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The Role of Silver in Self-Lubricating Coatings for Use at Extreme Temperatures

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THE ROLE OF SILVER IN SELF-LUBRICATING COATINGS

FOR USE AT EXTREME TEMPERATURES

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SUMMARY

The advantages and disadvantages of elemental silver as a tribological material are discussed. It is demonstrated that the relatively high melting point of 961 °C, softness, marked plasticity, and thermochemical stability of silver combine to make this metal useful in thin film solid lubricant coatings over a wide temperature range. Disadvantages of silver during sliding, except when used as a thin film, are shown to be gross ploughing due to plastic deformation under load with associated high friction and excessive transfer to counterface surfaces. This transfer generates an irregular surface topography with consequent undesirable changes in bearing clearance distribution. This paper describes research to overcome these disadvantages of elemental silver. A comparison is made of the tribological behavior of pure silver with that of silver formulated with other metals and high-temperature solid lubricants. The composite materials are prepared by co-depositing the powdered components with an airbrush followed by furnace heat treatment or by plasma-spraying. Composite coatings were formulated which are shown to be self-lubricating over repeated, temperature cycles from low temperature to about 900 °C.

INTRODUCTION

There is a great need in current technology for solid lubricated systems that will perform satisfactorily over a wide range of temperatures. For example, the achievement of major advances in high temperature lubrication are essential for the development of more fuel efficient engines such as the adiabatic diesel and advanced turbomachinery (ref. 1). Other examples are the advanced Stirling engine and numerous aerospace mechanisms. Maximum temperatures of 600 to 1100 °C are anticipated for critical sliding contacts in these applications (refs. 2 to 5).

Solid lubricant candidate materials that are thermally and chemically stable (nonreactive) at the temperatures quoted include certain soft oxides, vitreous glazes, fluorides of alkaline earth metals, and soft noble metals such as gold and silver. Gold and silver are of course costly, but the amount used is often small enough to make the use of these metals cost-effective in specialty lubricants. Silver in particular, is well-known to be an important tribological material for a wide range of applications. For example, it has been used for many years as an electroplated coating on the retainer of rolling element bearings; steel-backed electroplated silver bearings have been used as hydrodynamically-lubricated crankshaft and main bearings in high performance reciprocating engines for aircraft.

Silver, when present as a thin film between hard sliding surfaces, can function as a solid lubricant film because of its very marked ductility. This extreme ductility allows the silver film to plastically shear between the sliding surfaces and thereby provide a lubricating function. A problem with thin electroplated silver is the difficulty in maintaining adequate bonding to the substrate at elevated temperatures in an oxidizing environment (ref. 6). Also, if the silver is too thick, it will deform excessively under the normal load thus destroying dimensional accuracy and adding a significant ploughing force to the total frictional (tangential) force. Nevertheless, it will be demonstrated in this paper that silver can be used as a lubricating component of thick coatings from low temperatures to near its melting point of 961 °C if it is either: (1) heterogeneously alloyed with harder metals; or (2) incorporated as one component of composite, self-lubricating materials. It is possible to use silver over this large temperature range not only because of its desirable shear properties, but also because of its thermochemical stability (particularly oxidation resistance) at high temperatures.

In this paper, research is described in which silver was added to composite coatings containing high temperature solid lubricants in order to improve low temperature tribological properties. Friction and wear were determined in a pin on disk apparatus. The pin and disk materials were nickel base superalloys and titanium alloys. The disks were coated with the various experimental coatings that are the subject of this paper. Some of the data were previously presented in NASA publications, but not in the open literature. New data are presented for a recently developed chromium carbide based composite coating.

MATERIALS AND COATING PROCEDURES

Descriptive data for coating materials and coating compositions are given in tables I to IV.

Furnace-Fused Coatings

Reagent grade fluorides of greater than 99.9 percent purity and technical grades of silver and MoS_2 of 99 percent purity were used in preparing the furnace-fused coatings. The substrate material was a nickel base superalloy Inconel 750 precipitation hardened to R_C 35. The disks were solvent cleaned and sandblasted prior to applying the coatings. The fluoride and silver powders were suspended in a cellulose nitrate lacquer; then sprayed onto the disk surface with a paint sprayer. The specimens were baked at 90 °C, then heated in a hydrogen atmosphere at 1060 °C for 15 min (this temperature is 100 °C above the melting point of silver and 38 °C above the melting point of the fluoride eutectic.) The specimens were then moved to a cooling chamber and finally a nitrogen purge chamber before removal from the furnace. This procedure produced fusion-bonded coatings about 0.0038 cm thick in which microscopic silver globules were distributed throughout a fluoride eutectic matrix.

Plasma-Sprayed Coatings

Whenever possible, commercially available powders, which are specifically supplied for plasma spraying, were blended to give the compositions shown in table III. The Ti-6Al-4V or Inconel 750 substrates were first sandblasted, then plasma sprayed with a nichrome bond coat about 0.007 cm thick, and finally with the lubricant coating to a total coating thickness of about 0.050 cm. The coating was then surface ground to a total coating thickness of 0.025 cm. It is necessary to grind in a manner that does not cause surface smearing. This can be done by diamond grinding or with grinding wheels of cubic boron nitride. Final grinding passes should be very light cuts of no more than about 0.0005 cm depth.

Tribometer

Friction and wear experiments were run on a standard pin-on-disk apparatus in which a pin with a hemispherical radius of 0.48 cm is placed in sliding contact with the flat, coated surface of a rotating disk. Normal load is applied by dead weights and friction force is continuously measured with a temperature-compensated strain gauge bridge circuit.

RESULTS AND DISCUSSION

It has been known for some time that thermally-fused (or fusion-bonded) coatings of chemically stable fluoride such as calcium fluoride (CaF_2) and barium fluoride (BaF_2) provide effective lubrication of nickel-base superalloys from about 500 to 900 °C (ref. 7). Unfortunately these fluorides are not effective lubricants at lower temperatures. Metal-fluoride composites prepared by powder metallurgy techniques (refs. 8 and 9) and by plasma spraying (ref. 10) have similar lubrication/temperature characteristics. Therefore, formulation studies were conducted at NASA Lewis Research Center with the objective of improving low temperature lubrication while retaining the good high temperature lubricating properties of the fluorides (refs. 11 and 12). Current research has the further objective of achieving harder, very wear-resistant coatings with wide temperature spectrum capabilities. Results are summarized below for the following three approaches: (1) The influence of MoS_2 and of silver on lubrication with fused fluoride coatings; (2) the effect of silver on lubrication with fluoride-superalloy, plasma-sprayed coatings; (3) the lubricating characteristics of chromium carbide coatings containing BaF_2-CaF_2 eutectic and silver.

MoS_2 or Silver in Fused Fluoride Coatings

A research program was conducted with the objective of reducing the low-temperature friction of fused fluoride coatings by the addition of MoS_2 or silver, both of which are known to form effective solid lubricant films at moderate temperatures. Since MoS_2 oxidizes rapidly above about 350 °C, its use in this study was to determine whether it could be used in conjunction with the fluorides for <u>one-time applications</u> where lubrication is required over only one temperature cycle. Silver was studied as an additive to fluoride coatings expected to be used <u>repeatedly</u> over a wide temperature range.

<u>Effect of MoS_2</u> - MoS_2 was evaluated both as an overlay on, and as an addition to, the fluoride coating composition. Figure 1 shows the effect of MoS_2 on the friction-temperature characteristics of BaF_2-CaF_2 coatings. In one case, MoS_2 is employed as an overlay (top coat) on the fluoride coating; in the other it is an additive within the fluoride coating. Control experiments were performed with pre-oxidized but otherwise unlubricated

Inconel 750 pins and disks and with BaF_2-CaF_2 coated disks without MoS_2. The unlubricated specimens gave high wear rates and friction coefficients of 0.6 at room temperature gradually decreasing to 0.45 at 800 °C. Wear was much less at all temperatures with the fluoride coatings, but friction was characteristically high up to about 300 °C, then gradually decreased to about 0.2 at 500 °C and remained at that value to 800 °C. With the MoS_2 overlay, there was a predictable, dramatic reduction in friction coefficients below 300 °C. Upon oxidation of MoS_2 above 300 °C, friction was controlled by the fluorides and was therefore the same as observed for BaF_2-CaF_2 coatings without MoS_2. Similar behavior was observed when MoS_2 was incorporated into the coating composition, friction coefficients never exceeded 0.2 over the entire temperature range, but again, the MoS_2 oxidized and the beneficial effect was only obtainable over one temperature excursion from room temperature to 800 °C.

Effect of silver - Metallic silver powder was added to the fluoride coating composition in amounts up to 50 wt %. The data in figure 2 show that the friction coefficients at room temperature were at a minimum for 35 wt % of silver. At this silver concentration, friction coefficients were 0.2 or less over the entire temperature range studied. Figure 3 compares the friction-temperature characteristics of fluoride coatings with and without a 35 percent silver addition.

Silver in Plasma-Sprayed Fluoride-Metal Self-Lubricating Coatings

The thermally-fused coatings just discussed require heat treatment (firing) at high temperatures above the melting point of at least some of the coating components in order to achieve fusion bonding. (The procedure is very similar to that employed in applying porcelain enamels to metals or glazes to ceramics.) This heat treatment is not acceptable for some alloys including those of titanium. An alternative method is plasma spraying which needs only minimally to heat the substrate surface. Plasma spraying is also more convenient and can be used to deposit combinations of materials which are not amenable to fusion bonding.

It was reported in (ref. 12) that plasma-sprayed composite coatings of nichrome, CaF_2, silver, and glass lubricated nickel-base superalloys from cryogenic temperatures to 900 °C. Figure 4 is a photomicrograph of the composite coating. Figure 5 shows the friction-temperature characteristics of such a coating compared to a similar coating without silver. It is clear that the silver addition reduces friction at low temperatures without a significant adverse effect on lubrication at high temperature.

In a program to solve a specific lubrication problem with titanium alloys at 430 °C, plasma-spray coatings of increasing complexity from a one component (pure silver) to the four component composition reported in (ref. 12) were investigated (ref. 13). All multicomponent coatings contained a one-to-one weight ratio of silver and nichrome (1:1 Ag-NiCr). CaF_2 was the added material in the three component coatings, and a sodium-free glass was the fourth component. The purpose of the nichrome was to provide a harder, less deformable coating material than pure silver. CaF_2 was used because of its well-known high temperature lubricating ability. Glass was expected to provide a glazing tendency on the sliding surface as well as providing a degree of oxidation-protection to the nichrome. The identification numbers and compositions of the coating are given in table III. The coatings were applied to a titanium alloy (Ti-6 Al-4 V). A bond coat of nichrome about 0.07 mm thick was plasma-sprayed onto the disks prior to application of the lubricant coatings. The nichrome bonded well to the titanium alloy and the top coats all bonded well to the intermediate nichrome coating. An excess of top coat was applied, then ground back to give a total coating thickness of about 0.25 mm (0.010 in) except where otherwise specified.

The friction characteristics of the coatings in sliding contact with titanium alloy pins over test durations of up to 20 hr are shown in figure 6. Surface profiles of the wear tracks at the completion of the tests are shown in figure 7. Pin and coating wear rates are given in figure 8. Pin wear rates from (ref. 14) against the uncoated Ti-6 Al-4 V and against a titanium diboride hard coat on this alloy are also given for comparison.

The lowest sliding friction early in the tests was observed with pure silver. Friction coefficients were less than 0.1 for 5 hr then increased abruptly. No wear occurred on the hemispherical pin, but silver immediately transferred to it and the sliding combination was essentially silver on silver. 9Surface profilometry (fig. 7(a)) showed that the coating was very severely plastically deformed, but the pin had not penetrated through the silver to the substrate. This excessive plastic deformation and the heavy silver transfer make these relatively thick silver coatings unacceptable for the type of sliding contact employed in these tests. The effect of coating thickness will be described later.

The addition of nichrome produced coatings with a low initial friction coefficient of 0.1; however, the friction coefficient gradually and continuously increased during the test to 0.33 after 9 hr. The coating wear track profile (fig. 7(b)) shows considerable coating wear but much less indication of gross plastic deformation than pure silver. Also, the heavy transfer, which is characteristic of pure silver, did not occur. Pin and coating wear rates are given in figure 8.

Calcium fluoride addition (coating PS106) resulted in significant reductions in pin and coating wear rates. Initial friction coefficients were higher than with silver and one-to-one silver-nichrome, but were very steady and constant at a value of 0.2 for the entire 9-hr test duration.

The four-component coating (PS101) had about the same friction coefficient as PS106 (0.2). A small increase in pin wear rate occurred compared to that observed with PS106, but the coating wear rate was so low that the test was continued for a duration of 20 hr with very little penetration of the coating (fig. 7(d)).

These results indicate that plasma-sprayed silver is very effective as a dry lubricant coating but must be combined with other components to reduce plastic deformation and excessive silver transfer to the counterface material. Calcium fluoride did not reduce the friction coefficient for short test durations, but resulted in a coating with a very stable, moderate friction coefficient of about 0.2 over a long duration of sliding. It also had a beneficial effect in reducing pin and coating wear. The glass addition caused a small increase in pin wear but a substantial reduction in coating wear.

Effect of thickness of silver coatings - The main problems with the relatively thick (0.17 mm) silver coatings were severe plastic deformation of the silver and heavy adhesive transfer of silver to the titanium alloy pin, therefore, thinner silver coatings were also evaluated. It was expected that plastic deformation would necessarily be reduced for the thinner coatings for which the harder substrate would provide a more effective support.

The effects of silver coating thickness on friction and wear are shown in figures 9 to 11. The friction coefficients for 0.07 mm coating was about 0.1 or nearly the same as that of the 0.17 mm coating during the first 5 hr but remained at this low value for the total duration of 7 hr while the friction of the thicker coating took a sharp upturn after 5 hr. Surface profiles of the wear tracks on the coating (fig. 10(b)) showed much reduced plastic deformation of the silver. The depth of the track was about 0.03 mm or about one-half the coating thickness; therefore, no contact occurred between the titanium alloy pin and the coating substrate. Silver transfer to the pin was reduced and some pin wear occurred (fig. 11).

Finally a 0.02-mm coating was evaluated. Friction coefficients were about 50 percent higher (0.15) than in the case of the thicker coatings, but remained constant for 8 hr except for a time about halfway through the test when sliding was a little rough and the friction coefficient briefly peaked at 0.2. This was probably caused by a temporary penetration of the coating over a small area of the wear track which subsequently "healed" by plastic flow of silver over the failed area and the pick up of additional silver from the sides of the wear track as wear of the hemispherically tipped pin progressed. The wear data of figure 11 and the surface profiles of figure 10(c) show that both pin wear and coating wear rates were very low. The silver flowed readily under the shear stress of the sliding contact. Figure 10(d) shows that, in some of the tests, the silver was displaced and redeposited in other areas as particles or patches which were up to 0.03 mm thick. This flow characteristic can be advantageous as previously described for "healing" small areas where the coating is worn away. However, redistribution of the silver can also be a problem if it results in undesirable changes in bearing clearances.

In general, the results indicate the following: (1) Low friction coefficients (0.05 to 0.15) can be achieved with plasma-sprayed silver coatings of 0.02 to 0.17 mm thickness. However, excessive plastic deformation of the coatings occur in all but the 0.02 mm thick coatings; (2) among thick plasma-sprayed coatings (0.17 mm), the best results were obtained with a coating weight percent composition of 30Ag-30NiCr-25CaF_2-15 glass (PS101).

Chromium Carbide Coatings Containing Silver and a BaF_2/CaF_2 Eutectic

In a further attempt to achieve coatings with lower wear rates over a wide temperature range, a bonded chromium carbide wear control coating composition was modified by the addition of silver and other materials with the objective of achieving lower friction while retaining the wear resistance of the unmodified chromium carbide coating composition. Chromium carbide was chosen for this study because of its excellent combination of wear resistance and superior oxidation resistance; chromium carbide is more oxidatively stable; than for example, the well-known wear resistant hard materials such as tungsten carbide and titanium carbide or nitride (ref. 15).

Table V gives some preliminary friction and wear data for a new coating composition consistency of 80 wt % bonded chromium carbide, 10 wt % silver, and 10 wt % of a CaF_2/BaF_2 eutectic. This composition is designated as PS200; composition details are given in table III.

The data show that friction coefficients (typically 0.35) are higher than those shown for PS101 on figure 5. However friction coefficients were lower than those of the unformulated carbide coating, and coating wear was, in most cases, too low to be measured by profilometry tracer across the wear tracks. Pin wear rates were about the same as previously reported for sliding against PS101. It should be noted; however, that in these preliminary experiments, test duration was only 20 min. Therefore the wear rates shown are probably run-in wear rates which are generally higher than steady-state wear rates. These data, although preliminary, indicate that the new chromium carbide based coating formulations are promising candidates for self-lubricating bearing application where a long wear life over a wide temperature spectrum is required.

SUMMATION

This paper summarizes research at NASA Lewis Research Center on the influence of silver on various types of self-lubricating materials. Some conclusions that may be drawn from the data presented are:

1. Silver can be an effective lubricant additive in composite materials for use from low temperatures to 900 °C in an oxidizing atmosphere (air).

2. The primary benefit achieved with silver addition was to improve low and intermediate temperature lubricating properties without adversely affecting high temperature lubrication by other components of the composite coatings. Silver is chemically stable at the higher temperatures, thus allowing the coatings to be used over repeated temperature cycles.

3. A new plasma-sprayed composite coating composition consisting of bonded chromium carbide formulated with silver and CaF_2/BaF_2 eutectic was exceptionally wear resistant up to 900 °C and had better frictional characteristics than the unmodified carbide coatings. This composition, designated as PS200, appears to be a promising candidate for long-duration, high-temperature applications for which a moderately high friction coefficient of about 0.3 is acceptable.

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TABLE I. - MATERIALS USED IN PREPARATION OF FURNACE-FIRED COATINGS

Powder material	Range of particle sizes, μm	Grade	M.P. °C
Silver (Ag)	Submicron	Technical	961
CaF_2	5 to 20	Reagent	1410
BaF_2	5 to 20	Reagent	1280
Eutectic ($62BaF_2$-$38CaF_2$)	5 to 20	Reagent	1022
Molybdenum Disulfide, MoS_2	50 to 75	Technical 99 percent	Decomp. Below M.P.

TABLE II. - MATERIALS USED IN PREPARATION OF PLASMA-SPRAYED COATINGS

Powder material	Range of particle sizes, μm	Source
Silver (Ag)	50 to 150	Commercial, plasma spray powder
Nichrome (NiCr) 80 Ni-20 Cr	50 to 150	Commercial, plasma spray powder
Calcium fluoride (CaF_2)	5 to 20	Reagent grade chemical
Glass (58 SiO_2-21.2 BaO 7.8 CaO-13.0 K_2O)	50 to 150	Laboratory preparation from reagent grade chemicals
Chromium Carbide Nickel Aluminum Blend	10 to 70	Commercial plasma Spray powder

TABLE III. - COMPOSITIONS OF PLASMA-SPRAYED COATINGS

Identification number	Composition, wt %
PS109	100 Ag
PS108	50 Ag-50 NiCr
PS106	35 Ag-35 NiCr-30 CaF_2
PS101	30 Ag-30 NiCr-25 CaF_2-15 glass
PS100	67 NiCr-16.5 CaF_2-16.5 glass
PS200	80 NiAl bonded chromium carbide-10 Ag-10 BaF_2/CaF_2 eutectic

TABLE IV. - BULK PROPERTIES OF SOME HARD COAT MATERIALS[a]

Material	Microhardness, kg/mm^2	Oxidation temperature,[b] °C
B_4C	4200	1090
TiC	3200	540
SiC	2900	1650
Cr_3C4	2650	1370
WC	2050	540
Si_3N4	2000	1400
TiN	1950	540
Cr_2O3	[c]1800	----

[a]Data from: Engineering Properties of Ceramic Materials, Battelle Memorial Institute. Published by American Ceramic Society, Columbus, Ohio, 1966.
[b]Temperature for appreciable detrimental oxidation (passivating oxide films form at lower temperatures).
[c]Estimated conversion from published Moh hardness of 9.

TABLE V. - SUMMARY OF PRELIMINARY FRICTION AND WEAR DATA FOR PS200 (80 PERCENT BONDED CHROMIUM CARBIDE-10 PERCENT Ag-10 PERCENT BaF_2/CaF_2 EUTECTIC)

[0.8 m/s (300 rpm, 5 cm diam track), 0.5 kg, 20 min test duration.]

Materials		Temperature, °C	Wear factors, K (run-in wear), cm_3/cm • kg		Typical friction coefficients
Pin	Disk		Pin	Coating	
Inc 750	PS200	75	$2x10^{-9}$	Nondetectable	0.40±0.05
		760	$3.5x10^{-9}$	Nondetectable	0.35±0.05
		900	$1.5x10^{-9}$	5.1x10-9	0.35±0.05
αSiC	PS200	25	$1.2x10^{-11}$	Nondetectable	0.40±0.05
		760	$1.0x10^{-10}$		0.26±0.05
		900	$6.0x10^{-11}$		0.35±0.05
HS6B	Unformulated Bonded Chrome Carbide	25	$2.9x10^{-9}$		0.55±0.05
		530	$7.5x10^{-10}$		0.45±0.05

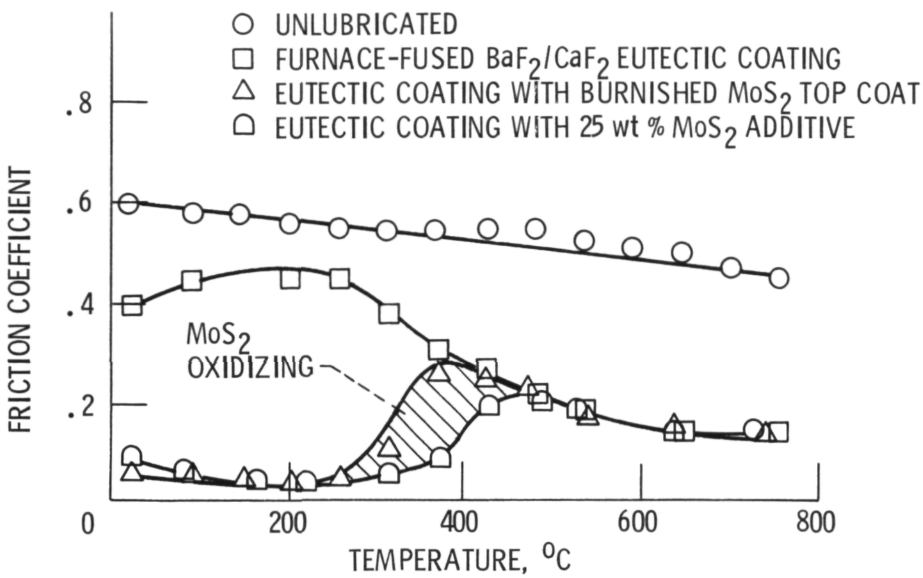

Figure 1. - Effect of MoS_2 as a burnished top coat and as an additive to BaF_2/CaF_2 eutectic furnace-fused coatings. Dry air atmosphere, 2.3 m/s, 500-gram load cast Inconel rider, Inconel 750 disks with 0.004 cm coatings.

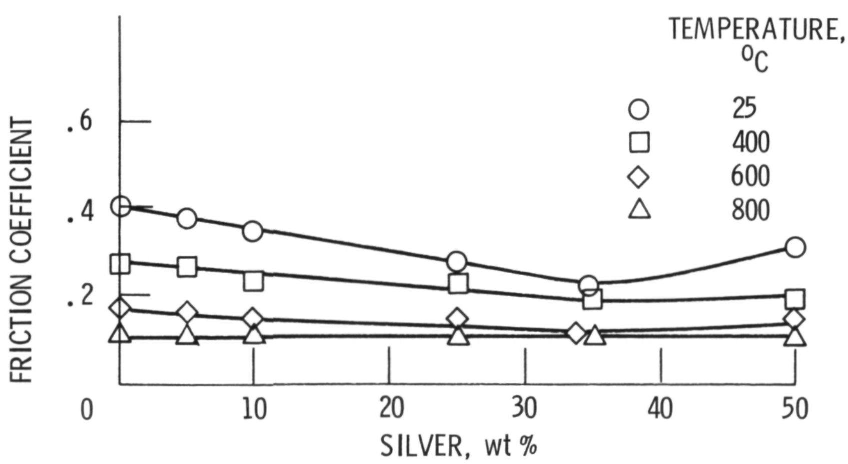

Figure 2. - Optimization of silver content in furnace-fused Baf_2/CaF_2 eutectic coatings.

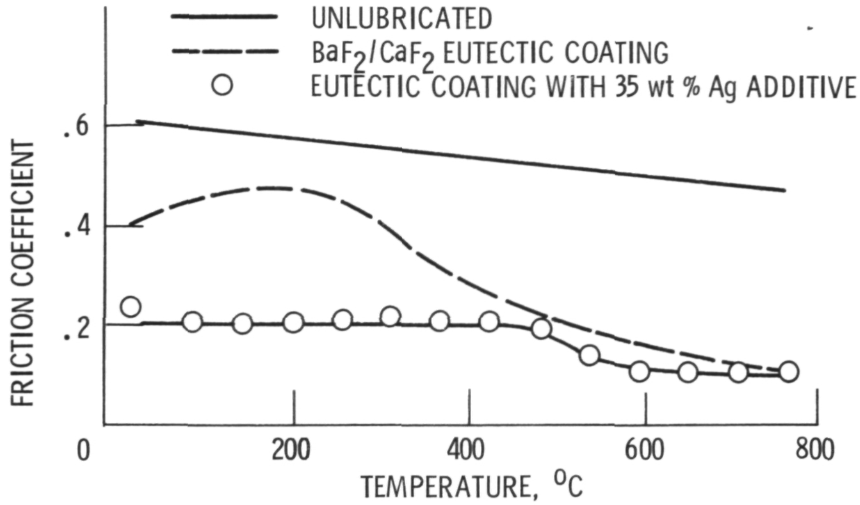

Figure 3. - Effect of silver addition to BaF_2/CaF_2 furnace-fused coating.

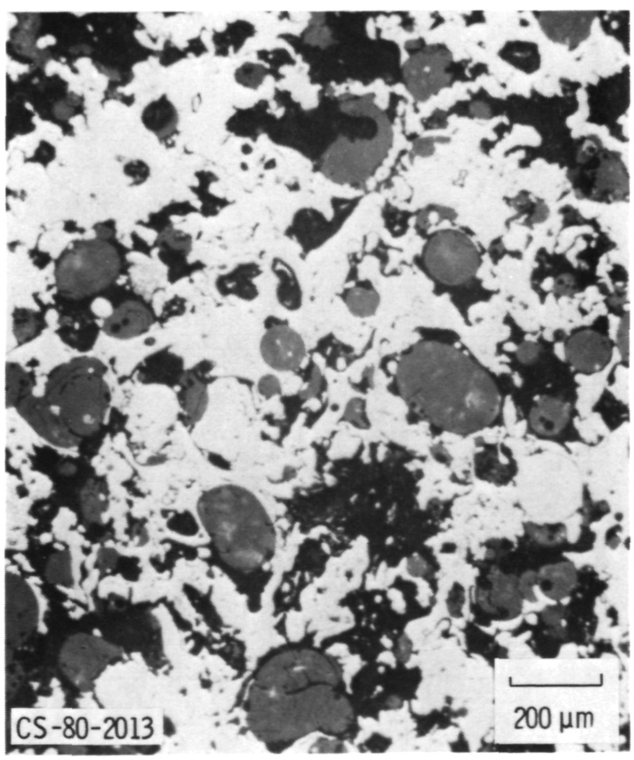

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Figure 4. - Microstructure of plasma sprayed composite.

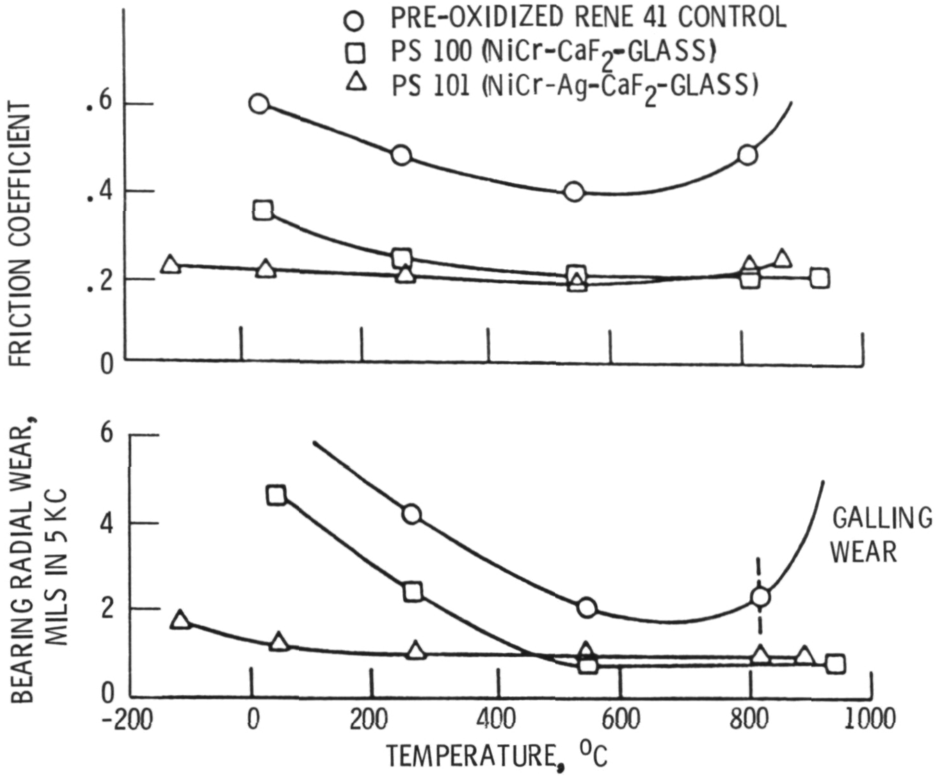

Figure 5. - Effect of silver addition on the friction and wear of plasma-sprayed, nichrome-matrix coatings (from Ref. 12). Lubricating plain spherical bearings at a radial load of 34 MPa. Bearing material, precipitation hardened nickel-chromium alloy.

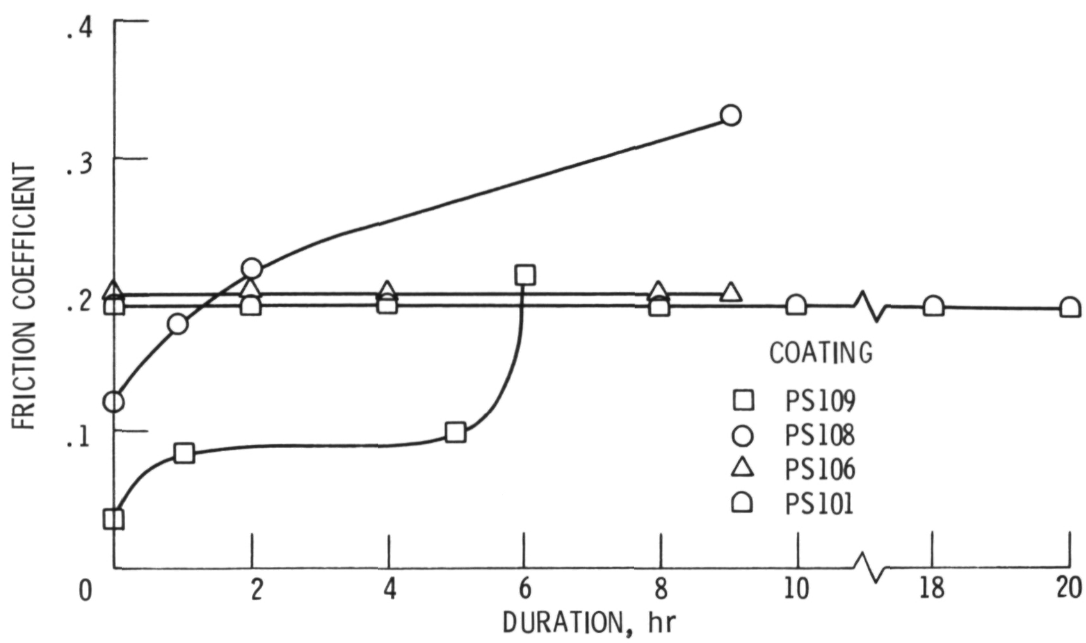

Figure 6. - Friction characteristics in air of 0.17-millimeter-thick plasma-sprayed composite coatings containing silver. Ti-5Al-2.5Sn pin material; 430 °C; 250-gram load; 13-centimeter-per-second sliding velocity at 50 rpm.

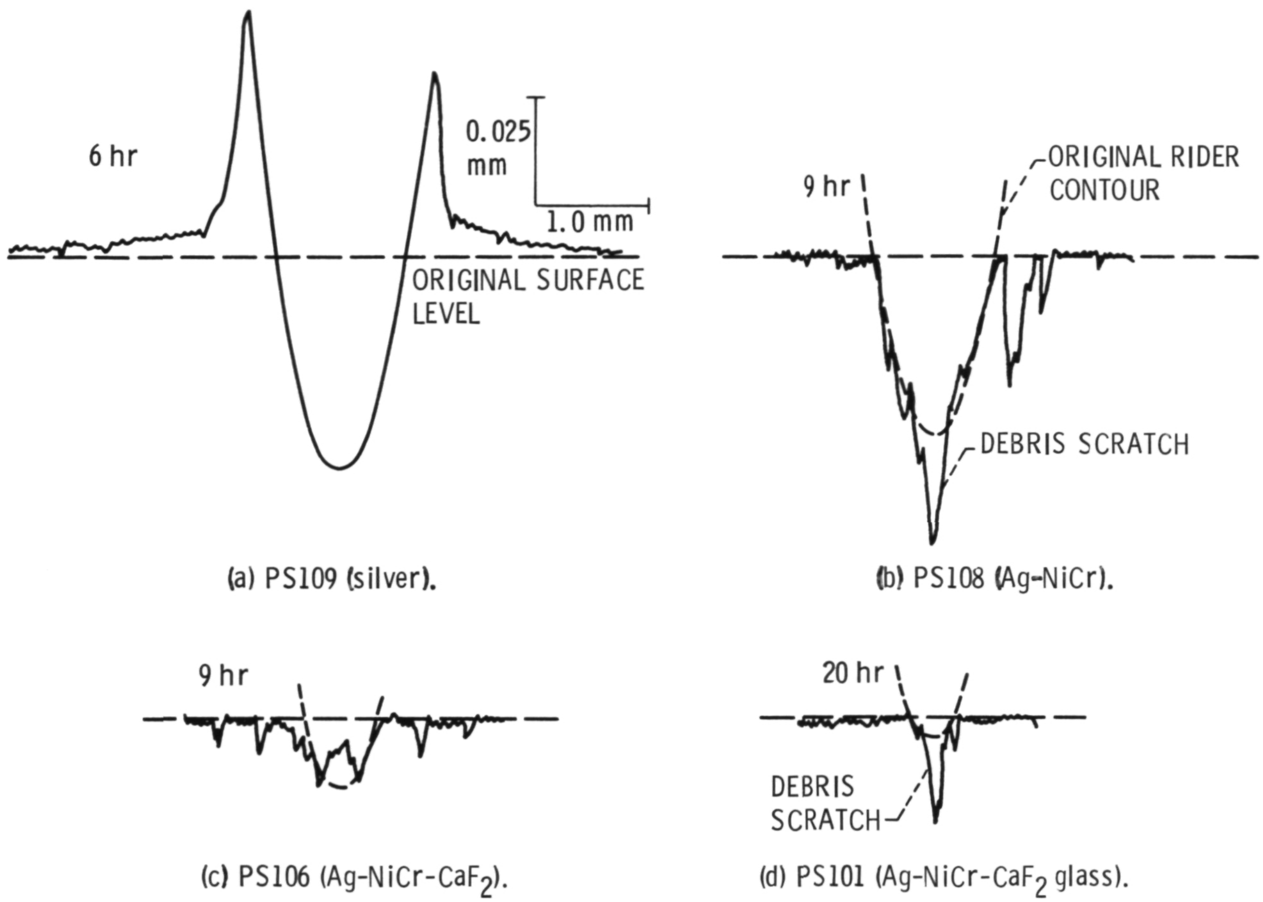

Figure 7. - Wear track cross sections showing wear and deformation of 0.17-millimeter-thick plasma-sprayed composite coatings containing silver.

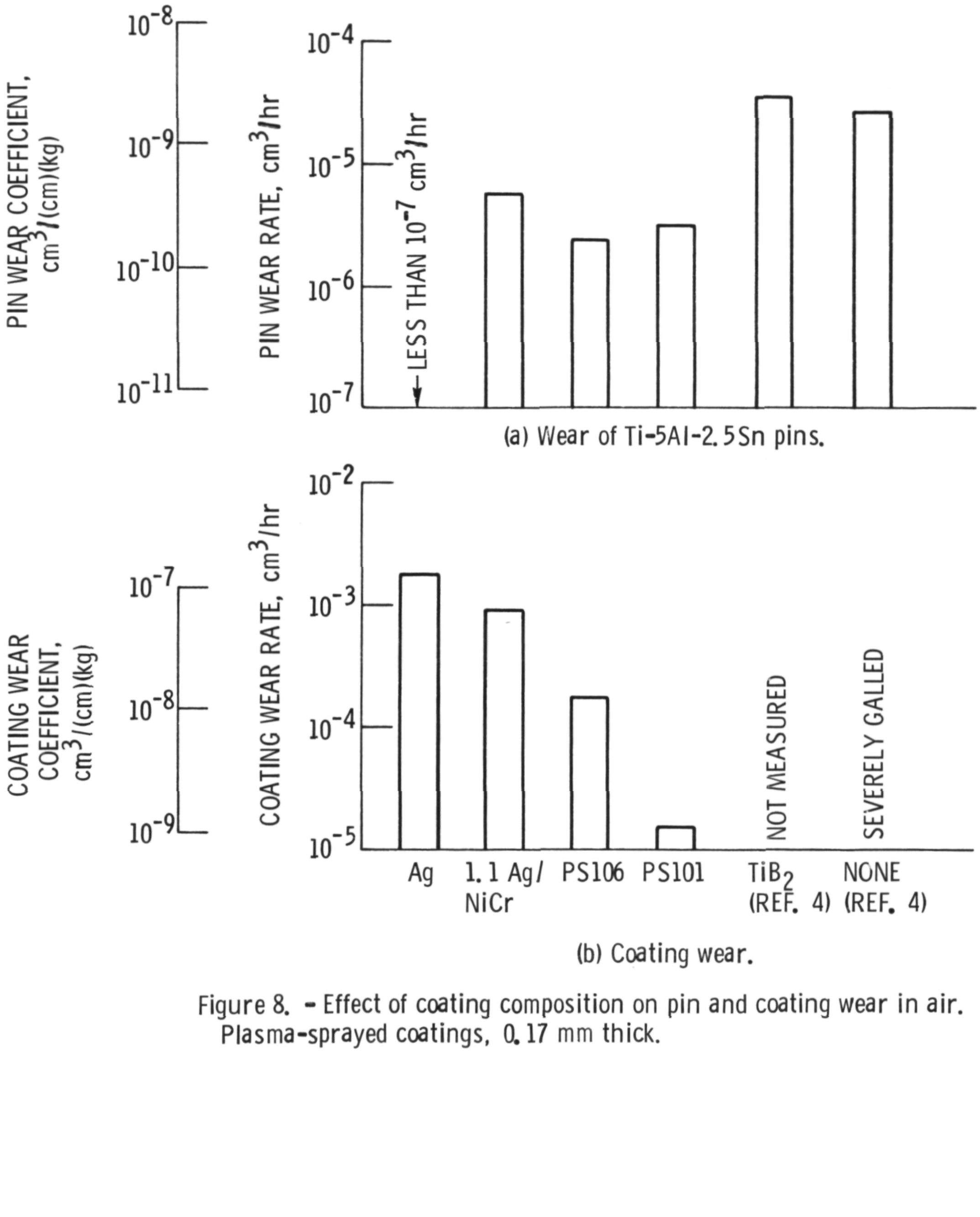

Figure 8. - Effect of coating composition on pin and coating wear in air. Plasma-sprayed coatings, 0.17 mm thick.

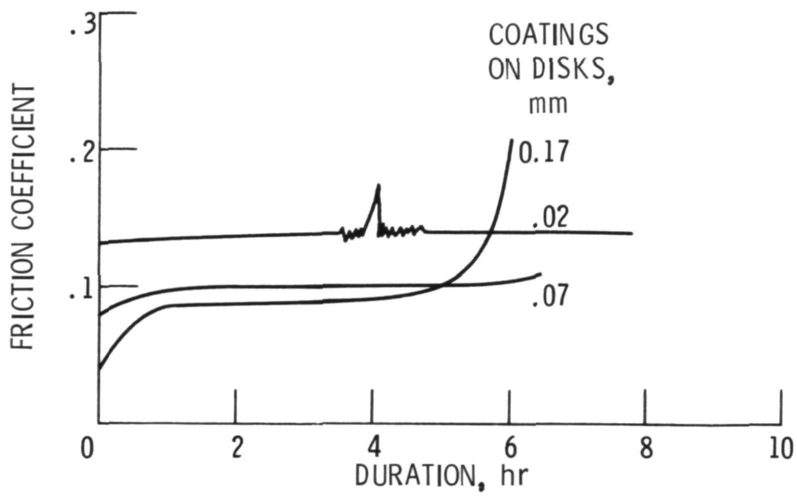

Figure 9. - Effect of coating thickness on friction of plasma-sprayed silver in air.

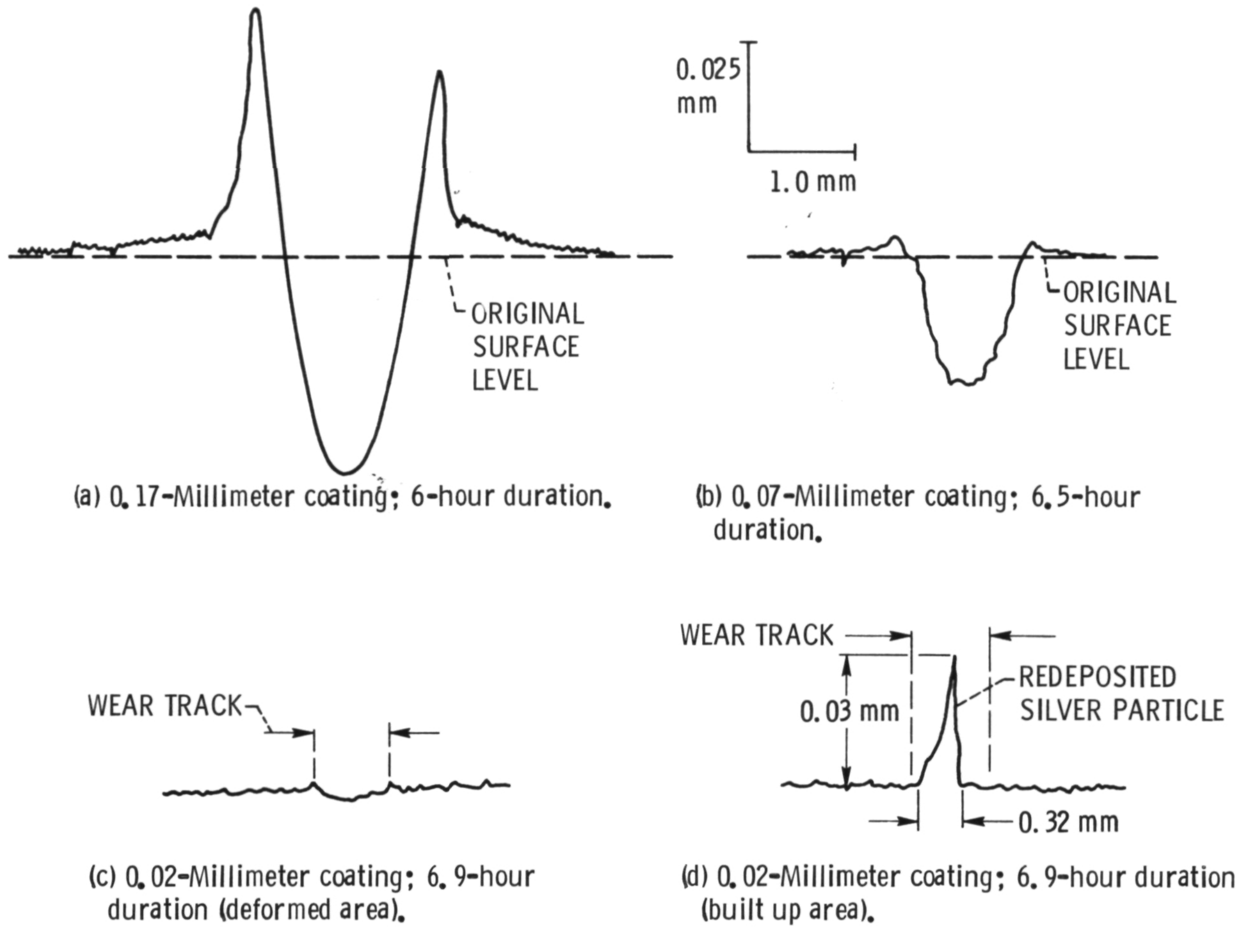

Figure 10. - Wear track cross sections showing effect of coating thickness on wear and deformation of plasma-sprayed silver coatings.

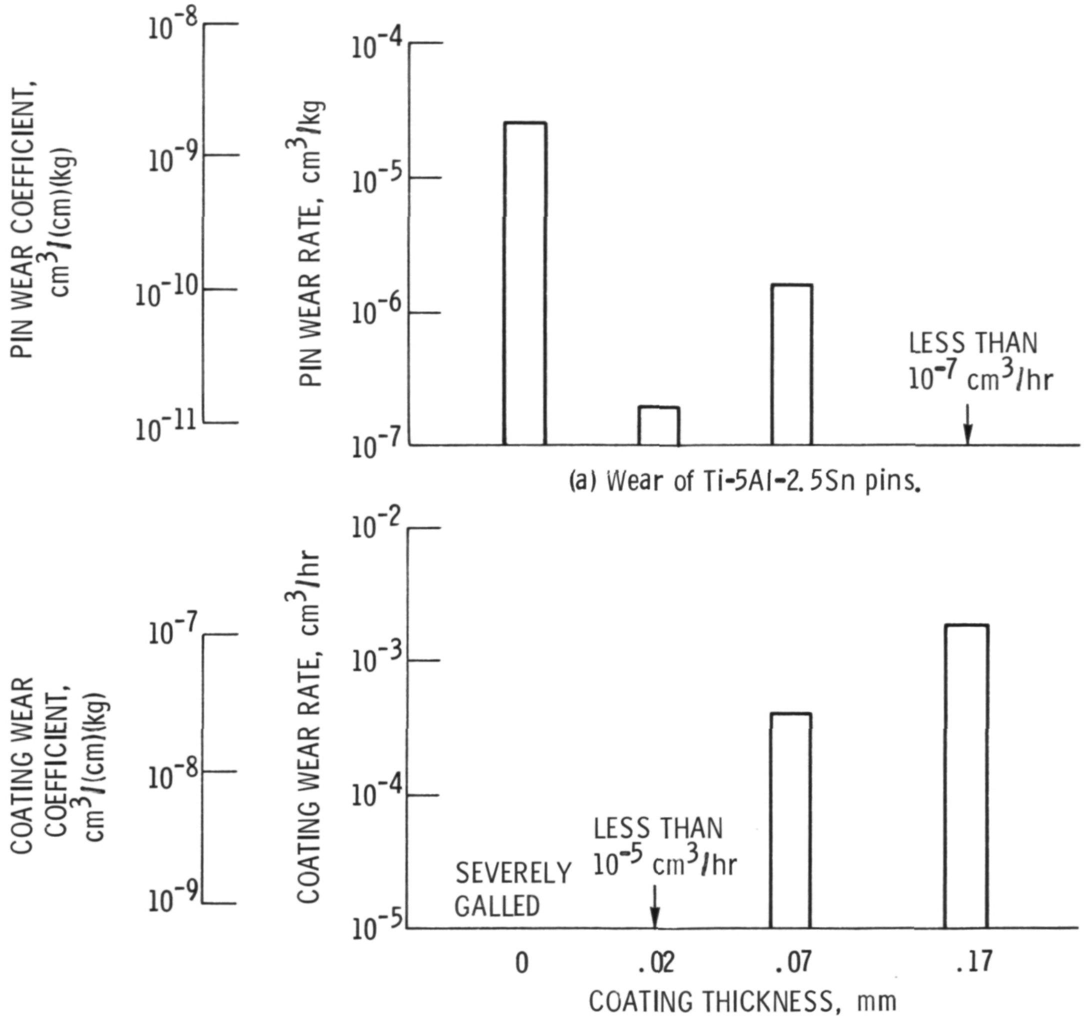

Figure 11. - Effect of silver coating thickness on pin and coating wear in air.

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16. Abstract

The advantages and disadvantages of elemental silver as a tribological material are discussed. It is demonstrated that the relatively high melting point of 961 °C, softness, marked plasticity, and thermochemical stability of silver combine to make this metal useful in thin film solid lubricant coatings over a wide temperature range. Disadvantages of silver during sliding, except when used as a thin film, are shown to be gross ploughing due to plastic deformation under load with associated high friction and excessive transfer to counterface surfaces. This transfer generates an irregular surface topography with consequent undesirable changes in bearing clearance distribution. This paper describes research to overcome these disadvantages of elemental silver. A comparison is made of the tribological behavior of pure silver with that of silver formulated with other metals and high-temperature solid lubricants. The composite materials are prepared by co-depositing the powdered components with an airbrush followed by furnace heat treatment or by plasma-spraying. Composite coatings were formulated which are shown to be self-lubricating over repeated, temperature cycles from low temperature to about 900 °C.

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