose in this region. At temperatures to 500° F, however, the polyimide material appears very attractive.

#### Epoxy Compositions

The evaporation rates for two epoxy compositions were also determined in vacuum. The material compositions were epoxy-MoS<sub>2</sub> and epoxy-PTFE. The results obtained are presented in figure 7. The evaporation rate for the epoxy-PTFE composition was similar to what was observed with the PTFE at temperatures below 600° F. Above 600° F the evaporation rate began to increase markedly. The epoxy-MoS<sub>2</sub> composition exhibited no measurable evaporation rate at ambient temperatures to 600° F. Above 600° F, however, the weight loss was very rapid.

#### Friction and Wear Studies

In the sliding process associated with lubrication systems, extremely high surface temperatures can be generated. In a vacuum environment where heat dissipation is primarily by conduction and radiation, temperatures experienced may be much greater than encountered in gaseous environments. Since polymers are sensitive to temperature, a knowledge of temperatures generated in the sliding process in vacuum would be helpful in understanding the behavior of polymers in lubrication experiments. Experiments were therefore conducted in vacuum with thermocouples installed in rider specimens in an attempt to measure the bulk temperatures of these specimens. In friction and wear studies with polymers, the polymer was generally the rider specimen and the disk was

generally a metal. It would be extremely difficult to gain an insight into specimen bulk temperatures near the sliding interface with polymers because of their poor heat-conduction characteristics. The specimen should be a good thermal conductor in order that with the specimen thermocouple displaced from the interface where the temperatures are being generated, representative temperature values of this region can be obtained. Electrolytic silver rider specimens were therefore selected, and thermocouples were installed on the axis of the specimen 1/8 inch from the initial point of rider disk contact. The bulk specimen temperatures measured as a function of sliding velocity with electrolytic silver sliding on 440-C stainless steel are presented in figure 8. At the sliding velocity used in friction and wear experiments with polymers in this investigation (390 ft/min), a temperature of approximately 1100° F is indicated by the curve. At 550 feet per minute a temperature of 1515° F was measured. The frictional heat generated in vacuum at 550 feet per minute is then sufficient to raise the temperature of the rider to near its melting point (1724° F). It is quite probable that a further increase in sliding velocity would have resulted in a melting of the silver rider. These results indicate the magnitude of temperature that might be expected in sliding friction experiments with plastics in these studies.

#### PTFE and PCFE

Friction and wear experiments were conducted in vacuum with PTFE and PCFE compositions sliding on 440-C stainless steel (ref. 11). The results obtained are presented in figure 8. These results were obtained at a sliding velocity of 390 feet per minute with a load of 1000 grams on the polymer rider specimen,

or environmental pressure of  $10^{-9}$  mm Hg and no external specimen heating. The friction coefficient obtained for all PTFE and PCFE compositions of figure 8 were between 0.25 and 0.30, which indicated that the presence of fillers in PTFE and PCFE had very little effect on friction coefficient.

The wear of the unfilled PTFE and PCFE were high and about the same value, which indicated that the presence of chlorine in the PCFE molecule exerted no influence on either friction or wear of PCFE. The wear of both PTFE and PCFE was one of an abrasion process. Large particles of the polymer abraded from the rider specimen surface struck and adhered to the disk specimen surface. These wear particles can be seen in figure 9(b). Examination of the wear particles after the experiments revealed that the particles were sticky to the touch and that they had actually adhered to the disk specimen surface. Although the disk specimen was rotating in a horizontal position with the interface surface down (see fig. 2), the major portion of the wear particles adhered to it, particularly in the region adjacent to the inner and outer edge of the wear track. The sticky nature of the particles indicated that heat generated at the sliding interface was transferred to the polymer particles, increasing the surface temperature of the particles, and was causing localized or surface degradation. The polymer particles are thus absorbing some of the frictional heat generated in the sliding process.

In order to determine the influence of fillers on the friction and wear characteristics of PTFE and PCFE, experiments were conducted with 25-percent glass-fiber-filled PTFE and PCFE. The results are presented in figure 9. The addition of 25-percent glass fiber to PTFE did not influence markedly the friction obtained with PTFE; it did, however, decrease the rider wear by

a factor of 100. This represents an appreciable reduction in wear. With the 25-percent glass-fiber-filled PCFE the specimen swelled and completely lost its shape (photo insert of fig. 9) so that rider wear could not be measured. The softening point for this material is about 400° F (ref. 14). The swelling and presence of gas pockets that could be seen within the specimen indicated that the specimen must have reached 400° F. This result was not obtained with glass-fiber-filled PTFE, which has a softening point of about 550° F. Examination of the wear area of the glass-filled PTFE composition revealed the presence of very fine black "soot-like" particles about the worn area (fig. 9(b)). Chemical tests indicated that the black debris contained iron, but about 70 percent of the film was carbon. This film will be discussed in greater detail with reference to figure 10.

With the CTFE compositions, the fact that the unfilled composition did not swell and lose its shape and the glass filled composition did may have resulted from the ability of the unfilled CTFE to dissipate a large quantity of heat in the wear particles. The glass-filled composition exhibits much less wear, and the heat generated at the sliding interface is therefore absorbed to a large extent by the polymer. The presence of glass fibers in the polymer may increase the ability of the heat generated to be carried within the polymer body. Some of the heat generated and carried within the polymer body is transferred to the rider specimen holder. Thus, the polymer temperature is not as high as it might be without the dissipation of some heat to the metal specimen holder.

The friction and wear of a 25-percent copper-powder-filled PTFE composition was also determined in vacuum. The results obtained are presented in

figure 9(a). The friction coefficient obtained was nearly the same as obtained with the unfilled PTFE. The wear, however, was 100 times less and was comparable to that obtained with glass-filled PTFE. The rider specimen wear surface is shown in figure 9(b).

Changing sliding velocity will change surface temperatures. In an attempt to gain some understanding of the polymer decomposition mechanism at the sliding interface, some friction experiments were conducted at various sliding velocities using the mass spectrometer to identify the decomposition products formed. A plot of the decomposition products formed (ion concentration) as a function of sliding velocity for various PTFE compositions is presented in figure 10.

In figure 10(a), with unfilled PTFE sliding on 440-C stainless steel, the principal decomposition products observed were  $M/e\ 31(CF^+)$  and  $M/e\ 50(CF_2^+)$ . It is interesting to note that as the sliding velocity increases a decrease in ion concentration for both  $M/e$  ratios occurs. Since increasing sliding velocity increases surface temperatures (fig. 8), the reverse trend would normally be expected. Increasing sliding velocity, however, results in an increase in the amount of wear particle generated. In reference to figure 9 a mention was made of heat being carried from the interface by PTFE wear particles (heat dissipation in the localized surface decomposition of polymer particles). With an increase in the mass of wear particles formed, as evidenced by the two wear values presented on the curve of figure 10(a), a great quantity of heat is dissipated by wear particles and a decrease in concentration of decomposition products is observed.

The decomposition products formed with 25-percent glass-filled PTFE sliding

on 440-C is shown in figure 10(b). With the 25-percent glass-filled PTFE composition, the ion concentration for M/e 19 ( $F^+$ ), M/e 31 ( $CF^+$ ), and M/e 50 ( $CF_2^+$ ) were found to increase with increasing sliding velocity. This result is not like that obtained with unfilled PTFE where very little M/e 19 ( $F^+$ ) was observed. With the 25-percent glass-filled PTFE, M/e 19 ( $F^+$ ) is the principal decomposition product detected by the mass spectrometer. This result indicates that carbon to fluorine bond cleavage must be occurring at the sliding interface. The wear with glass-filled PTFE is 100 times less than that obtained with unfilled PTFE, and the heat, therefore, is not carried away as readily from the interface. A thin film of PTFE may be interposed between the metal surface and the glass fibers. As a result of the thin film present, it is hypothesized that localized decomposition of the PTFE film occurs and increases with an increase in sliding velocity or surface temperature. The presence of carbon on the glass-filled PTFE rider surface was noted in figure 10(b), and only a small concentration of carbon was observed in the mass spectrometer tracing (not shown in fig. 10(b)). This indicated that the decomposition occurring was taking place at the sliding interface and not in the mass spectrometer. If decomposition were occurring in the mass spectrometer by carbon to fluorine bond cleavage, an increase in fluoride ion concentration would be accompanied by the increase in carbon ion concentration. This was not observed. It must be pointed out, however, that some cleavage of the carbon to fluorine bond must be expected to occur in the mass spectrometer.

In the sliding velocity experiments conducted with copper-filled PTFE, very small concentrations were observed for the two M/e ratios 19 and 31 over the range of sliding velocities investigated (fig. 10(c)). These results

indicate that decomposition of the PTFE was slight. This effect cannot be related to wear because the wear was the same for both glass- and copper-filled compositions (fig. 9(a)). With copper it is believed a similar mechanism prevails as was observed with glass; that is, a thin film of PTFE is interposed between the copper particles and the metal disk surface functioning as a lubricant. Unlike the glass fillers, however, copper is a good heat conductor. It serves to conduct heat away from the interface. The copper dissipates the heat generated and reduces the decomposition of PTFE. Although there is only 25-percent copper nominally present in the PTFE composition at the sliding interface, the discrete particles flow out, and the exposed area of interface copper increases (see bright areas of photo insert of fig. 10(c)).

Since heat rejection appears to play an important role in the decomposition of PTFE, some experiments were conducted with a 15-percent glass composition sliding on (1) aluminum oxide (a poor heat conductor), (2) silver, and (3) copper (both good conductors) to determine the influence of thermal conductivity of the disk material on the wear characteristics observed for the glass-filled PTFE compositions. The friction and wear results obtained are presented in figure 9(a). The mass spectrometer data obtained in the experiments is presented in figure 11(a). The friction and wear results are comparable for aluminum oxide and copper disk materials. A comparison of the mass spectrometer data of figure 11(b) indicates that, as might be anticipated, the concentration of the polymer degradation products is much less with the copper disk than with aluminum oxide.

The silver disk specimen has the highest thermal conductivity, but the results are not in agreement with those obtained for the other two disk



materials. It might be expected that the least amount of PTFE decomposition would occur with silver. The friction and wear were higher, and a higher concentration of decomposition products was observed with the silver disk than was obtained with copper. The friction coefficient appeared to more closely approach the friction for metal-metal combinations. Examination of the rider surface after the experiment indicated transfer of silver to the rider surface (see photo insert of fig. 11(a)). These results indicate that silver was sliding on silver rather than PTFE on silver. With metal on metal greater frictional heating occurs (with a thin silver film on the PTFE rider), and a higher concentration of PTFE decomposition products are obtained than with PTFE sliding on metal. Silver, at the ambient pressures of this investigation, does not have an oxide present on its surface. This would suggest a physical transfer of silver to the PTFE surface. Reaction of the disk surfaces with H<sub>2</sub>S gas did not reveal the presence of PTFE film on the disk surface.

#### Polyimides

Because of the encouraging results obtained in evaporation studies with the polyimide composition (fig. 6), some friction and wear studies were conducted in vacuum ( $10^{-9}$  mm Hg) with polyimides sliding on both metals and on itself. The results obtained in these experiments are presented in figure 12. In figure 12(a) the friction and wear for polyimide riders on 440-C stainless-steel disks, with the polyimide cut in two directions from the parent material block, are compared. Although the friction for polyimides on 440-C stainless steel was relatively low, the wear to the polyimide rider was

somewhat high. If these results are compared to those obtained in figure 8, with unfilled PTFE, however, the wear for the polyimide is only 1/15 that obtained with PTFE, and the friction is also lower.

Friction and wear experiments were next conducted with the polyimide sliding on polyimide. The results obtained are presented in figure 12(a). The friction coefficient obtained with polyimide sliding on polyimide was much higher than that obtained with polyimide sliding on 440-C stainless steel. The wear with polyimide on polyimide, however, was 1/500 that obtained with polyimide on metal. Surface profile traces and photomicrographs are presented in figure 12(b) for the wear area to the metal and polyimide disk specimens. With the metal disk, a thin nearly transparent polyimide film was transferred to the disk surface. The film was not thick enough to be detected in the surface profile traces (magnification X1000 in the vertical direction). The polyimide disk specimen did, however, show evidence of wear.

A 15-percent graphite-filled polyimide composition was also examined in friction and wear studies. The results obtained are presented in figure 9. The presence of graphite in the polyimide did not improve its lubricating characteristics; relatively high friction and wear were obtained. Friction and wear experiments were conducted with a 20-percent copper-fiber-filled polyimide composition. In the experiments the filled polyimide slid on 440-C stainless steel. The friction and wear results obtained are presented in figure 10. Both friction and wear for the copper-fiber-filled composition were lower than that obtained with the unfilled composition. The friction and wear obtained with 20-percent copper-fiber-filled polyimide were lower than that obtained with 25-percent copper-powder-filled PTFE (fig. 9).

The results obtained in this investigation with polyimides and PTFE in vacuum indicate the polyimide to be a superior material to PTFE in friction, wear, and outgassing characteristics at ambient temperatures.

The friction and wear characteristics were determined for the two epoxy compositions examined in the evaporation studies of figure 7. The results obtained in these studies are presented in figure 13. The epoxy-MoS<sub>2</sub> composition sliding on 440-C stainless steel produced a friction coefficient of 0.015. The wear to the rider specimen was also very low. The epoxy-PTFE composition exhibited very low friction. The specimen, however, swelled, changed color, and developed surface cracks, which indicated that thermal decomposition had occurred.

#### SUMMARY OF RESULTS

From the data obtained in this investigation with various polymer compositions in vacuum evaporation, friction, and wear studies, the following summary remarks can be made:

1. The quantity and nature of decomposition products formed in the process of polymers sliding on various substrates is determined both by the substrate material and the polymer. Variation of the filler material in polymers can result in differences in the decomposition mechanism of the polymer. With glass-filled PTFE compositions, sufficient heat is generated at the sliding interface to result in carbon to fluorine bond scission. The presence of copper as a filler in PTFE (good heat conductor) reduces the decomposition observed.
2. The evaporation, friction, and wear for unfilled polyimide indicate it to be a superior slider material to unfilled PTFE in vacuum at ambient temperatures.

3. The presence of fillers (glass and metal) in polymers, such as PTFE and PCFE, did not improve the friction characteristics of the basic polymer in vacuum. They did, however, in some instances reduce wear by as much as a factor of 100.

4. Evaporation rates for various fluorocarbon compositions in vacuum were found to be related to the molecular weight of the material. Where different samples of the same material were examined, differences in weight loss were noted (PTFE). This was believed to have resulted from a difference in distribution of molecular weights within the material.

#### REFERENCES

1. Wisander, D. W., and Johnson, R. L., "A Solid Film Lubricant Composition for Use at High Sliding Velocities in Liquid Nitrogen," Trans. ASLE, vol. 3, no. 2, 1960, pp. 225-231.
2. Wisander, D. W., Maley, C. E., and Johnson, R. L., "Wear and Friction of Filled Polytetrafluoroethylene Compositions in Liquid Nitrogen," Trans. ASLE, vol. 2, no. 1, April 1959, pp. 58-66.
3. Blainey, A., "Fluon-Impregnated Self-Lubricating Bearing Materials," Atomic Energy Research Establishment; A.E.R.E. M/R 1519, Harwell, Berks, 1955.
4. Twiss, S. B., Willson, P. J., and Sydor, E. J., "Friction of Polytetrafluoroethylene Dry Bearings," Lubrication Engineering, vol. 14, no. 6, June 1958, p. 251.
5. Allan, A. J. G., "Plastics as Solid Lubricants and Bearings," Lubrication Engineering, vol. 14, no. 5, May 1958, pp. 211-215.

6. Tabor, D., and Williams, W. D. E., "The Effect of Orientation on the Friction of Polytetrafluoroethylene," *Wear*, vol. 4, 1961, pp. 391-400.
7. Passor, M. W., and Tabor, D., "The Friction and Deformation of Polymers," *Proceedings of the Royal Society, ser. A*, vol. 235, 1956, pp. 210-224.
8. Johnson, R. L., and Anderson, W. J., "Summary of Lubrication Problems in Vacuum Environment," Paper presented at 1963 USAF Aerospace Fluids and Lubricants Conference, San Antonio, Texas, April 16-19, 1963.
9. Bowden, F. P., and Tabor, D., "Friction and Lubrication of Solids," Oxford University Press, 1954.
10. Buckley, D. H., Swikert, M., and Johnson, R. L., "Friction, Wear and Evaporation Rates of Various Materials in Vacuum to  $10^{-7}$  mm Hg," *Trans. ASLE*, vol. 5, no. 1, April 1962.
11. Buckley, D. H., and Johnson, R. L., "Friction, Wear, and Decomposition Mechanisms for Various Polymer Compositions in Vacuum to  $10^{-9}$  Millimeter of Mercury," NASA TN D-2073, 1963.
12. Buckley, D. H., and Johnson, R. L., "Evaporation Rates for Various Organic Liquid and Solid Lubricants in Vacuum to  $10^{-8}$  Millimeter of Mercury at Temperatures from  $55^{\circ}$  to  $1100^{\circ}$  F," NASA TN D-2081, 1963.
13. Belgium Patent No. 582,597; Belgium Patent No. 589,179; French Patent No. 1,239,491; French Patent No. 1,256,203.
14. Rudner, M. A., "Fluorocarbons," Reinhold Publishing Corporation, New York (1958).

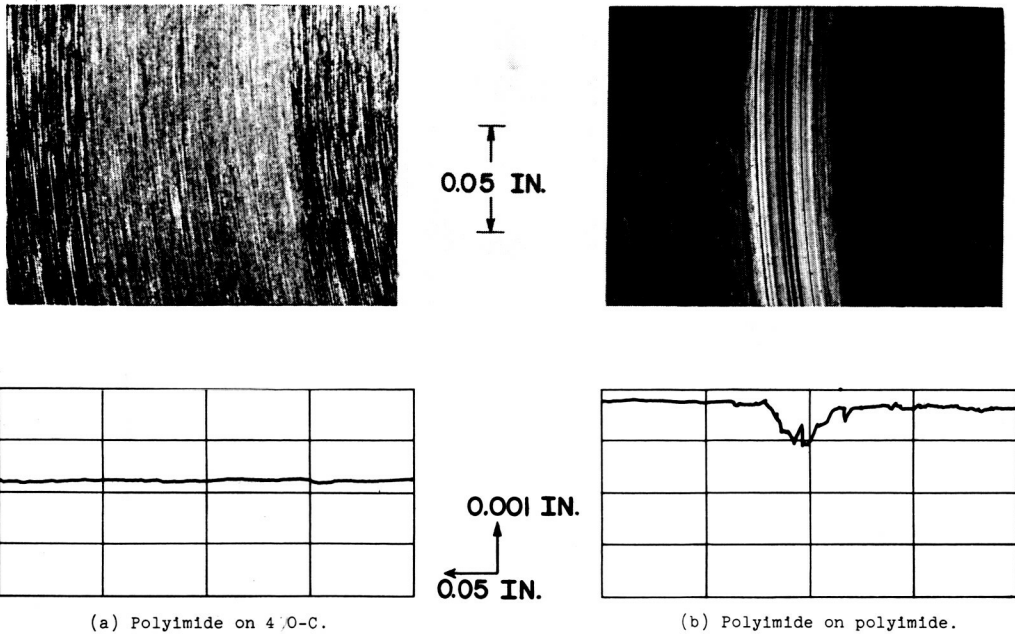


Fig. 12(b) Photomicrographs and surface profile traces for polyimide sliding on 440-C S.S. and polyimide in vacuum. Sliding velocity, 390 ft/min; load, 1000 G; ambient pressure,  $10^{-9}$  mm Hg; no external heating; duration, 1 hr.

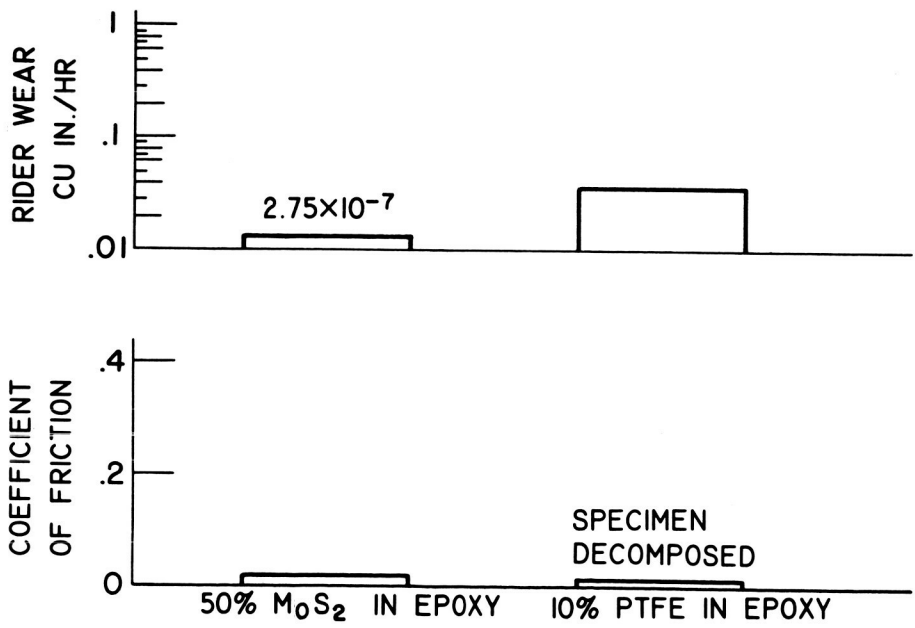


Fig. 13. - Friction and wear of two epoxy compositions in vacuum. Disk, 440-C S.S.; sliding velocity, 390 ft/min; load, 1000 G; ambient pressure,  $10^{-9}$  mm Hg; duration, 1 hr; no external specimen heating.

E-2242

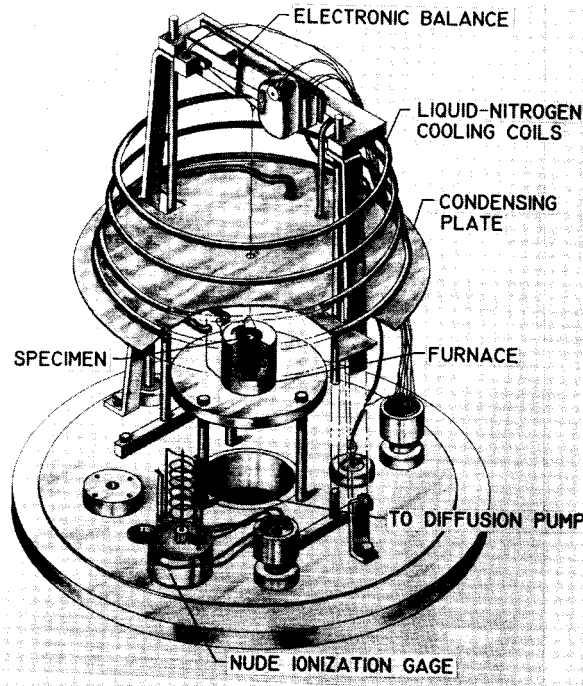


Fig. 1. - Vacuum evaporation apparatus.

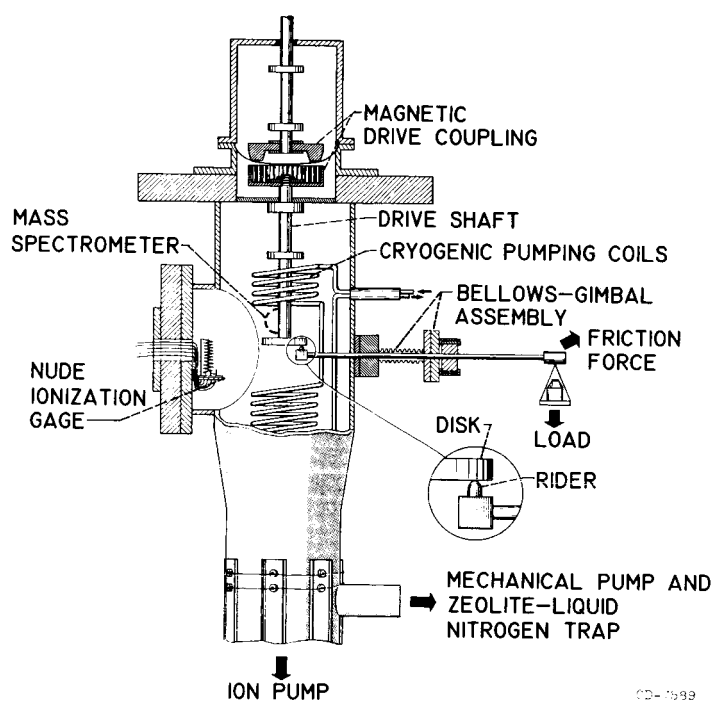


Fig. 2. - High-vacuum friction and wear apparatus.

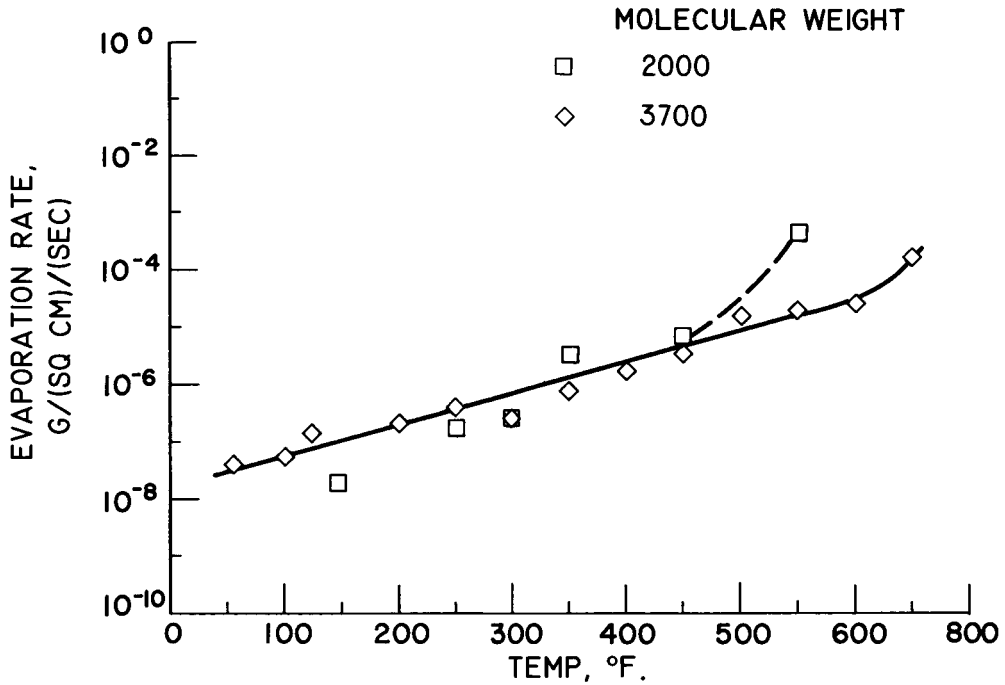


Fig. 3. - Evaporation rates for two telomer compositions in vacuum. Ambient pressure, 10<sup>-7</sup> mm Hg.

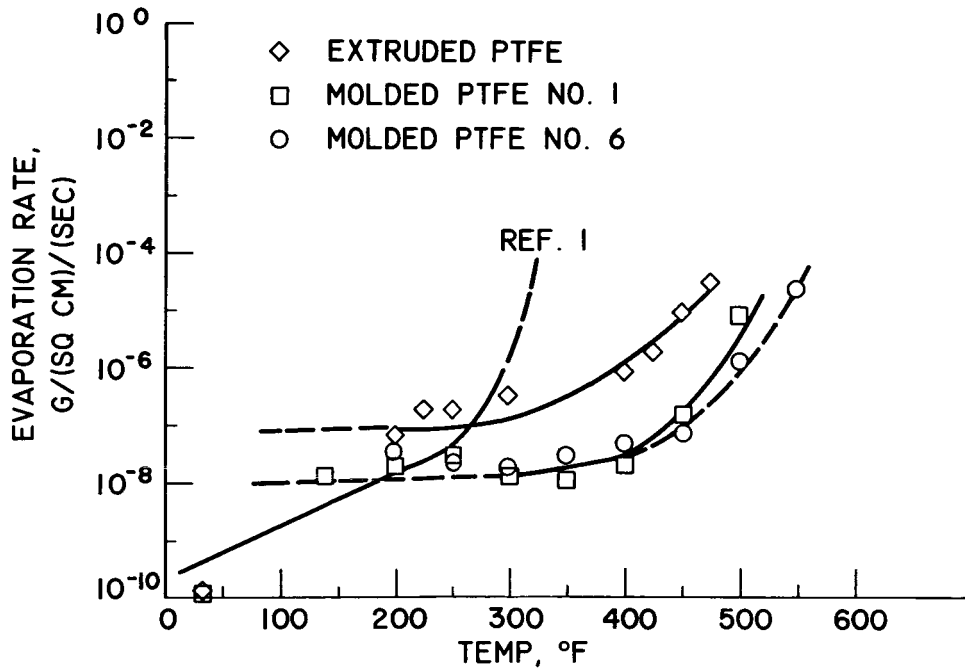


Fig. 4. - Evaporation rate for various teflon compositions in vacuum. Ambient pressures, 10<sup>-7</sup> to 10<sup>-8</sup> mm Hg.



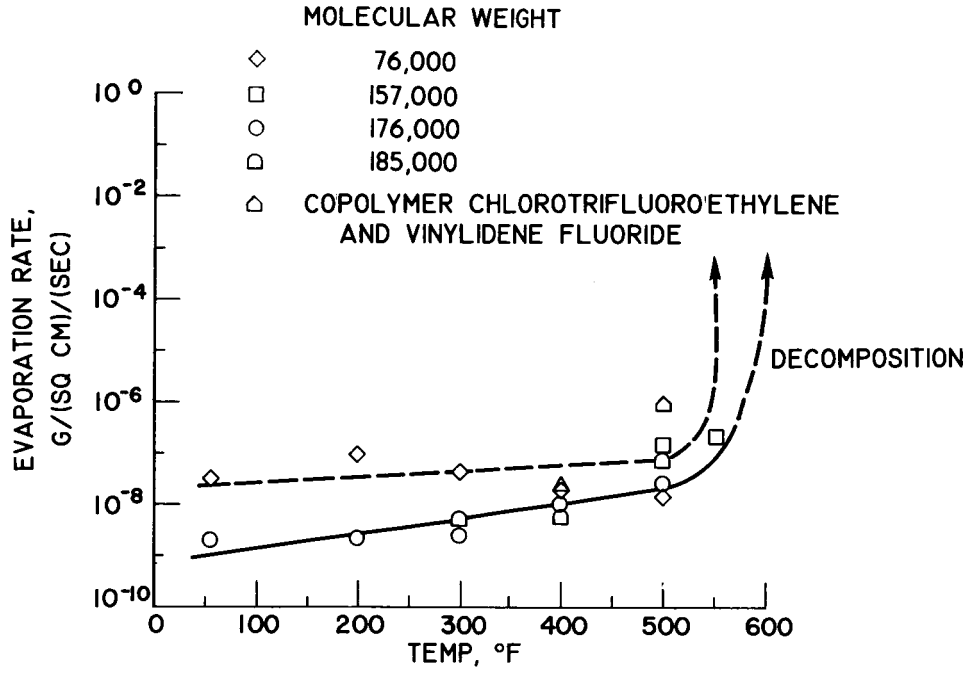


Fig. 5. - Evaporation rate of PCPE for various molecular weights in vacuum.

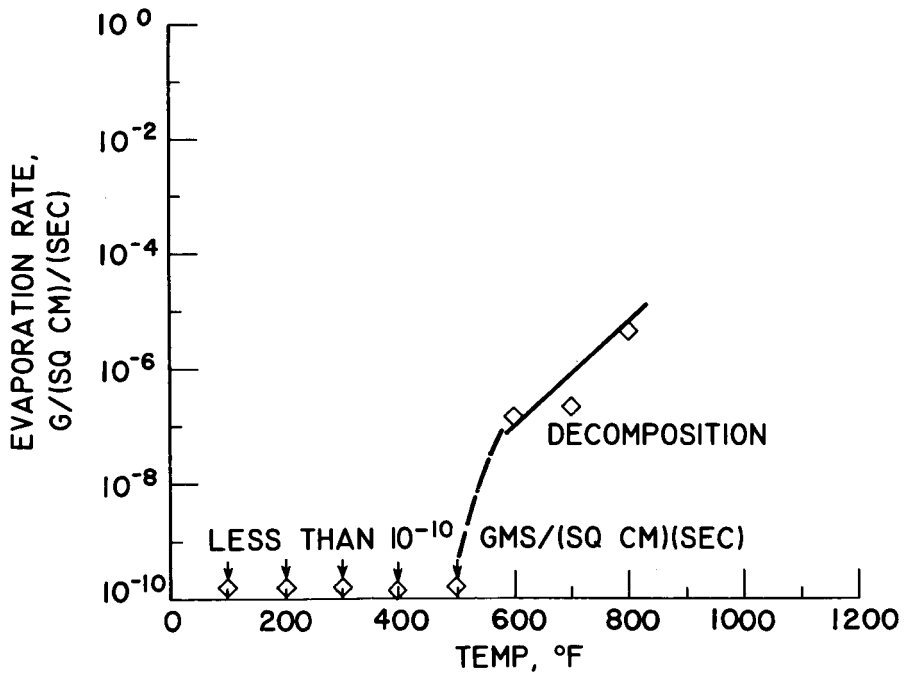


Fig. 6. - Evaporation rate of polyimide in vacuum. Ambient pressures,  $10^{-7}$  to  $10^{-8}$  mm Hg.

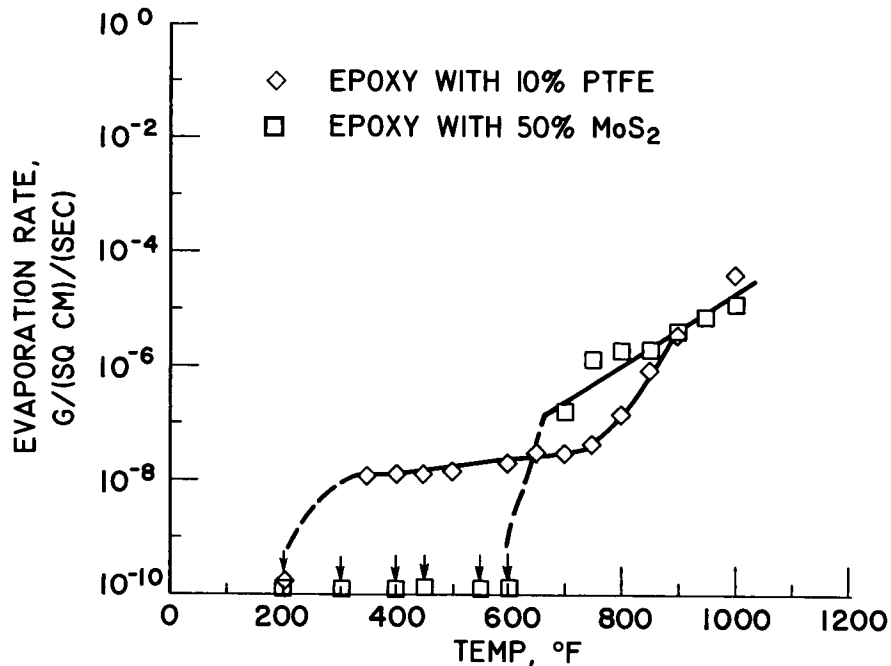


Fig. 7. - Evaporation rates for two epoxy compositions in vacuum. Ambient pressure, 10<sup>-7</sup> mm Hg.

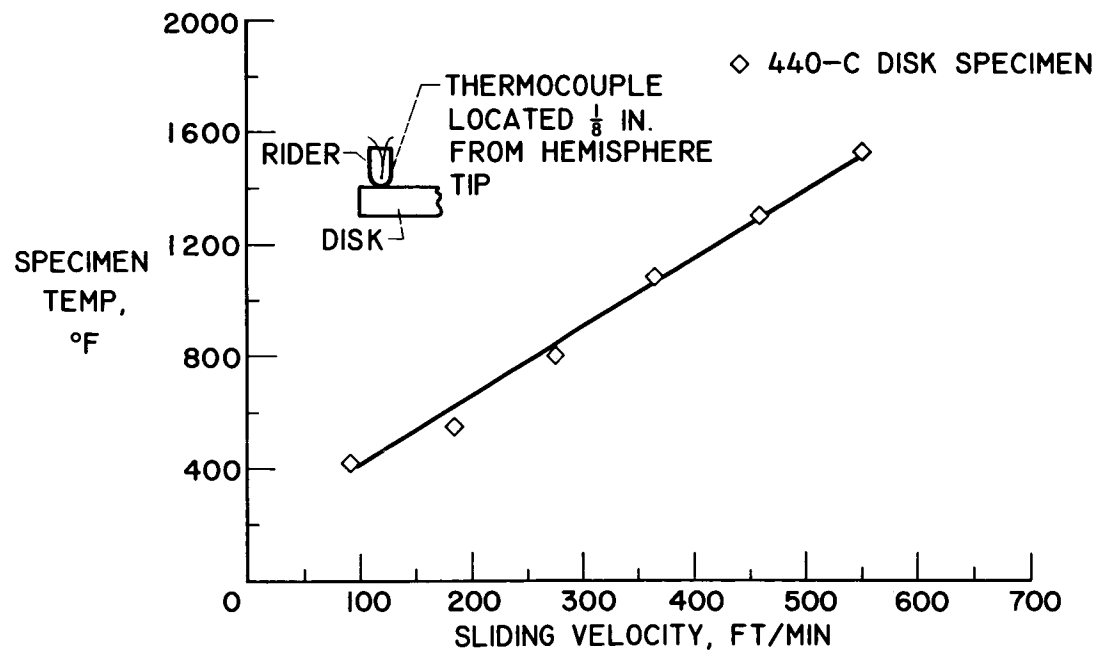


Fig. 8. - Electrolytic silver rider specimen temperatures generated in vacuum. Load, 1000 G; ambient pressure, 10<sup>-9</sup> mm Hg; no external heating of specimen.

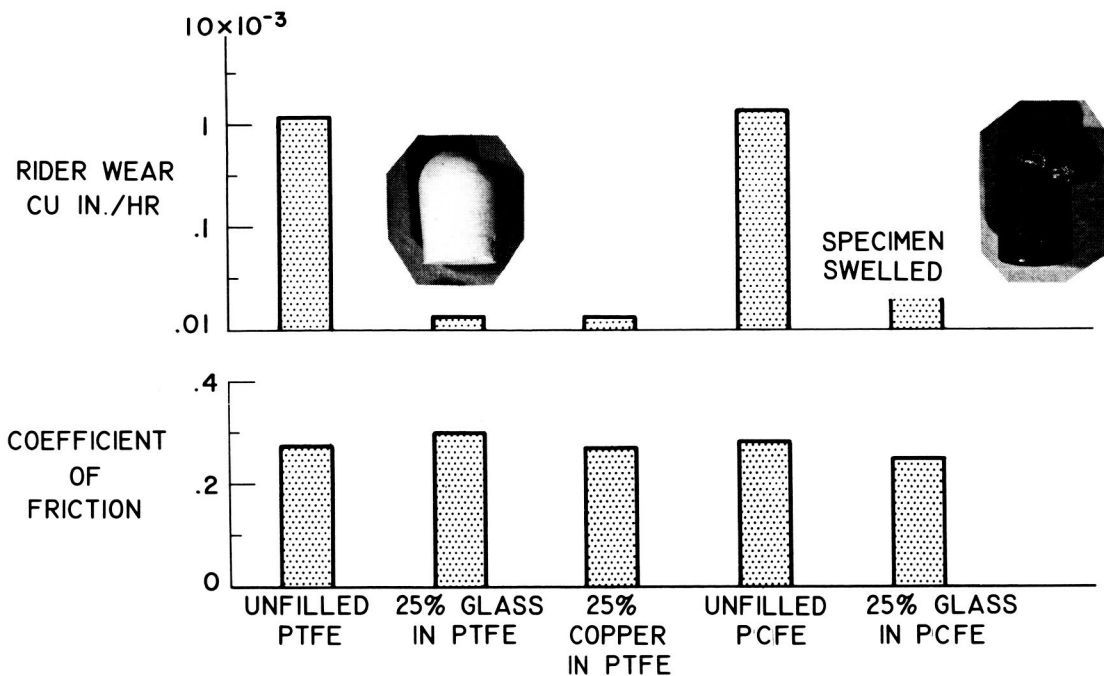
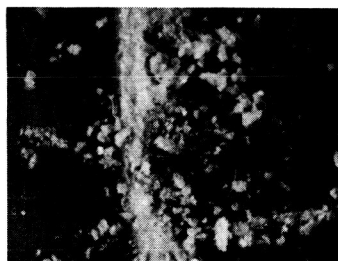


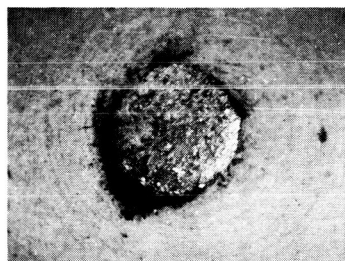
Fig. 9(a). - Friction and wear of PTFE and PCFE in vacuum. Sliding velocity, 390 ft/min; load, 1000 G; ambient pressure,  $10^{-9}$  mm Hg; no external specimen heating.



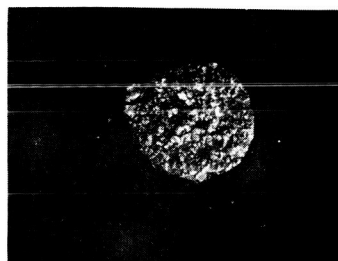
1. PTFE WEAR PARTICLES



2. PCFE WEAR PARTICLES



3. 25% GLASS-FILLED PTFE RIDER WEAR



4. 25% COPPER-FILLED PTFE RIDER WEAR

Fig. 9(b). - Wear and wear particles of PTFE and PCFE compositions obtained in vacuum friction and wear experiments. Magnification (20X).

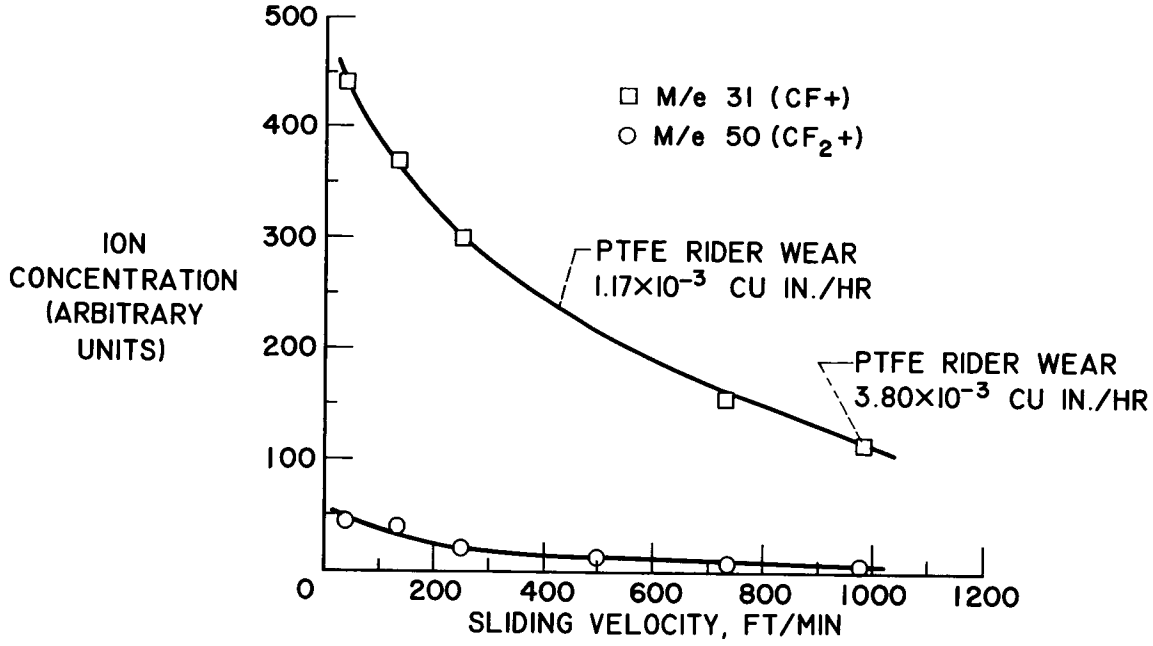


Fig. 10(a) Decomposition products of unfilled PTFE obtained in vacuum friction studies. Disk, 440-C.S.S; 1000 G; mm Hg; no external specimen heating.

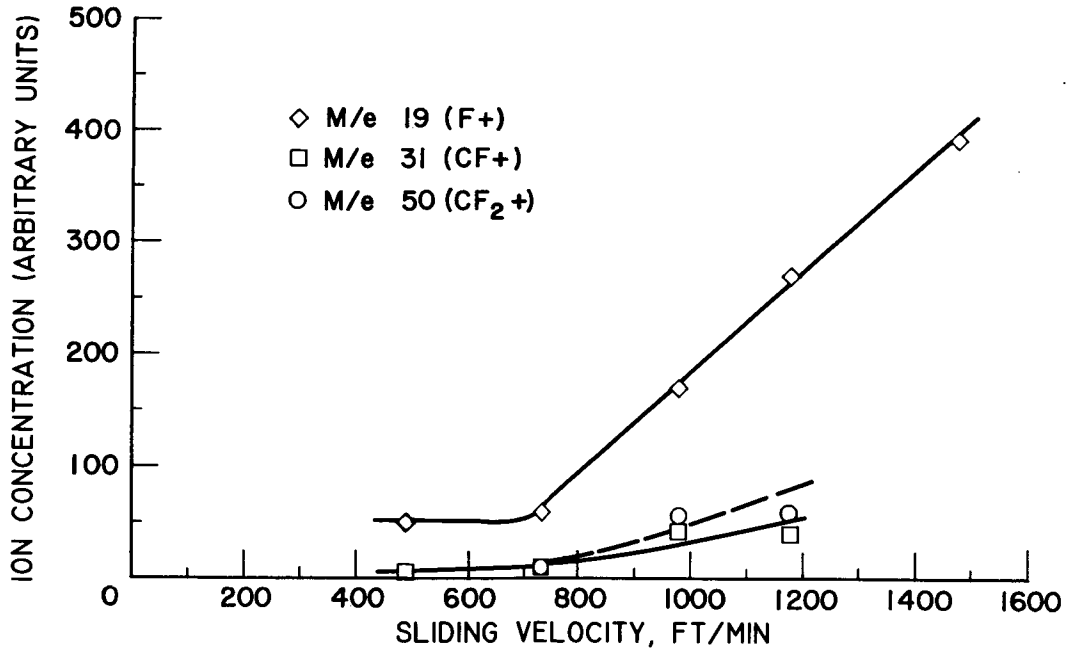


Fig. 10(b). - Decomposition products of 25% glass-filled PTFE obtained in vacuum friction studies. Disk, 440-C S.S.; 1000 G; 10<sup>-9</sup> mm Hg; no external specimen heating.

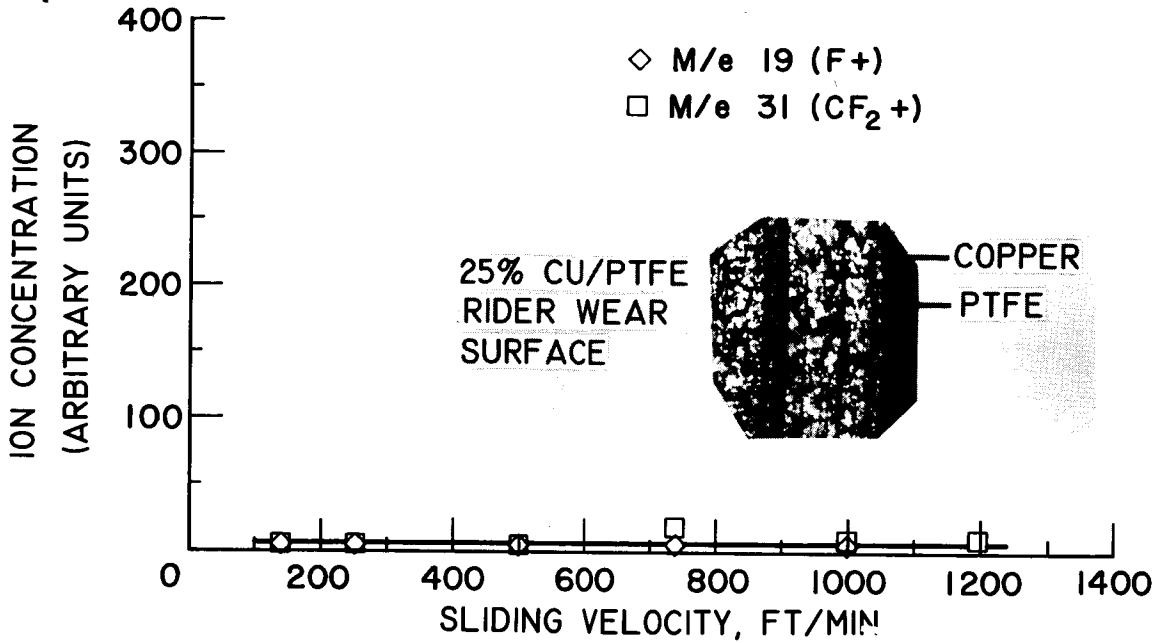


Fig. 10(c). - Decomposition products of 25% copper-filled PTFE obtained in vacuum friction studies. Disk, 440-C S.S.; 10<sup>-9</sup> mm Hg; no external specimen heating.

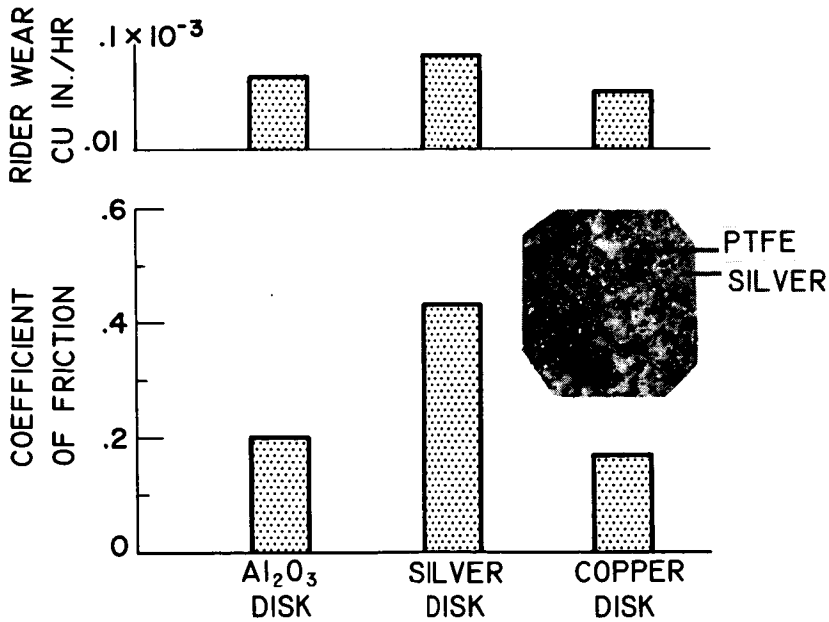


Fig. 11(a). - Friction and wear of 15-percent glass-filled PTFE in vacuum. Sliding velocity, 390 ft/min; load, 1000 G; ambient pressure, 10<sup>-9</sup> mm Hg; no external specimen heating.

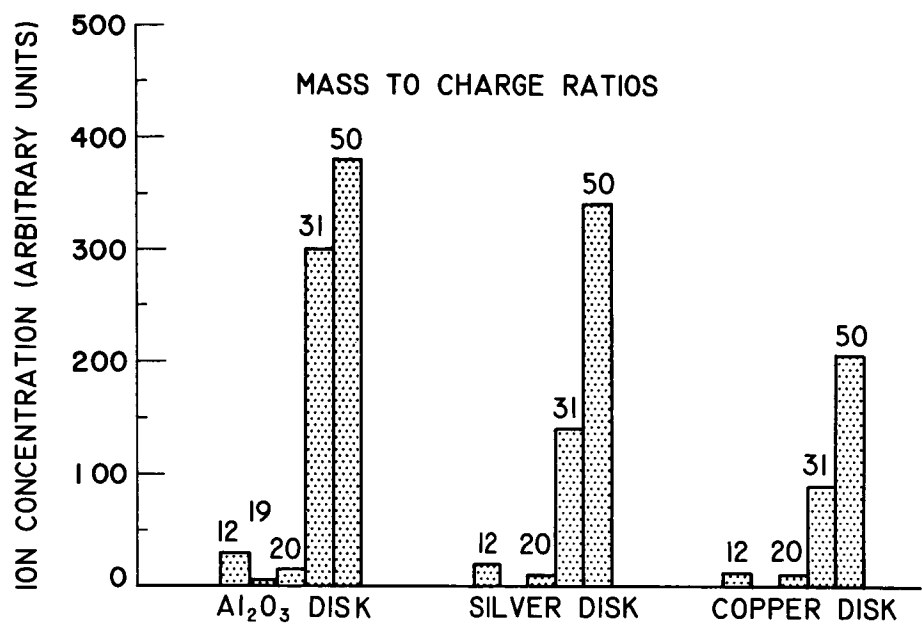


Fig. 11(b). - Mass to charge ratios for PTFE decomposition products obtained in vacuum friction studies. 15% glass-PTFE rider; load, 1000 G; sliding velocity, 390 ft/min; ambient pressure,  $10^{-9}$  mm Hg; no external heating of specimen.

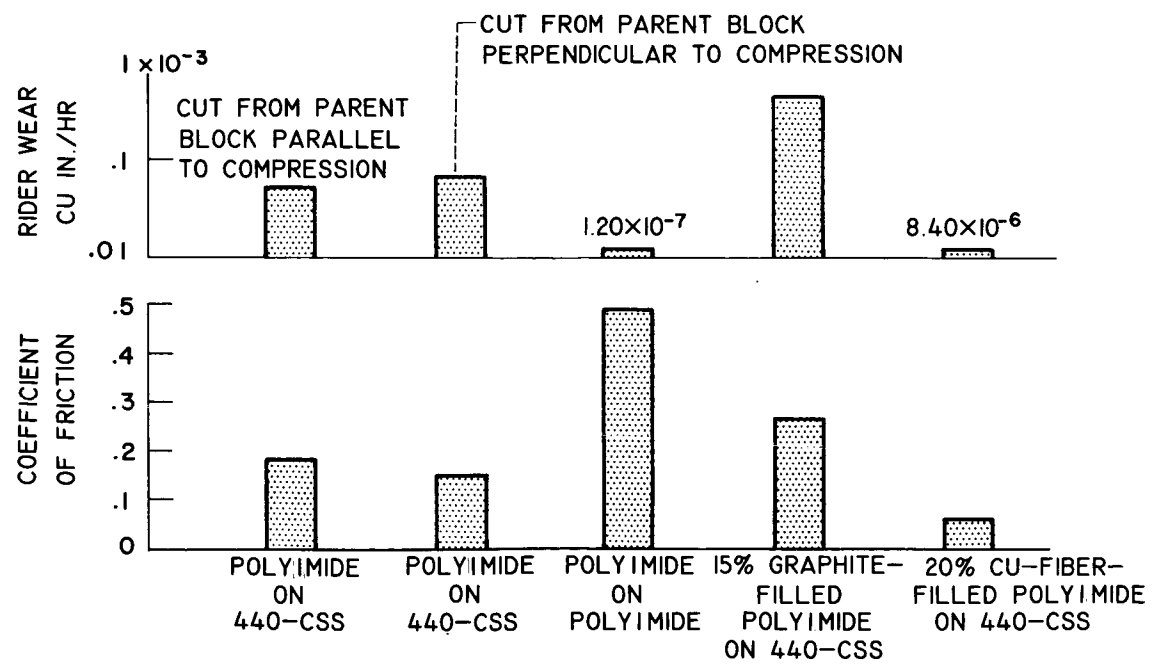


Fig. 12(a). - Friction and wear of polyimide on polyimide and 440-C S.S. in vacuum. Sliding velocity, 390 ft/min; load, 1000 G; ambient pressure,  $10^{-9}$  mm Hg; no external specimen heating.