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A Solid Film Lubricant Composition for Use at High Sliding Velocities in Liquid Nitrogen

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Solid-lubricant-containing compositions can be of value as films and solid bodies for bearing and seal surfaces in low-temperature liquefied gases. An experimental composition including polytetrafluoroethylene (PTFE), an epoxy resin, and lithium-alumina-silicate was studied in friction, wear, and endurance experiments in liquid nitrogen (-320°F). This composition was formulated to approximate the thermal expansion of metals used in cryogenic systems. Hemisphere ($\frac{3}{16}$ -in. radius) rider specimens were used and in most experiments the load was 1000 g.

Films (0.005-in. thick) on disk specimens gave good endurance life, low rider wear, and desirable friction ($f = 0.02$ to 0.07). They functioned at a higher sliding velocity (no failure at 16,000 ft/min) with copper rider specimens than with stainless steel riders (failure at 9000 ft/min).

Solid rider material of the experimental composition had good friction and wear properties at sliding velocities above 4000 ft/min. It is important to use the experimental composition with mating materials having good thermal conductivity.

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Introduction

CRYOGENIC fluids (low-temperature liquefied gases such as liquid nitrogen) are now useful engineering materials rather than laboratory curiosities. Their roles are becoming increasingly important in propulsion systems for missiles and space flight. In most instances the fluids are used in mechanical devices that include sliding surfaces subject to wear and surface failure; for example, pumps require bearings and seals that can or must be lubricated by the cryogenic liquids.

The physical and chemical properties of most cryogenic liquids are such that they might be expected to have very poor lubricating ability. Previous research has demonstrated that liquid nitrogen (-320°F) would not lubricate clean metal surfaces in sliding contact (1, 2) and that surface welding occurred. Thus it becomes apparent that mating sliding surfaces operating in fluids such as liquid nitrogen must have good friction and wear properties and preferably be self-lubricating.

Published data (1) show that solid materials and coatings of polytetrafluoroethylene (PTFE) can provide effective lubrication in liquid nitrogen. The usual limitations on the use of PTFE compositions as slider materials arise from poor thermal conductivity, making it difficult to dissipate frictional heat; their use with cryogenic fluids, however, has the advantage of utilizing the heat-removal capability of the low-temperature liquid. Further, PTFE has good mechanical properties at cryogenic temperatures (3).

Unreported experience has shown that there is a tendency for adhesive-bonded PTFE tape and resin-bonded

PTFE particles to spall from metal surfaces when immersed in liquid nitrogen at -320°F . The degree of spalling is dependent on the thickness of the adhesive or the film thickness of the resin-PTFE mixture; thicker films had greater tendency to spall. Spalling was attributed to differences in thermal expansion (i.e. shrinkage) of the coatings and the base metals.

Another problem caused by differences in thermal expansion has been found in dynamic seals for low-temperature fluids. Carbon nose pieces are normally shrunk into metal retainer housings. At the extreme low temperatures of operation, experience has shown that the retainer contracted more than the carbon and caused distortion of the nose piece sufficient to affect adversely essential radial and circumferential flatness.

Experimental compositions including PTFE as a lubricant, a modified epoxy resin, and a lithium-alumina-silicate ceramic filler were prepared. These compositions were intended to have thermal expansion approximating that of austenitic stainless steel.

The objective of the research reported was to determine the friction and wear characteristics of the special PTFE composition as a film material (on flat disk surfaces) and as a solid slider material (rider specimens) in liquid nitrogen.

Data were obtained at sliding velocities to 16,000 ft/min, and in most experiments the load was 1000 g with a $\frac{3}{16}$ -in. radius hemisphere rider specimen contacting the flat surfaces of a rotating $2\frac{1}{2}$ -in. diameter disk. Mating surfaces of 304 stainless steel or electrolytic copper were utilized.

Materials

The experimental composition including an epoxy, a ceramic, and PTFE was used to prepare surface films and rider specimens. The constituents of the mixture were selected for specific reasons. First, the epoxy was selected

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TABLE 1

Some Thermal Properties of Material

Components of base materials
(Numbers in parentheses refer to bibliography)

| Material | Specific gravity | Average expansion coefficient at RT | Expansion coefficient at -320 F | Total contraction 70 to -320 F, % (measured) | Average expansion coefficient 70 to -320 F (calc.) |
|--------------------------|------------------|---|---|--|--|
| PTFE | 2.33 | $55 \times 10^{-6}/^{\circ}\text{F}$ (3) | $47.5 \times 10^{-6}/^{\circ}\text{F}$ (3) | 1.9 | $48 \times 10^{-6}/^{\circ}\text{F}$ |
| Cured epoxy | 1.48 | $\approx 50 \times 10^{-6}/^{\circ}\text{F}$ | — | 1.2 | $32 \times 10^{-6}/^{\circ}\text{F}$ |
| Lithium-alumina-silicate | 2.40 | $-3.36 \times 10^{-6}/^{\circ}\text{F}$ (4) | — | -0.1* | $-2.5 \times 10^{-6}/^{\circ}\text{F}$ |
| 304 Stainless steel | 7.9 | $9.2 \times 10^{-6}/^{\circ}\text{F}$ (9) | $\approx 3.7 \times 10^{-6}/^{\circ}\text{F}$ (9) | 0.3 | $8.2 \times 10^{-6}/^{\circ}\text{F}$ |
| Electrolytic copper | 8.96 | $\approx 8.9 \times 10^{-6}/^{\circ}\text{F}$ (6) | $\approx 4.4 \times 10^{-6}/^{\circ}\text{F}$ (6) | 0.4 | $9.0 \times 10^{-6}/^{\circ}\text{F}$ |

* Cold-pressed solid body.

Experimental composition

(10% PTFE (8 mesh); 40% diglycidyl ether of bisphenyl-A; 50% lithium-alumina-silicate)

| | |
|--|--------------------------------------|
| Coefficient of expansion by weight (calc.) | $16 \times 10^{-6}/^{\circ}\text{F}$ |
| Coefficient of expansion by volume (calc.) | $19 \times 10^{-6}/^{\circ}\text{F}$ |
| Coefficient of expansion by volume (exp.) | $14 \times 10^{-6}/^{\circ}\text{F}$ |

for its adhesive strength at low temperatures, processing conveniences, and its lack of fillers. The ceramic (lithium-alumina-silicate) was selected primarily because of its large negative expansion coefficient and also for its fine particle size (to facilitate homogeneous mixing with epoxy). PTFE was used because it is a good lubricant and has exceptional mechanical properties at extremely low temperatures.

A formulation was obtained with approximately the same expansion coefficient as type-304 stainless steel (Table 1). It did not, however, have a consistency satisfactory for the application method. Decreased ceramic content and increased epoxy diluent gave a satisfactory consistency but resulted in a larger expansion coefficient. The first composition had a total contraction equal to 62% of the 304 stainless steel, and the second composition was equal to 169% of the stainless steel. A closer match would be possible with diluents presently available, but experience has shown that exact matching is unnecessary.

The mixture used to obtain the data reported was 10 parts PTFE (<8 mesh particle size), 40 parts epoxy base (diglycidyl ether of bisphenol-A), and 50 parts ceramic (lithium-alumina-silicate) (<325 mesh) by weight plus 4 parts amine catalyst. The dry powders were mixed thoroughly and then added to the epoxy to which the catalyst had been added. It was important that all materials used were free from contamination.

Surfaces for coating were vapor-blasted, scrubbed with levigated alumina, and rinsed in ACS certified acetone. The paste was applied with a spatula and cured in air at 200 F for 2 hr. The specimens were then easily machined to required thickness using a carbide-tipped tool. The machined coatings had surface roughness of 16 to 32 r.m.s. Final film thickness was 0.004 to 0.006 in.

Rider specimens were made by casting the paste mixture into a mold lightly coated with a silicone grease. After curing, the specimen was removed from the mold and machined.

In the curing of cast solids such as slider specimens,

the temperature had to be raised slowly or large voids and distortion of the body occurred. Small subsurface voids were hard to prevent and normally were of little concern.

The epoxy system used had considerable versatility that made it possible to match the contraction range of common metals. With the epoxy, successful coatings have 0 to 10% PTFE and 50 to 70% ceramic were formulated.

The ceramic composition has superior mechanical properties compared with the unfilled epoxy. A type-304 stainless-steel disk having the PTFE-epoxy-ceramic coating was quenched in liquid nitrogen at -320 F. While cold, the coating survived repeated blows with a ball-pene hammer without spalling. After removal of coating, the supporting disk was found to be indented by the hammering.

The coatings were applied to type 304 stainless steel disks. Metal rider specimens were type 304 stainless steel and electrolytic copper (99.9+%).

Apparatus and procedure

The apparatus used in this investigation is shown in Fig. 1. The basic elements consist of a rotating-disk specimen ($2\frac{1}{2}$ in. diam., $\frac{1}{2}$ in. thick) and a hemisphere-tipped ($\frac{3}{16}$ in. rad.) rider specimen. The rider specimen slides in a circumferential path on the lower flat surface of the rotating disk. The specimens were run submerged in liquid nitrogen. Varied surface speeds up to 16,000 ft/min were used for the data reported herein.

The rider specimen was supported by an arm assembly that allowed the measurement of friction force outside the test chamber. The vertical shaft of this assembly was pivoted through a bearing assembly mounted in the top housing. Friction force was measured by a strain-gage dynamometer ring connected to the top of the shaft. The deflection necessary for friction measurement was so small (0.003 in. at the top) that the restraining spring action of the bellows was negligible.

The rider specimen was loaded (1000 g) with a helium-pressurized piston assembly. The internal pressure of the

test chamber was held at a gage pressure of $1\frac{1}{2}$ psi by a pressure relief valve in the vent line.

The liquid nitrogen was transferred to the test chamber through a closed system (1). The Dewar was pressurized from 2 to 6 psi to transfer the liquid. It was possible to maintain the desired flow rate to maintain the proper liquid level during an experiment (about 3 in. above the test specimens) by controlling the Dewar pressure.

The metal disks and riders were all finish-ground on the test surface and had surface roughness from 4 to 8 micro-inches root mean square as measured with a profilometer. The non-metallic rider specimens were finished on a lathe. The radius ($\frac{3}{16}$ in.) of each rider specimen was checked with a radius gage.

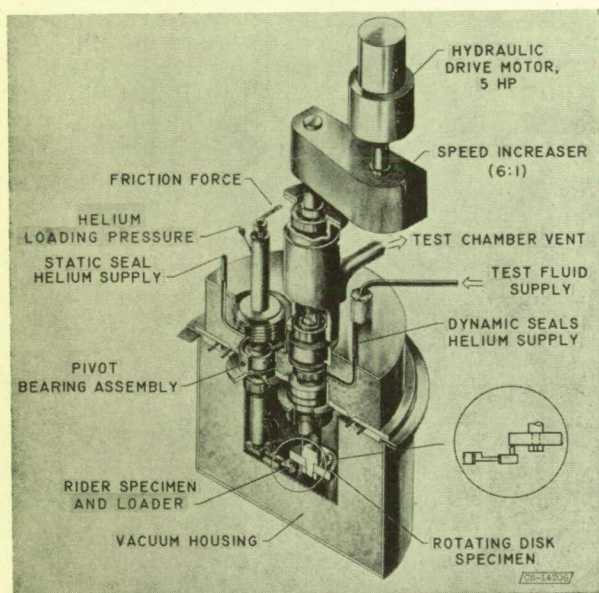


FIG. 1. Cryogenic friction apparatus.

The metal specimens were cleaned by the following procedure: (a) washed with acetone, (b) repeatedly scrubbed with moist levigated alumina, (c) washed in tap water, (d) washed in distilled water, (e) washed in 95% ethyl alcohol or ACS certified acetone, and (f) dried in clean warm air and stored in a desiccator.

The plastic specimens were also cleaned with acetone and stored in a desiccator. The test chamber was cleaned with acetone just prior to each run.

After stabilization of the liquid level, the drive motor was adjusted to the proper speed. The load was then applied to begin the experiment. Frictional force was measured by a recording potentiometer used as a strain indicator. Most runs were of 1-hr duration. The wear of the rider specimens was determined by measuring the diameter of the wear scar and calculating wear volume.

Results and discussion

Coatings

A number of solid lubricants have characteristics suggesting they should be useful in cryogenic liquids. As mentioned earlier, one of the problems with such coatings

in cryogenic liquids has been poor adherence that was manifested in cracking or spalling of the films. A basic reason for the spalling appeared to be poor matching of thermal expansion properties.

Commercial resin-bonded PTFE films of various thickness have been exposed to liquid nitrogen. Only the least thick (0.001 in.) coating would adhere to the 304 stainless steel disk; thicker coatings of this composition spalled from the disk. Friction, wear, and endurance data for this commercial coating (0.001 in. thick) and several other coatings of interest are shown in Fig. 2 for comparison with the experimental coating of this investigation. These reference coatings are described elsewhere (2, 4, 5).

The experimental coating of this investigation (0.005 in. thick) shows acceptable (0.07) but slightly higher friction than other coatings containing PTFE under the conditions reported in Fig. 2. The endurance of the material is, how-

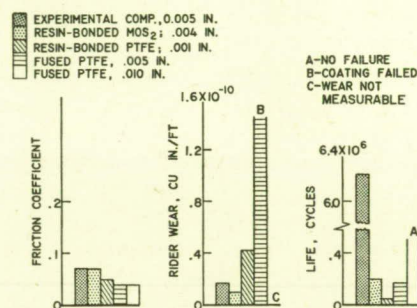


FIG. 2. Friction, wear, and endurance of various solid lubricant coatings in liquid nitrogen. Experimental composition, 10% PTFE, 40% epoxy and 50% lithium-alumina-silicate. Rider material, type 304 stainless steel; load, 1000 g; sliding velocity, 2300 ft/min.

ever, superior to all coatings except possibly the 0.010-in. thick fused PTFE, which was not run to failure. Adherence of the fused PTFE coatings was satisfactory in liquid nitrogen; however, previous data (2) show that relatively thick films are required for good endurance as a slider material; the thick films are built up by repeated application of 0.0005-in. layers. The high-temperature sintering (700 F) required for each layer of the fused coating limits its usefulness in certain cases. Coating thickness was also of primary importance to the endurance of resin-bonded molybdenum disulfide (MoS_2). The MoS_2 coating (0.004 in. thick) used to obtain the data of Fig. 2 was much thicker than usual commercial MoS_2 films (0.0003 in.).

Considering the results of data in Fig. 2 and Reference (2) as well as unreported liquid-nitrogen quenching experiments, contradictory requirements become evident. Best film adherence was obtained with the coatings of least thickness, but best endurance in sliding was with the coatings having greatest thickness. The experimental coating composition, however, was not subject to film-adherence failure. Therefore, coating thickness could be selected on the basis of friction, wear, and endurance properties, or the most convenient thickness for processing. When necessary, similar coating compositions can be formulated to use with other cryogenic liquids and base metals.

The 0.005 in. thick experimental coating (PTFE-epoxy-ceramic composition) was run over a range of sliding velocities to 16,000 ft/min. For stainless-steel rider specimens the friction coefficient decreased from 0.07 to 2300 ft/min to a stable value of 0.02 at 6000 ft/min and higher sliding velocities. The coating failed abruptly during runs at 9000 ft/min. The appearance of the specimens suggested that, in spite of the cryogenic environment, failure had resulted from poor dissipation of frictional heat from the riders. Data were therefore obtained with copper rider specimens to determine whether improved thermal conductivity of the rider would enable operation at higher sliding velocities. The results (Fig. 3) show that copper specimens allowed operation at up to 16,000 ft/min.

Wear of the copper and stainless steel rider specimens at comparable sliding velocities are shown in Fig. 4. Periodic unstable operation of the apparatus at a 16,000 ft/min sliding velocity prevented obtaining dependable wear data. No film failure was, however, experienced with copper rider specimens at even the maximum sliding velocity.

Photomicrographs of stainless steel rider wear areas and of disk wear tracks on the coated disks for two sliding

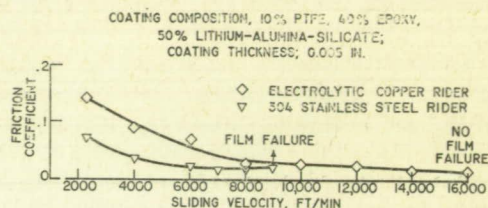


FIG. 3. Friction of type 304 stainless steel and electrolytic copper riders on PTFE-epoxy-ceramic coating in liquid nitrogen at varied sliding velocities. Load, 1000 g.

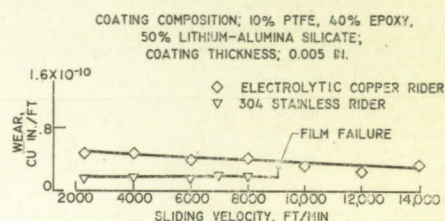


FIG. 4. Wear of type 304 stainless steel and electrolytic copper riders on PTFE-epoxy-ceramic coating in liquid nitrogen at varied sliding velocities. Load, 1000 g.

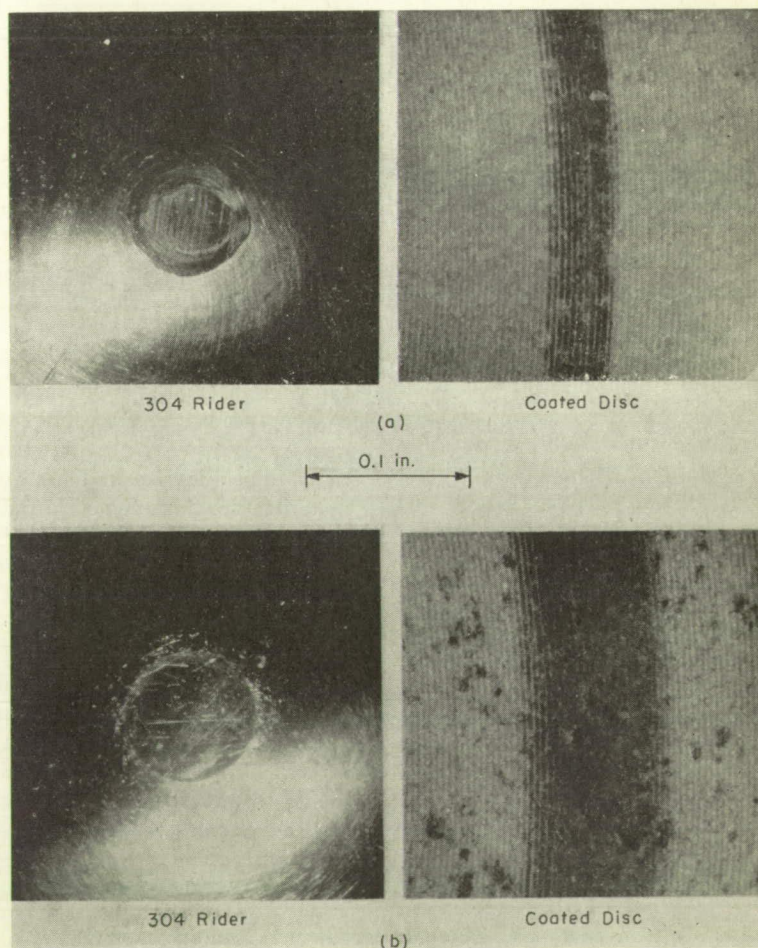


FIG. 5. Surfaces of specimens after experiments in liquid nitrogen using type 304 stainless steel disks coated with PTFE-epoxy-ceramic. Load, 1000 g; duration, 1 hr; magnification 15 \times .

(a) Sliding velocity, 2300 ft/min.

(b) Sliding velocity, 8000 ft/min.

velocities are presented in Fig. 5. The rider in Fig. 5(a) shows a contact area circumscribing the area of normal abrasive wear. The width of the wear track on the disk suggests that the circumscribing area of the rider was not exerting substantial pressure of contact on the film material. Perhaps this contact zone resulted from elasticity in the coating. The elongation of the abrasive wear area normal to the direction of sliding is a result of plasticity in the mating surface. The surfaces from the higher sliding velocity run, Fig. 5(b), showed sufficient abrasive wear so that the area exceeded the circumscribing portion on the rider of Fig. 5(a).

The total wear at the higher sliding velocity is a reflection of the total distance of sliding; wear rate per unit distance of sliding was actually the same at both sliding velocities (Fig. 4). The wear process with this coating is essentially abrasive wear contributed largely by the ceramic constituent in the composition. The wear tracks of Fig. 5 continue to show original finishing marks indicating there was very little wear or other types of degradation to these coatings. The disk of Fig. 5(b) show surface voids caused by gas evolution from the epoxy-catalyst reaction during the curing process. The voids had no apparent effect on the friction, wear, or endurance life of the coating provided they were of small size.

Figure 6 provides comparable friction data over a range

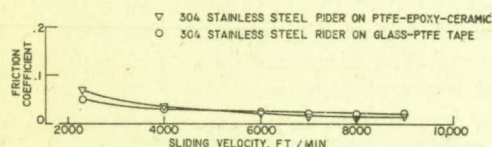


FIG. 6. Friction of type 304 stainless steel riders on PTFE-epoxy-ceramic and on glass-fiber-filled-PTFE bonded tape coatings in liquid nitrogen at varied sliding velocities. Load, 1000 g.

of sliding velocities to failure for the PTFE-epoxy-ceramic composition of this investigation compared with another useful PTFE (0.030 in. thick) coating. The comparative coating is a skived tape of PTFE containing about 25% glass fibers. One surface of this tape was etched so that it could be bonded to the flat surface of a metal disk using an epoxy adhesive. With austenitic stainless steel rider specimens, friction values were very similar.

Film failure occurred at 8000 ft/min with the glass-filled PTFE tape as compared with the 9000 ft/min failure point for the PTFE-epoxy-ceramic composition. Friction data (Fig. 6) at the maximum sliding velocities/were obtained during the time increments (20 to 35 min) preceding failure. The rider wear with the two film materials is presented in Fig. 7. Since the failure of the PTFE tape was

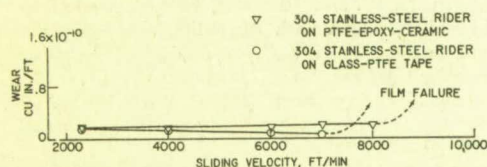


FIG. 7. Wear of type 304 stainless steel riders on PTFE-epoxy-ceramic and on glass fiber filled PTFE bonded tape coatings in liquid nitrogen at varied sliding velocities. Load, 1000 g.

thought due to thermal degradation, an electrolytic copper rider was used in place of the stainless steel. With a sliding velocity of 9000 ft/min using the copper rider, no film failure was experienced nor was failure evident after a 60-min run.

The data of Figs. 6 and 7 show that PTFE tape can provide a very effective surface film for lubrication. A primary disadvantage to its use, however, is that bonding is cumbersome and very critical. The adhesive system and method of application must be carefully developed for every problem area.

Solid Bodies

Solid bodies with thermal expansion properties approximately matching those of adjacent metal housings have merit for use in seals and bearings. In both seals and bearings, dimensional stability over extreme temperature ranges (e.g. 75 to -320 F) can be important for sliding contact surfaces. For face-type seals, sliding contact surfaces are finished to a flatness of under three helium light bands. Constriction of the nosepiece by its mounting housing in cryogenic applications has caused dishing and other modes of distortion of the critical running face. With low-viscosity liquids such as cryogenic fuels and oxidants, distortion can allow prohibitive leakage past contact seals.

The PTFE-epoxy-ceramic composition of this investigation can easily be formed into solid bodies of configurations useful for seals and possibly for bearings. Previous research with PTFE materials (1) such as an extruded-glass-fiber-filled composition indicated promise as sliders operating in cryogenic liquids. Figure 8 presents comparative wear data

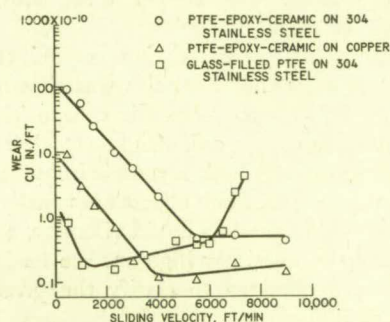


FIG. 8. Wear of cast PTFE-epoxy-ceramic riders on type 304 stainless steel and electrolytic copper disks in liquid nitrogen at varied sliding velocities. Load, 1000 g.

for rider specimens of the PTFE-epoxy-ceramic composition and the 25% glass fiber filled PTFE. Different types of critical wear behavior are noted. For the experimental composition, the initial high wear rate decreased with increased sliding velocity according to a negative exponential function up to 5500 ft/min. Above that point there was little effect of sliding velocity on wear. The glass-filled composition had lower initial wear, but above 5500 ft/min sliding velocity wear rate increased approximately according to a positive exponential function. Thus, the PTFE-epoxy-ceramic composition might be considered to have its greatest utility at higher sliding velocities.

Experience with PTFE and other plastic compositions has shown that their behavior as slider materials is often critically influenced by ability to dissipate frictional heat. To determine the importance of this factor with the PTFE-epoxy-ceramic composition, data were obtained with electrolytic copper (high thermal conductivity) disk specimens for comparison with the data for type 304 stainless steel (low thermal conductivity) disk specimens. The data of Fig. 9 show that, with the exception of low sliding

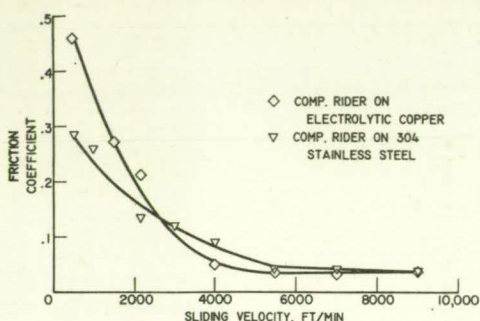


FIG. 9. Friction of PTFE-epoxy-ceramic riders on type 304 stainless steel and electrolytic copper disks in liquid nitrogen at varied sliding velocities. Load, 1000 g.

velocities, there were no substantial differences in friction for the experimental composition rider specimens on copper and on stainless steel. There were, however, very substantial differences in wear (Fig. 8) that may have been influenced by thermal conductivity. Rider wear with the copper disks was one-fifth to one-tenth that with the stainless steel disks. The copper disks showed only a brightly polished track.

Substantial reduction in friction coefficient and rider wear with greater sliding velocities was shown in Figs. 8 and 9 for the PTFE-epoxy-ceramic composition on disks of both electrolytic copper and stainless steel. The question logically arises as to any hydrodynamic influence the liquid nitrogen may have had. Such effects are usually considered negligible because of the low liquid viscosity, the specimen geometry, and the relatively high surface loads. The data of Fig. 10 were obtained to clarify the possibility of a hydrodynamic influence.

Plastic materials usually show a decrease in friction coefficient with increased loads. This effect was obtained for loads greater than 750 g (Fig. 10). Hydrodynamic lift

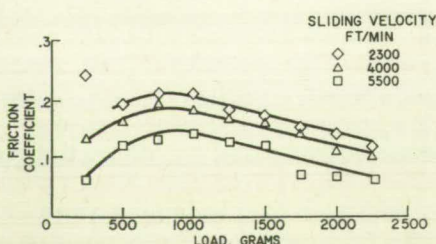


FIG. 10. Friction of type 304 stainless steel riders on PTFE-epoxy-ceramic coatings in liquid nitrogen at varied loads and speeds.

on the rider would result in a finite reduction in load at each sliding velocity. The relative effect of this load reduction would be reduced as greater applied loads were used. Therefore, if a substantial hydrodynamic component were present, the three friction coefficient curves for different sliding velocities in Fig. 10 would converge at the greater loads. The three curves are essentially equidistant from each other at most loads. Therefore, any hydrodynamic component present was insufficient to be considered important to these experiments.

Summary of results

Experimental friction and wear data were obtained in liquid nitrogen for a composition material that included polytetrafluoroethylene (PTFE), an epoxy resin, and lithium-alumina-silicate (ceramic). The composition was used both as a solid film lubricant material and as a solid slider material. It has the advantage of thermal expansion approaching that of structural metals. The following results were obtained:

1. Surface films of the experimental composition (0.005 in. thick) on type 304 stainless steel gave acceptable friction (0.02 to 0.07) and rider wear and outstanding endurance properties as compared with other solid lubricant films. Friction decreased with increasing sliding velocities, and wear rate either decreased or was independent of sliding velocities. A critical sliding velocity for film failure was encountered. The failure sliding velocity for films on type 304 stainless-steel disks was 9000 ft/min with type 304 rider specimens; with copper rider specimens the film did not fail at 16,000 ft/min.

2. Solid bodies of the experimental material cast into rider specimens show promise for use in liquid nitrogen at sliding velocities of 4000 ft/min and higher. At the higher sliding velocities, friction coefficients less than 0.10 were obtained. Both wear and friction were relatively high at low sliding velocities. Lower rider wear was experienced with copper disk specimens than with stainless-steel disks.

3. The use of high thermal conductivity metals in sliding contact with the experimental material has real advantage. At liquid nitrogen temperatures, the sliding velocity for film failure with copper disks was more than twice that with the stainless-steel substrate.

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