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SUMMARY

Although fused-fluoride solid lubricants exhibit low wear and long lives from 70° to 1500° F (21° to 816° C) in air, at low sliding velocities their friction is high at temperatures from 70° to 900° F (21° to 482° C). An experimental program was conducted to reduce this low-temperature friction by the addition of solid lubricants such as molybdenum disulfide (MoS_2) and silver, which are effective at low temperatures. The friction experiments were conducted in air with a pin-on-disk apparatus. Friction coefficients were measured on each coated disk from 70° to 1200° F (21° to 649° C) or higher and back to 70° F (21° C) where applicable.

While MoS_2 additions could be used only once because of their oxidation above 750° F (399° C), they did greatly reduce the friction from 70° to 600° F (21° to 316° C). Better results were obtained if MoS_2 was incorporated into the coating rather than applied over the fused-fluoride coating. Silver was added to calcium fluoride - barium fluoride slurries either as reducible silver halides or as elemental silver powder. The friction coefficients of coatings with 35 weight percent silver powder were 0.2 or less at temperatures from 70° to 1400° F (21° to 760° C), and less than 0.3 during cooling to 70° F (21° C).

While they reduced low-temperature friction, neither MoS_2 nor silver additions adversely affected the wear of the specimens or the life of the fused-fluoride coatings under the conditions of these experiments.

INTRODUCTION

The technology of solid lubricants has reached the point where, for a particular set of conditions, a satisfactory lubricant can frequently be selected or formulated. However, a solid lubricant is needed for use over a wider range of conditions (temperature, atmosphere, and load) than is possible with lubricants presently available. In this study, the temperature range was extended; the atmosphere (air) and the load were kept constant.

In the temperature range 70° to 600° F (21° to 316° C), molybdenum disulfide (MoS_2) is among the best-known solid lubricants (refs. 1 and 2). Although it performs well in inert atmospheres to at least 1300° F (704° C) (ref. 3), in air, it begins to oxidize rapidly at 750° F (399° C) (ref. 4). For MoS_2 in air, the useful temperature limit (which is usually dependent on the bonding agent) is most commonly about 600° F (316° C).

In another temperature range, 900° to 1500° F (482° to 816° C), fused coatings of calcium fluoride - barium fluoride mixtures (CaF_2-BaF_2) are effective (ref. 5). Although the fused-fluoride coatings perform especially well above 900° F (482° C), they do provide surface protection in the lower range, 70° to 900° F (21° to 482° C). They have exhibited low wear and long lives in air from 70° to 1500° F (21° to 816° C) (ref. 5); however, friction at the lower temperatures is much higher than that of MoS_2.

Silver films give a satisfactory coefficient of friction over a wide range of temperatures and atmospheres, but they are not long-lived and are difficult to bond (ref. 6). Friction and evaporation data for all three materials, silver (refs. 7 and 8), CaF_2-BaF_2 (refs. 5 and 7), and MoS_2 (refs. 7 to 10), indicate that they have potential for use in vacuum (10^{-6} mm Hg or better) to temperatures as high as 1000° F (538° C).

The objective of this study was to reduce the friction of the fused fluoride (CaF_2-BaF_2) coating at low temperatures (70° to 900° F). Experiments were conducted in which

(1) MoS_2 was added to the fused-fluoride coating to reduce the friction at low temperatures for circumstances in which lubrication would be required only once at 70° F (21° C), and the temperature would then remain above 900° F (482° C).

(2) Silver was added to the fused fluorides to evaluate the combined lubrication benefits for circumstances in which lubrication would be required for a complete temperature cycle. The friction experiments were conducted with a pin-on-disk apparatus in air. Friction coefficients were generally measured on each coated disk over the temperature cycle 70° to 1200° F (21° to 649° C) or higher and back to 70° F (21° C).

FRICTION APPARATUS AND EXPERIMENTAL PROCEDURE

The friction and wear apparatus used is shown in figure 1. A $2\frac{1}{2}$-inch-diameter (6.35-cm-diam) rotating disk slides in contact with a 3/16-inch-radius (0.476-cm-rad) hemispherically tipped rider under a normal load of 500 grams. The rider describes a 2-inch-diameter (5.08-cm-diam) track on the disk; in all cases the specimen coated is the disk, never the rider. Friction torque is measured with strain gages and continuously

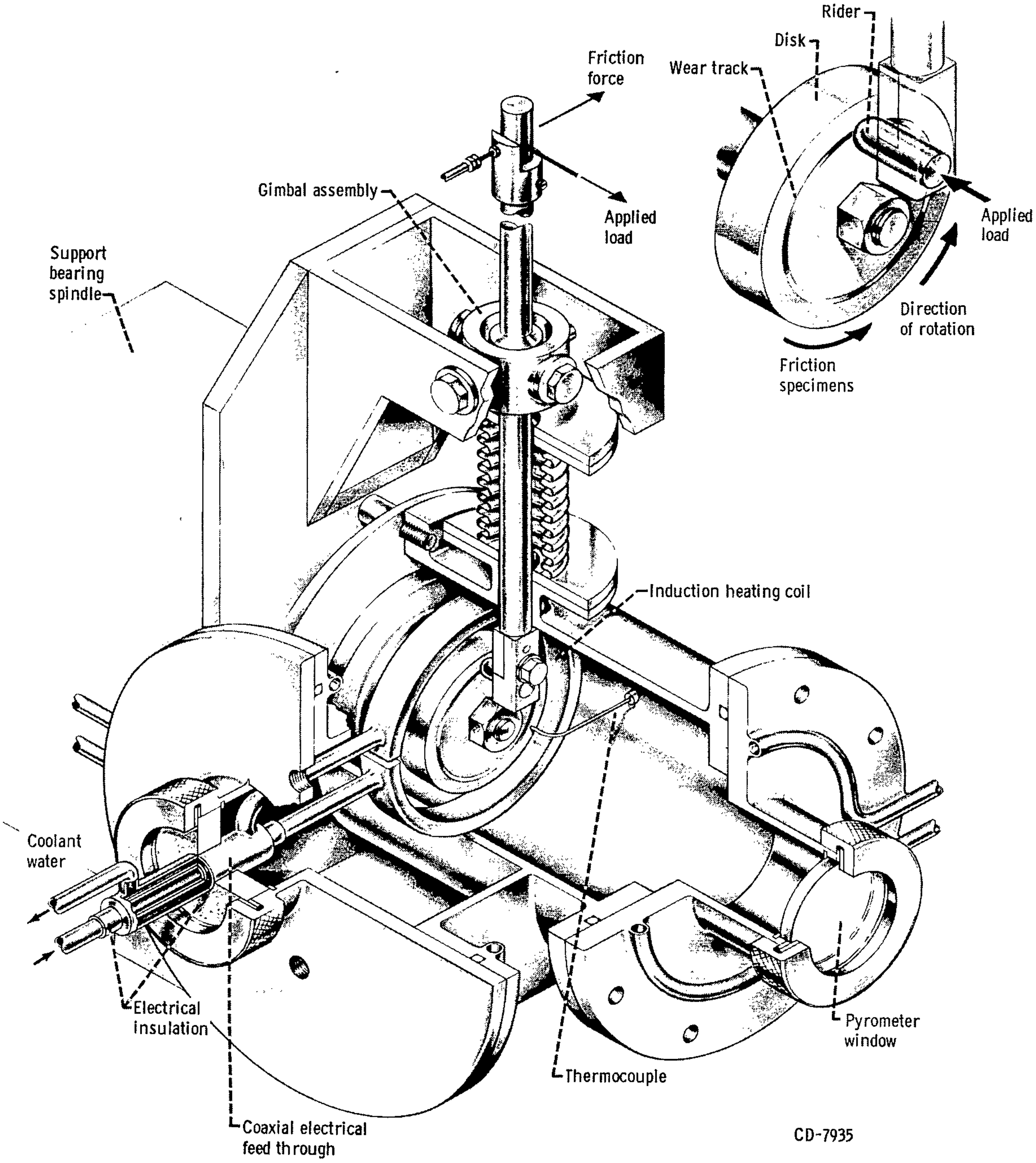


Figure 1. - High-temperature friction apparatus.

recorded. Temperature is measured with an infrared optical pyrometer.

In this program the sliding velocity was maintained at 450 feet per minute (137 m/min), a slow speed specifically chosen to exaggerate the tendency of the fused-fluoride coating toward high friction below 900° F (482° C) (ref. 5). Each friction data point reported was obtained during a 5-minute period at the indicated temperature. Unless otherwise indicated, each curve represents one specimen set of rider and disk used over the entire temperature range. Temperatures were cycled from 70° F (21° C) to the maximum in each case and back to 70° F (21° C) where applicable; sometimes there was a second cycle.

The disks used in this investigation were Inconel 750; the riders were cast Inconel. A description of the disk coating materials may be seen in table I.

TABLE I. - DESCRIPTION OF DISK COATING MATERIALS

Material	Purity	Size
Calcium fluoride (CaF_2) Barium fluoride (BaF_2)	Reagent grade	-325 Mesh
Silver chloride (AgCl) Silver bromide (AgBr) Silver iodide (AgI)	Reagent grade	Received in lump form, crushed or ball-milled to -325 mesh
Silver fluoride (AgF)	Technical grade	Received in lump form, dissolved in distilled water and filtered
Silver (Ag)	Technical grade	Submicron
Molybdenum disulfide (MoS_2)	99 Percent pure; 1 percent impurities (oil, water, iron sulfide and silicon dioxide)	-200 Mesh
Molybdenum disulfide spray (MoS_2)	Submicron molybdenum disulfide suspended in air-drying resin	
Lacquer	Cellulose nitrate solution	

COATING PROCEDURE

All disks were sandblasted and cleaned in alcohol before spraying. Coatings were applied to the disks only.

Water-Base Slurries

Coatings of CaF_2-BaF_2 without additions, and those with additions of MoS_2 were mixed with enough distilled water to give a sprayable consistency. (A sprayable consistency was obtained by initially mixing powder and distilled water in a 1-to-1 ratio by weight and then adding more distilled water if necessary. The final ratio of powder to water was not critical.) With occasional mixing, CaF_2, BaF_2, and MoS_2 remained in water suspension long enough for spraying. When silver fluoride (AgF) was to be part of a fluoride mixture, it was dissolved in distilled water and filtered to remove possible impurities. Then CaF_2 and BaF_2 powders were added, and the proportion of water was adjusted. The amount of AgF required in the slurry to give the correct amount of silver in the coating was calculated by assuming complete chemical reduction of the AgF to silver during firing. These coatings were sprayed onto a preheated (200° F) (93° C) disk with an artist's airbrush. The coating dried on contact with the disk, and coating thickness was built up to 0.0015 inch (0.0038 cm).

Lacquer-Base Slurries

Because of their high specific gravities, silver chloride (AgCl), silver bromide (AgBr), silver iodide (AgI), and silver (Ag) could not be held in water suspension for any reasonable spraying time. Therefore, a cellulose nitrate lacquer was used as the suspending agent for slurries containing these powders. Lacquer was added to the various silver powders until a sprayable consistency was obtained, in the same manner as for the water-based slurries. Again, the exact amount of lacquer was not critical. The amount of AgCl, AgBr, and AgI required in their respective slurries to yield the correct amount of silver in the coatings was calculated by assuming complete chemical reduction of the silver halide to silver during firing. A paint sprayer was used, and it was not necessary to preheat the disks. The thickness of these coatings after firing was also 0.0015 inch (0.0038 cm).

The appearance of the silver halides and the phases detected by X-ray diffraction at various stages in the coating process are summarized in table II. Indexing the coating diffraction patterns after firing was particularly difficult. All silver halide - fused-fluoride coatings after firing showed CaF_2, BaF_2, and silver in approximately the proportions calculated. However, coatings which had contained AgF or AgCl before firing sometimes exhibited on their X-ray diffraction patterns a number of peaks which could not be indexed through the ASTM powder-diffraction file. After longer firing and subsequent reexamination by X-ray diffraction, these peaks usually disappeared. The presence of these unclassified compound(s) apparently did not affect friction.

TABLE II. - VISUAL AND X-RAY DIFFRACTION EXAMINATION OF SILVER HALIDES AS RECEIVED, AS SPRAYED, AND AFTER FIRING

Halide	Appearance		Phases detected by X-ray diffraction		
	As received	As sprayed	As received	As sprayed	After firing
Silver fluoride (AgF)	Yellow; transparent when first dissolved in water, then turned grey to black	Grey to black; on standing or heating, turned yellow	AgF	Grey phase: AgF, small amounts of Ag_2F and silver oxide (Ag_2O) Yellow phase: Ag_2O, small amounts of Ag_2F, no AgF	Silver (Ag)
Silver chloride (AgCl)	Slight purple discoloration darkened on exposure to light	Light purple	AgCl	AgCl	Ag
Silver bromide (AgBr)	Yellow	Yellow	AgBr	AgBr	Ag
Silver iodide (AgI)	Yellow	Yellow	AgI	AgI	Ag

Firing of Coatings

Standard firing conditions for the fused-fluoride coatings in this study were 1950° F (1066° C) for 15 minutes in hydrogen. (In other work the time or temperature may vary if the size or geometry of the parts varies from that used in these experiments.) The furnace operated continuously, with flowing hydrogen. The disks were placed in a purge chamber for a few minutes, then pushed into the hot zone, where they remained for the nominal firing time, and finally pushed into a cooling zone. Additions to the fused-fluoride coatings caused the following variations from the standard firing conditions:

(1) Since MoS_2 is reduced in hydrogen to molybdenum, MoS_2 - fused-fluoride coatings were fired in inert atmospheres. One series was fired at 1900° F (1038° C) for 15 minutes in nitrogen, another at 1750° F (954° C) for 10 minutes in argon. A further exception to the standard conditions for those coatings fired in argon was that a batch type furnace was used. Since it required 1 to 2 hours to reach the firing temperature, firing conditions for this series of disks were different from the conditions indicated nominally.

(2) No variation from standard firing conditions was required for any of the silver or silver halide additions to the coating, although coatings containing AgI could not be fired satisfactorily at any temperature tried (1700° to 2200° F) (927° to 1204° C). (Firing satisfactorily means that the coating showed melting, was uniform in texture and thick-

ness, and was bonded to the disk. The fused-fluoride coatings were soft, and all could be scratched with mild steel.)

Overlays

The MoS_2 overlays were very thin (0.0002 in., 0.0005 cm) and were applied over the fluoride coating by spraying or burnishing. The coated disks were cleaned in alcohol before the MoS_2 overlay was applied. The MoS_2 in air-drying resin was sprayed from a pressurized spray can; the MoS_2 powder was burnished by hand with a soft cotton cloth and the excess wiped off. In some cases MoS_2 was applied to the glazed wear track of a previously run fluoride coating.

DISCUSSION OF RESULTS

Coatings Without Additions

The lubricating properties of a coating can be evaluated by three criteria, the life of the coating, the wear of the rider in sliding contact with the coating, and the friction coefficient of the coating. The fused-fluoride coating has shown long coating life and low wear of the mating metal surface even under circumstances where the friction coefficient is high (ref. 5). This is illustrated in figure 2, where the friction coefficients and rider wear for unlubricated, MoS_2-lubricated, and fused-fluoride-coated specimens are compared at 70° and 1000° F (21° and 538° C). Coating life is not shown; however, the coating was assumed to be intact if the metal wear rate and the friction coefficient remained constant. This assumption was later verified by microscopic examination. In figure 2, it can be seen that at 70° F (21° C) and at the comparatively slow sliding velocity of 450 feet per minute (137 m/min) the friction coefficient of the fused-fluoride coating is high (0.4). The rider wear rate is low, which indicates that at 70° F (21° C) the fused-fluoride coating is adequate to ensure protection to the metal surface for at least a limited period of time (in this case, 1 hour). Based on the wear rate for MoS_2-lubricated specimens at 70° F (21° C) (fig. 2), rider wear below 10^{-10} cubic inch per foot of sliding (4.9×10^{-9} cm^3/m of sliding) will be considered low in this report.

All experiments with fused-fluoride coatings in the present study were also checked at a sliding velocity of 2000 feet per minute (610 m/min) to confirm that the friction coefficients at the higher speed would not exceed about 0.2 at temperatures from 70° to 900° F (21 to 482° C).

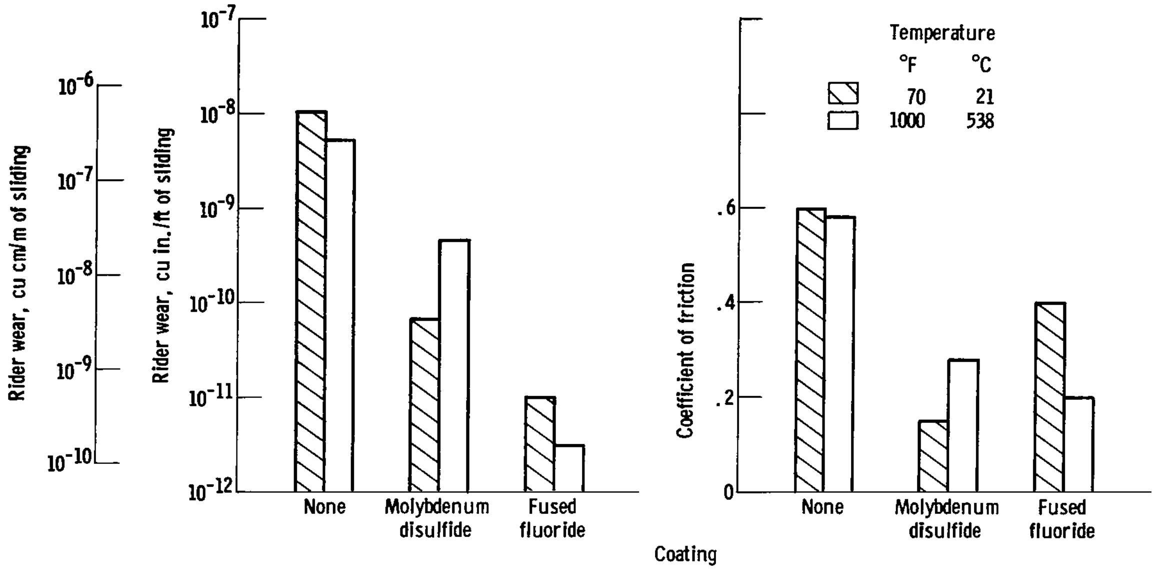


Figure 2. - Comparison of friction coefficient and rider wear of unlubricated, molybdenum disulfide - lubricated, and fused-fluoride-coated specimens at 70° and 1000° F (21° and 538° C). Atmosphere, air; sliding velocity, 450 feet per minute (137 m/min); load, 500 grams; rider, uncoated cast Inconel, 3/16-inch (0.476-cm) hemispherical radius; disk, coated or uncoated Inconel 750. Molybdenum disulfide coating was 0.0002 inch (0.0005 cm) thick and resin bonded; fluoride coating was 60 percent calcium fluoride and 40 percent barium fluoride.

Molybdenum Disulfide Additions

A simple way to reduce the friction at 70° F (21° C) and 450 feet per minute (137 m/min) is to apply MoS_2 over the fused-fluoride coating. In figure 3, the friction coefficients are given for fused-fluoride coatings, both with and without an MoS_2 overlay, over a temperature range of 70° to 1200° F (21° to 649° C). As expected, there was a dramatic drop in the friction coefficients, corresponding to the friction coefficient of MoS_2, at temperatures to 400° F (204° C). Upon oxidation of the MoS_2, the friction coefficient rose to that of the fused-fluoride coating. Oxidation of MoS_2 was readily observed by the color change from black MoS_2 to white molybdenum trioxide (MoO_3). After the experiment, no differences between the coatings with overlays and those without them could be detected either microscopically or by X-ray diffraction.

The MoS_2 completely oxidized to gaseous sulfur oxides and MoO_3, which presumably vaporized. A fused-fluoride coating which had been run in (tested at 1000° F (538° C) for 30 minutes before the MoS_2 overlay was applied) had a glazed wear track on the disk. When MoS_2 was applied over this glazed wear track, the disk showed the typically lower friction of MoS_2 to an appreciably higher temperature (600° F (316° C) rather than 400° F (204° C)). This process was repeated, and reproducibility was verified.

Rider wear data are included in these figures, because high wear usually indicates

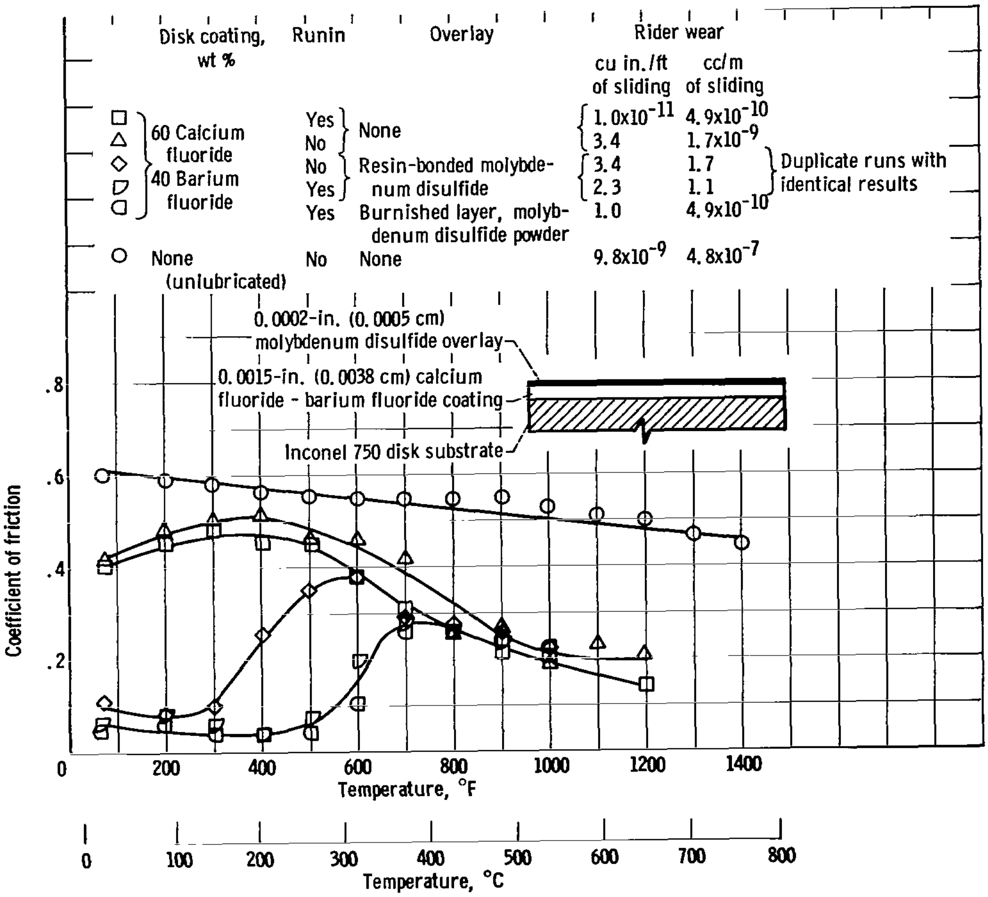


Figure 3. - Friction coefficients of fused-fluoride coating with and without molybdenum disulfide overlay. Atmosphere, air; sliding velocity, 450 feet per minute (137 m/min); load, 500 grams; rider, uncoated cast Inconel, 3/16-inch (0.476-cm) hemispherical radius; disk, coated Inconel 750. Run-in specimens were tested at 1000° F (538° C) for 30 minutes in air before molybdenum disulfide overlay was applied or friction experiment was begun.

coating failure. In general, the rate of rider-metal removal was low for all coatings discussed in this report. Moreover, if rider wear is below 10^{-10} cubic inch per foot of sliding (4.9×10^{-9} cu cm/m of sliding), generally no comparisons among coatings can be made.

When MoS_2 is incorporated into the coating before firing, a comparatively large amount of MoS_2 must be added to the fused fluorides. Nevertheless, the decomposition of the MoS_2 additions during testing did not affect the integrity of the fused-fluoride coating. The friction characteristics of two series of coatings containing MoS_2, one fired in nitrogen and the other in argon, are shown in figures 4 and 5. Although firing conditions were quite different for each coated-disk series, the object was the same in each case: to incorporate enough MoS_2 into the coating slurry so that a significant amount of MoS_2 would be left in the final coating.

No MoS_2 was detected by X-ray diffraction in either of the nominally 5-percent-MoS_2

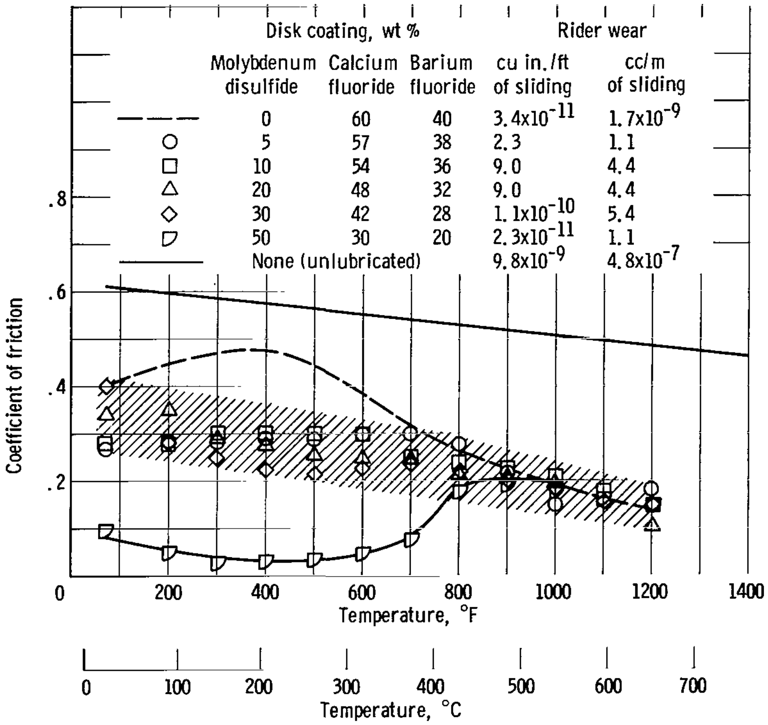


Figure 4. - Friction coefficients for molybdenum disulfide - fused-fluoride coatings fired in nitrogen for 15 minutes at 1900° F (1038° C). Atmosphere, air; sliding velocity, 450 feet per minute (137 m/min); load, 500 grams; rider, uncoated cast Inconel, 3/16-inch (0.476-cm) hemispherical radius; disk, Inconel 750 with 0.0015-inch (0.0038-cm) coating.

coatings. In the 50-percent-MoS_2 (nitrogen-fired) and 30-percent-MoS_2 (argon-fired) coatings, significantly large amounts of MoS_2 were detected. It was estimated that 20 to 25 percent of MoS_2 was left in these two coatings after firing. This was based on the X-ray diffraction patterns before and after firing, but no accurate quantitative analysis was made. In all the other coatings, small amounts of MoS_2 were identified. The MoS_2 not retained in the coatings evidentally was oxidized by water vapor or oxygen impurities in the furnace. No MoO_3 or Mo was identified by X-ray diffraction in any of the fired coatings. However, barium molybdate ($BaMoO_4$) was present on those coatings fired in nitrogen, the amount varying with the amount of MoS_2 in the original coating. After the friction and wear experiments, CaF_2, BaF_2, and varying amounts of $BaMoO_4$ were identified by X-ray diffraction in all the MoS_2 - fused-fluoride coatings. Since furnace atmospheres passed through a drier and entered the furnace with a dew point of approximately -70° F (-47° C), the most likely cause of oxidizing atmospheric impurities was incomplete purging either of specimens entering the furnace or of the furnace itself.

For MoS_2 - fused-fluoride coatings fired in nitrogen, those with up to 30 percent MoS_2 in the slurry (and with little MoS_2 retained in the coating after firing) fell into a specific band of values (fig. 4). The 50-percent-MoS_2 coating (with a significant amount

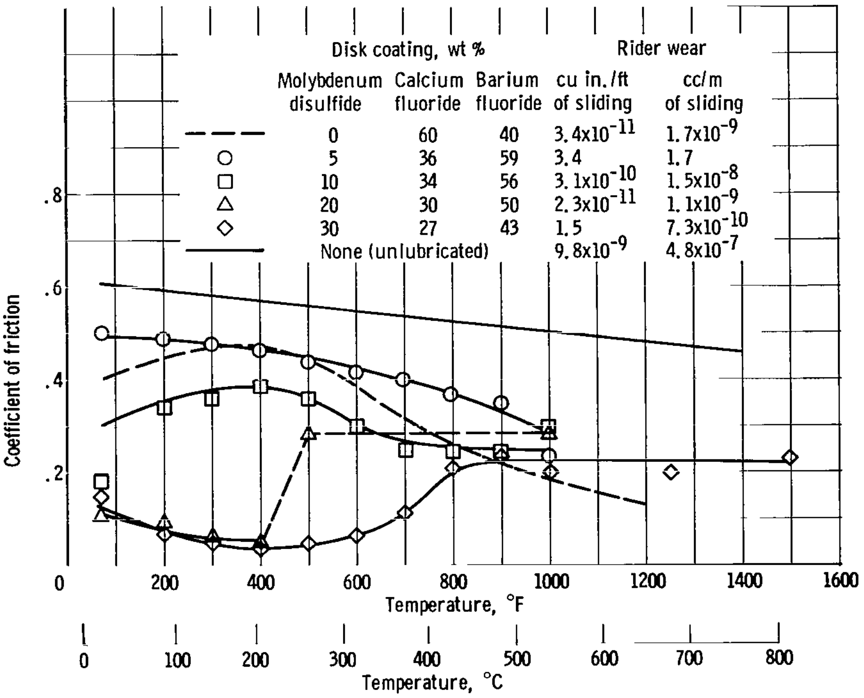


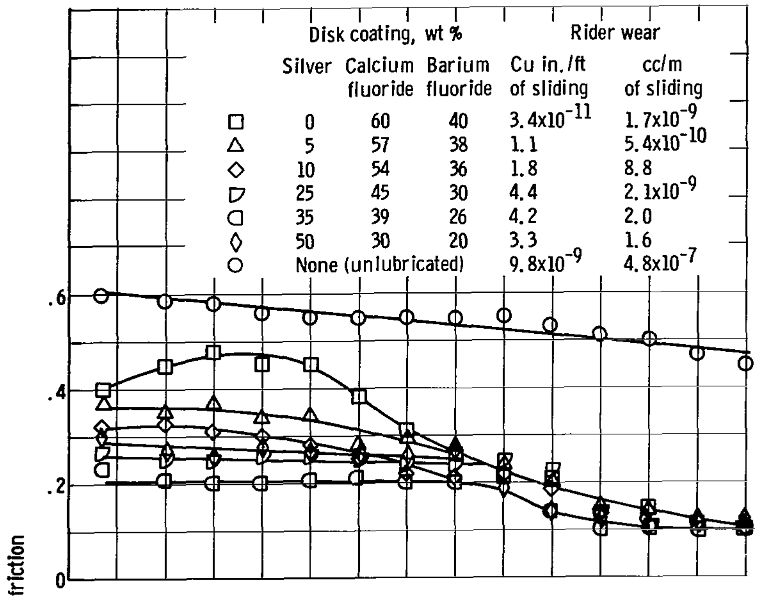
Figure 5. - Friction coefficients of molybdenum disulfide - eutectic calcium fluoride-barium fluoride coatings fired in argon for 10 minutes at 1750° F (954° C). Atmosphere, air; sliding velocity, 450 feet per minute (137 m/min); load, 500 grams; rider, uncoated cast Inconel, 3/16-inch (0.476-cm) hemispherical radius; disk, Inconel 750 with 0.0015-inch (0.0038-cm) coating. Eutectic fluoride mixture (38 percent calcium fluoride, 62 percent barium fluoride) gives identical performance as 60-40 mixture (ref. 5).

of MoS_2 present after firing) showed the typically low friction of MoS_2 to 700° F (371° C). Its friction coefficient did not rise above 0.2 at any temperature to 1200° F (649° C).

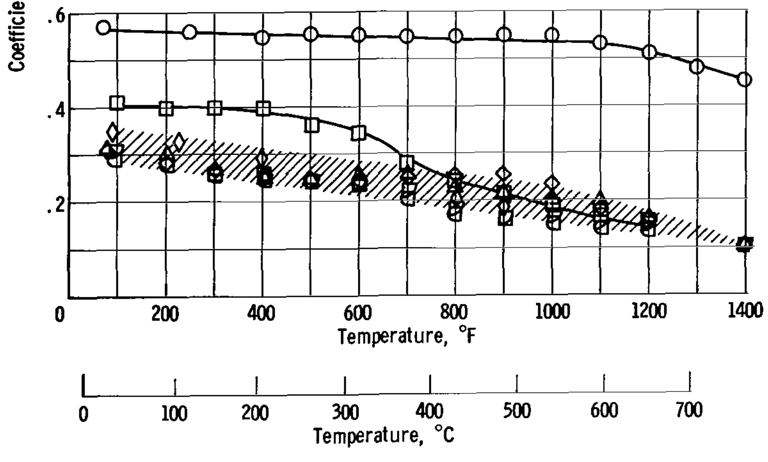
When fired in argon, the MoS_2 - fused-fluoride combinations showed friction coefficients that decreased with each increase in MoS_2 (fig. 5). At 20-percent MoS_2, friction was at 0.1 or below at temperatures to 400° F (204° C); at 500° F (260° C) there was sudden failure of the MoS_2 in the coating, but the fused fluorides continued to give surface protection. The friction coefficient for 30 percent MoS_2 (with a significant amount of MoS_2 present after firing), remained near or below 0.2 at temperatures to 1500° F (816° C). The data were almost identical to those of nitrogen-fired 50 percent MoS_2 (fig. 4). Both coatings exhibited satisfactory friction coefficients over the temperature range tested.

Silver Additions

Elemental silver was added to the fused-fluoride slurries in varying amounts up to 50 weight percent. The data in figure 6 show that friction coefficients gradully decreased with each successive silver addition, to 35 percent. The friction coefficients for the



(a) Increasing temperature.

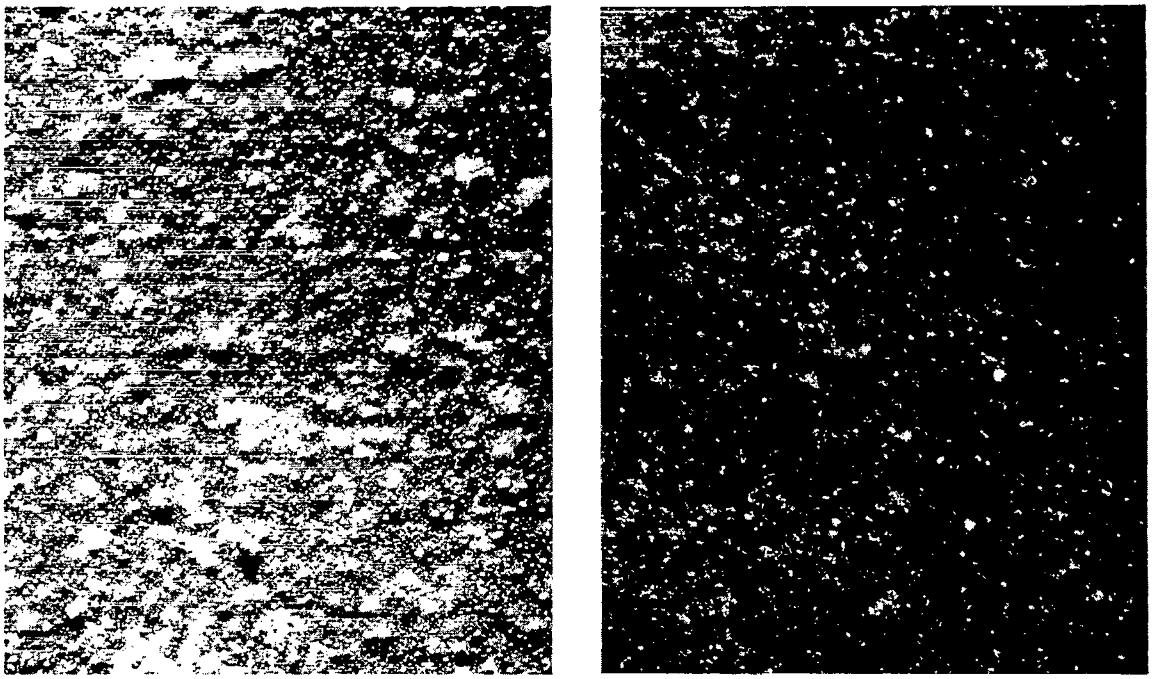


(b) Decreasing temperature.

Figure 6. - Friction coefficients of silver - fused-fluoride coatings fired in hydrogen for 15 minutes at 1950° F (1066° C). Silver additions were submicron silver powder. Atmosphere, air; sliding velocity, 450 feet per minute (137 m/min); load, 500 grams; rider, uncoated cast Inconel, 3/16-inch (0.476-cm) hemispherical radius; disk, Inconel 750 with 0.0015-inch (0.0038-cm) coating.

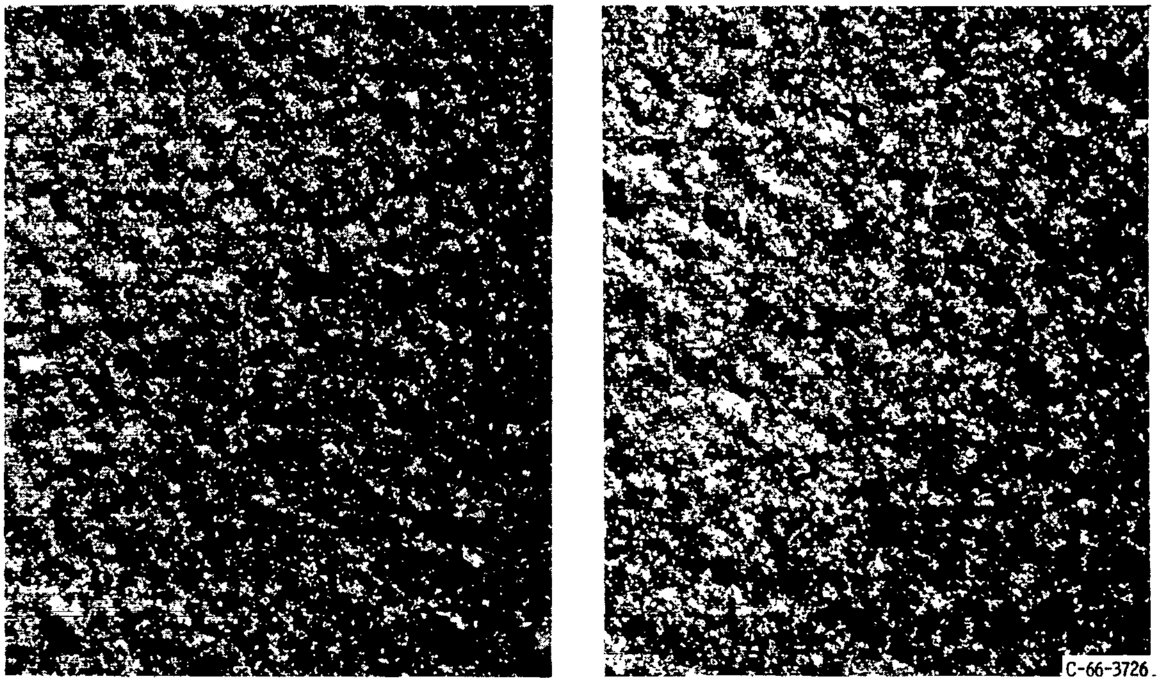
35-percent-silver coating were the lowest of this series, near or below 0.2 at all temperatures from 70° to 1400° F. The friction coefficient rose to nearly 0.3 as the temperature was brought back to 70° F (21° C). However, the silver addition still significantly lowered the friction coefficient of the fused-fluoride coating. An increase to 50-percent silver raised the friction coefficients slightly over those of the 35-percent-silver coating.

The only difficulty with additions of elemental silver to the fused-fluoride coating is that the silver in the coating tended to agglomerate during firing (fig. 7). Two fused-fluoride coatings, fired under the same conditions, with and without a 25 percent addition



(a) Silver, 25 weight percent; fused fluorides, 75 weight percent. Severe silver agglomeration.

(b) Fused fluorides (calcium and barium fluorides); no silver.



(c) Silver, 25 weight percent; fused fluorides, 75 weight percen Mild silver agglomeration.

(d) Silver (from silver chloride), 25 weight percent; fused fluorides, 75 weight percent. Mild silver agglomeration.

Figure 7. - Fused fluoride coatings with and without silver additions, showing degrees of agglomeration. All coatings fired at 1950° F (1066° C) for 15 minutes in hydrogen. X35.

of silver, are shown in figures 7(a) and (b). Balls of silver in the 25-percent-silver coating are plainly visible.

In an effort to lessen the agglomeration, the silver was added to the fused-fluoride coating in the form of the silver halides listed in table III. There were several reasons for the choice of these four materials. They melt and/or decompose below the standard firing temperature of the fused-fluoride coating. It is quite probable that in hydrogen all would be chemically reduced to silver; however, should any silver halide remain, it might act as a lubricant. The possiblities of AgCl, for example, as a lubricant have been investigated (ref. 10). Further, of the silver halides, only AgF is soluble in water; at 60° F (15.5° C) 182 grams of AgF will dissolve in 100 grams of water. Therefore, it is homogeneously dispersed throughout the CaF_2-BaF_2 mixture in a water-base slurry.

TABLE III. - SILVER HALIDES ADDED TO FUSED-FLUORIDE COATINGS

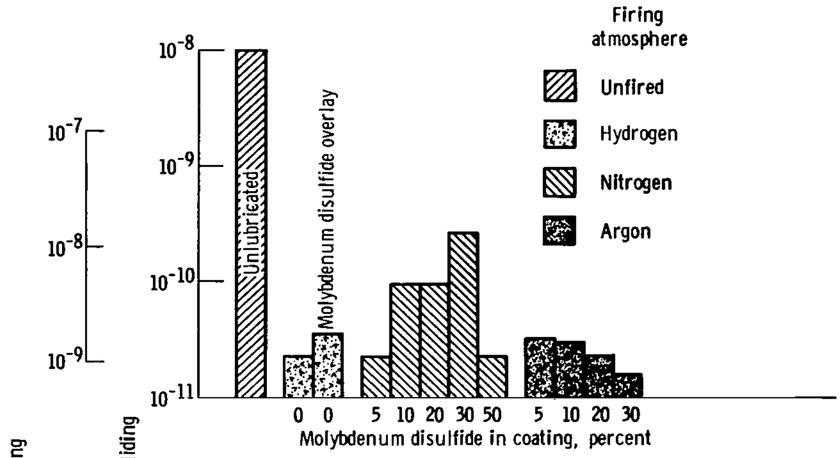
Halide	Melting point, °F (°C)	Boiling point, °F (°C)
Silver fluoride (AgF)	816 (435)	----------
Silver chloride (AgCl)	851 (455)	2822 (1550)
Silver bromide (AgBr)	815 (434)	(a)
Silver iodide (AgI)	(b)	----------

[a] Decomposes at 1292° F (700° C).
[b] Decomposes at 1026° F (552° C).

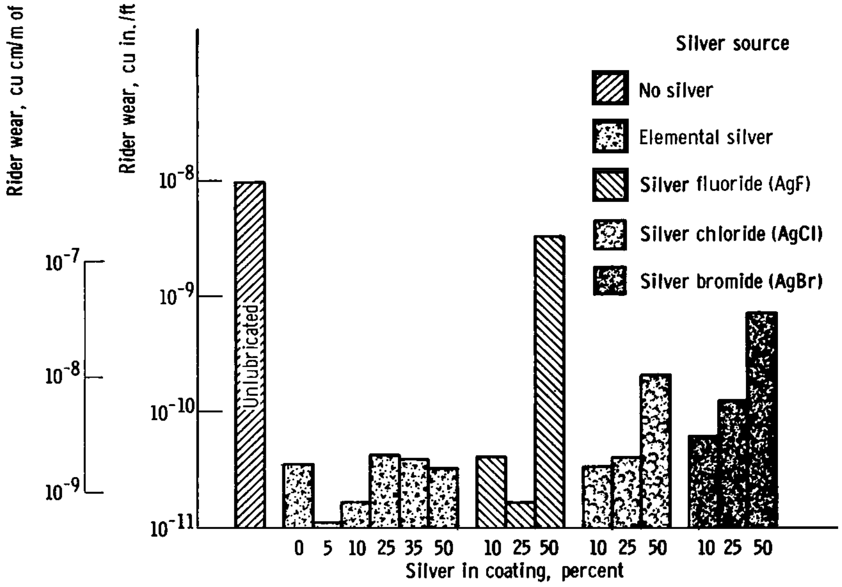
Silver - fused-fluoride coatings with a more uniform distribution of silver are shown in figures 7(c) and (d). However, while the silver in figure 7(d) was derived from AgCl, the silver in figure 7(c) was added as elemental silver powder as in figure 7(a). It cannot be said, therefore, that adding silver in the form of silver halides reduces agglomeration. Most silver - fused-fluoride coatings showed some degree of agglomeration. This agglomeration became more severe with small increases in firing time and temperature, and was not dependent on the original form of the silver.

The lubricating properties of most coatings containing silver were evaluated at 1000° F (538° C) for 30 minutes before temperature cycling. A summary of rider wear for all the various coating combinations is shown in figure 8, which includes data for the MoS_2 - fused-fluoride coatings for comparison. As previously noted, metal removal below 10^{-10} cubic inch per foot of sliding is considered a low rate of rider wear. Excessive rider wear during the initial runin eliminated from further consideration coatings containing 50 percent Ag derived from AgF or AgBr.

The remaining friction results are shown in figures 9 to 11. Data in figures 9 and 10 indicate that 25 weight percent silver, added to the fused fluorides in the form of AgF or AgCl, reduced the friction coefficients in the temperature range 70° to 900° F (21° to 482° C) to less than or about 0.3, values which are below those of the fused-fluoride coating without additions. Further, increasing the silver content to 50 weight percent silver did not lessen friction more than the 25-weight-percent addition (fig. 10). Wear also increased with the higher silver content. The friction coefficients of various silver -

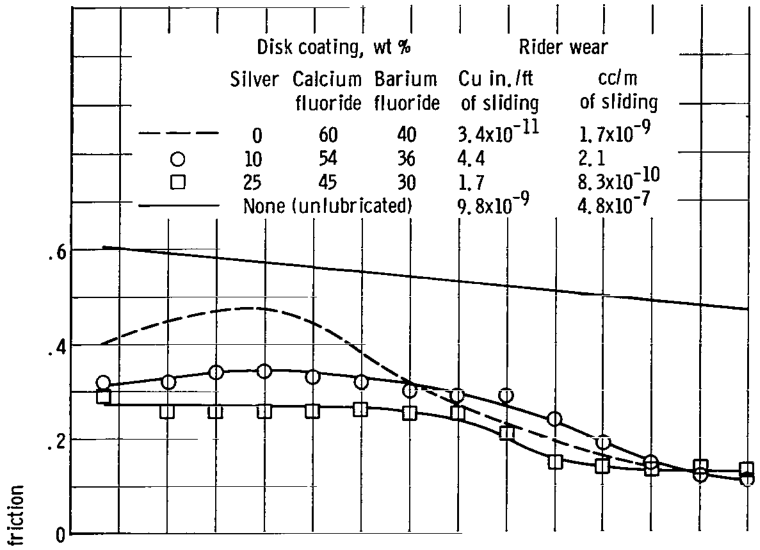


(a) Molybdenum disulfide - fused-fluoride coatings.

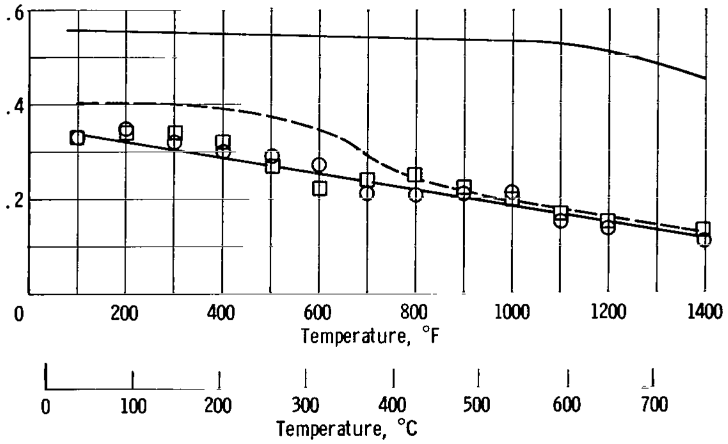


(b) Silver - fused-fluoride coatings. All coatings fired in hydrogen.

Figure 8. - Summary of rider wear rates for various molybdenum disulfide - and silver - fused-fluoride coating combinations.

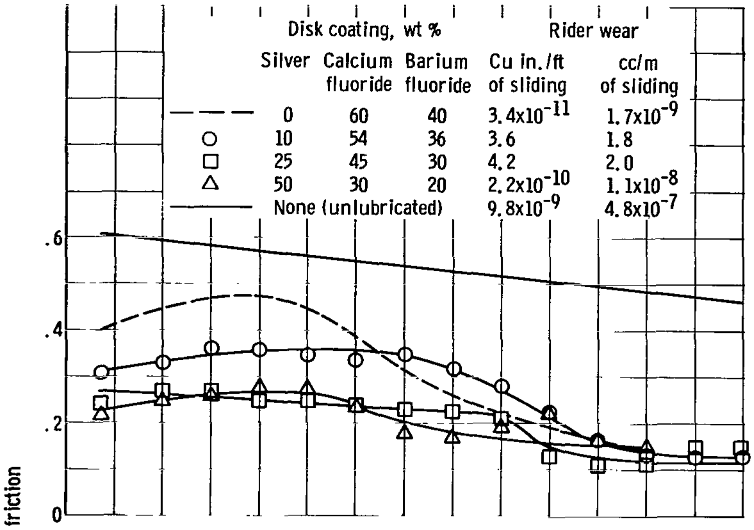


(a) Increasing temperature.

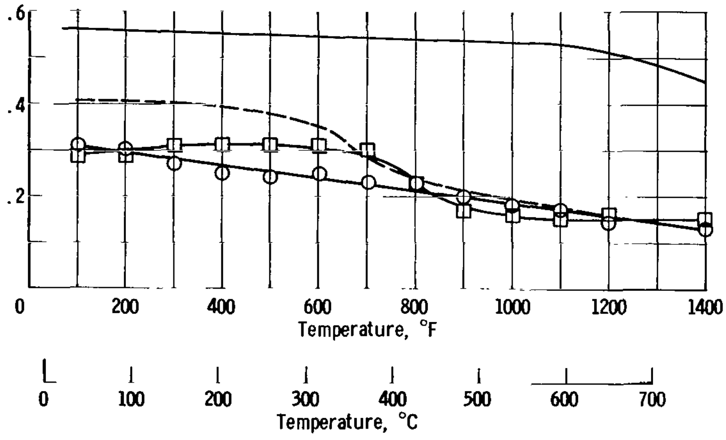


(b) Decreasing temperature.

Figure 9. - Friction coefficients of silver - fused-fluoride coating fired in hydrogen for 15 minutes at 1950° F (1066° C). Silver derived from reduction of silver fluoride during hydrogen firing. Atmosphere, air; sliding velocity, 450 feet per minute (137 m/min); load, 500 grams; rider, uncoated cast Inconel, 3/16-inch (0.476-cm) hemispherical radius; disk, Inconel 750 with 0.0015-inch (0.0038-cm) coating.

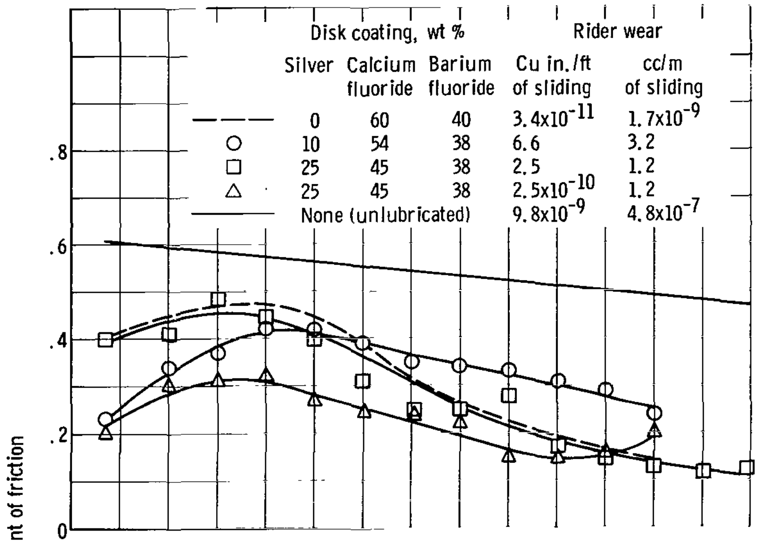


(a) Increasing temperature.

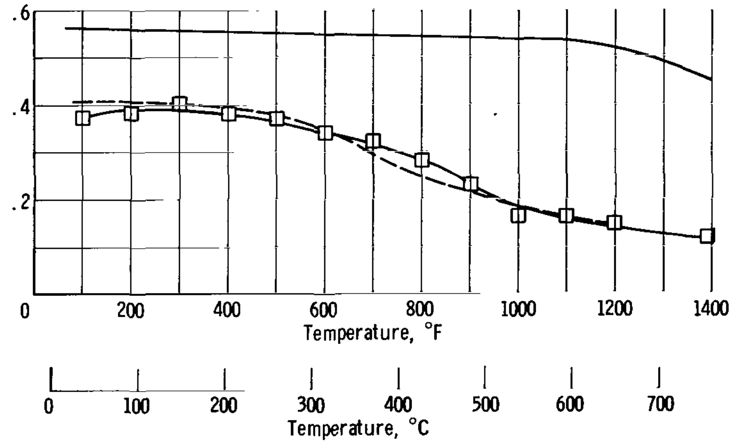


(b) Decreasing temperature.

Figure 10. - Friction coefficients of silver - fused-fluoride coatings fired in hydrogen for 15 minutes at 1950° F (1066° C). Silver derived from reduction of silver chloride during hydrogen firing. Atmosphere, air; sliding velocity, 450 feet per minute (137 m/min); load, 500 grams; rider, uncoated cast Inconel, 3/16-inch (0.476-cm) hemispherical radius; disk, Inconel 750 with 0.0015-inch (0.0038-cm) coating.

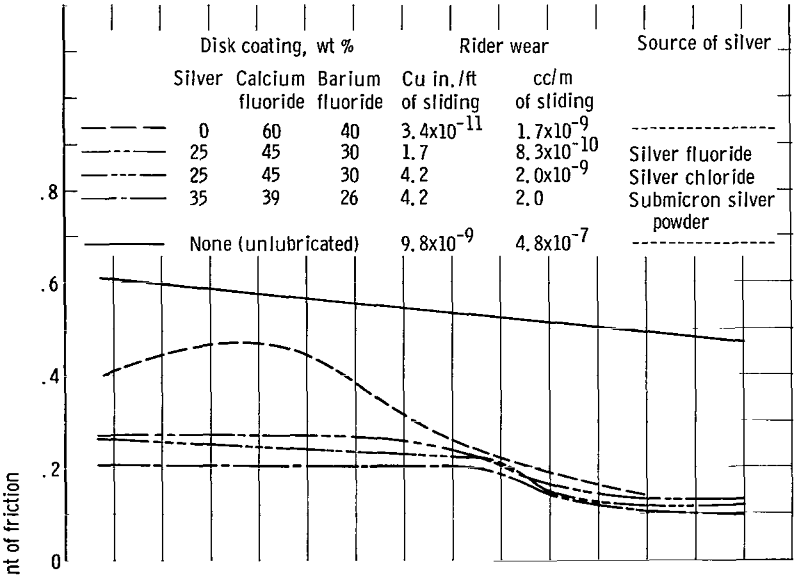


(a) Increasing temperature.

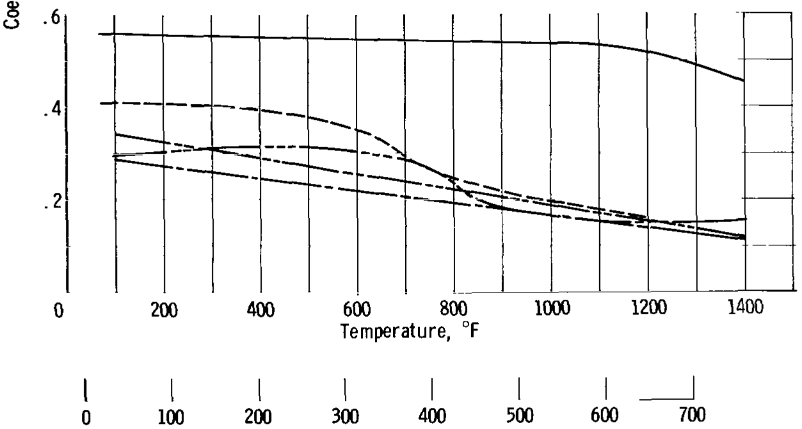


(b) Decreasing temperature.

Figure 11. - Friction coefficients of silver - fused-fluoride coatings fired in hydrogen for 15 minutes at 1950° F (1066° C). Silver derived from reduction of silver bromide during hydrogen firing. Atmosphere, air; sliding velocity, 450 feet per minute (137 m/min); load, 500 grams; rider, uncoated cast Inconel, 3/16-inch (0.476-cm) hemispherical radius; disk, Inconel 750 with 0.0015-inch (0.0038-cm) coating.



(a) Increasing temperature.



(b) Decreasing temperature.

Figure 12. - Summary of friction coefficients for silver - fused-fluoride coating combinations. Atmosphere, air; sliding velocity, 450 feet per minute (137 m/min); load, 500 grams; rider, uncoated cast Inconel, 3/16-inch (0.476-cm) hemispherical radius; disk, Inconel 750 with 0.0015-inch (0.0038-cm) coating.

fused-fluoride coatings with the silver derived from AgBr (fig. 11) were quite inconsistent. Two separate experiments on a 25-percent-silver - 75-percent - fused-fluoride coating, with the silver derived from AgBr, showed widely varying friction and wear data and no particular improvement in friction coefficient over the fused-fluoride coating without additions. Friction results with all silver - fused-fluoride coatings, except where the silver was derived from AgBr, were reproducible.

For clarification, a summary of the best silver - fused-fluoride coating results is shown in figure 12. It must be concluded from these curves that at temperatures below 900° F (482° C) elemental silver powder added to the fused-fluoride coatings is superior to silver additions derived from AgF or AgCl; elemental silver powder is also easier to mix and apply.

SUMMARY OF RESULTS

An experimental program was conducted to reduce the low-temperature friction of fused-fluoride coatings by the addition of solid lubricants, such as molybdenum disulfide (MoS_2) and silver, which are effective at low temperatures. The results of these experiments are summarized as follows:

1. Elemental silver powder (35 weight percent) added to the fluoride slurry before spraying gave best overall results, the friction coefficient being 0.2 or less as the temperature was increased from 70° to 1400° F (21° to 760° C) and below 0.3 upon return to room temperature.

2. Silver (25 weight percent) added to the coating slurry in the form of silver fluoride or silver chloride before spraying also reduced the friction coefficient somewhat (to less than 0.3) at temperatures below 900° F (482° C).

3. When temperature cycling did not occur, spraying or rubbing MoS_2 onto a fluoride-coated specimen greatly reduced the friction coefficient at low temperatures. The beneficial effects of the overlay did not last above 400° to 600° F (204° to 316° C), and consequently high friction occurred at temperatures of 400° to 900° F (204° to 482° C). However, incorporating MoS_2 powder (30 to 50 weight percent) into the fluoride coating before spraying kept the friction coefficient near or below 0.2 to 1500° F (8.16° C).

4. While reducing the low-temperature friction coefficient, silver and MoS_2 additions did not adversely affect wear of the specimens or life of the coatings under the conditions of these experiments.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 26, 1966,
720-03-01-01-22.

REFERENCES

1. Bisson, Edmond E.; and Anderson, William J.: Advanced Bearing Technology. NASA SP-38, 1964.

2. Braithwaite, Edward R.: Solid Lubricants and Surfaces. MacMillan Co., 1964.

3. Johnson, R. L.; and Sliney, H. E.: Ceramic Surface Films for Lubrication at Temperatures to 2000° F. Am. Cer. Soc. Bull., vol. 41, no. 8, Aug. 1962, pp. 504-508.

4. Godfrey, Douglas; and Nelson, Erva C.: Oxidation Characteristics of Molybdenum Disulfide and Effect of Such Oxidation on Its Role as a Solid-Film Lubricant. NACA TN 1882, 1949.

5. Sliney, Harold E.; Strom, Thomas N.; and Allen, Gordon P.: Fused Fluoride Coatings as Solid Lubricants in Liquid Sodium, Hydrogen, Vacuum, and Air. NASA TN D-2348, 1964.

6. Peterson, M. B.; Murray, S. F.; and Florek, J. J.: Consideration of Lubricants for Temperatures above 1000° F. ASLE Trans., vol. 2, no. 2, 1960, pp. 225-234.

7. Buckley, Donald H.; Swikert, Max; and Johnson, Robert L.: Friction, Wear, and Evaporation Rates of Various Materials in Vacuum to 10^{-7} mm Hg. ASLE Trans., vol. 5, no. 1, Apr. 1962, pp. 8-23.

8. Jaffe, L. D.; and Rittenhouse, J. B.: Behavior of Materials in Space Environments. Rep. No. TR-32-150, California Inst. of Tech., Jet Propulsion Lab., Nov. 1, 1961.

9. Craig, W. D., Jr.: Friction Variation of PTFE and MoS_2 During Thermal Vacuum Exposure. Lubr. Eng., vol. 20, no. 7, July 1964, pp. 273-277.

10. Hopkins, Vern; and Gaddis, D. H.: Research on Bearing Lubricants for Use in High Vacuum. Midwest Research Inst. (NASA CR-58204), Aug. 30, 1963.

NASA-Langley, 1967

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