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Christopher DellaCorte  
National Aeronautics and Space Administration  
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Work performed for  
**U.S. DEPARTMENT OF ENERGY**  
**Conservation and Renewable Energy**  
**Office of Vehicle and Engine R&D**

Prepared for the  
International Conference on Metallurgical Coatings and Thin Films  
sponsored by the American Vacuum Society  
San Diego, California, April 22-26, 1996

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# THE EFFECT OF COUNTERFACE ON THE TRIBOLOGICAL PERFORMANCE OF A HIGH TEMPERATURE SOLID LUBRICANT COMPOSITE FROM 25 TO 650 °C

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## SUMMARY

The effect of counterface selection on the tribological performance of a Ag/BaF<sub>2</sub>-CaF<sub>2</sub> containing composite coating is studied. Ceramic (Al<sub>2</sub>O<sub>3</sub>) and metal (Inconel X-750) pins are slid against PS300 (a metal bonded chrome oxide coating with Ag and BaF<sub>2</sub>/CaF<sub>2</sub> lubricant additives) in a pin-on-disk tribometer at 25 500 and 650 °C.

Compared to the ceramic counterface, the metal counterface generally exhibited lower friction and wear at 25 °C but higher friction and wear at 650 °C. Friction coefficients, for example, for the Al<sub>2</sub>O<sub>3</sub>/PS300 combination at 25°C were 0.64 compared to 0.23 for the Inconel/PS300 sliding couple. At 650 °C the ranking was reversed. The Al<sub>2</sub>O<sub>3</sub>/PS300 combination gave a friction coefficient of 0.19 while the friction for the metal counterface increased slightly to about 0.3. Based upon these tribological results and other information found in the literature, it appears that the performance of each counterface/PS300 combination is affected by the ability of the solid lubricant additives to form an adequate transfer film. The effects of surface wettability and tribological compatibility are discussed in relation to the observed tribological results.

## INTRODUCTION

High temperature lubrication presents a significant challenge to the tribology community (refs. 1 and 2). At temperatures above 350°C, conventional liquid lubricants and most conventional solid lubricants (e.g. graphite and MoS<sub>2</sub>) begin to degrade (ref. 3). Under these circumstances, advanced solid lubricants must be employed. Soft noble metals (Au, Ag, Pt), inorganic fluorides (LiF, CaF<sub>2</sub>, BaF<sub>2</sub>) and some metal oxides (NiO, MoO<sub>3</sub>) have been successfully employed as solid lubricants (ref. 4). These materials generally possess stable thermochemistry at elevated temperatures as well as low shear strength properties which make them good solid lubricants.

An often overlooked aspect of solid lubrication is the selection of an appropriate counterface. To function as an effective solid lubricant a material must not only possess good thermochemical stability and low shear but must be able to form a transfer film on the sliding counterface in order to reduce friction and wear. Transfer film formation is affected by adhesion and wettability of the lubricants to the counterface. These characteristics are sometimes referred to as tribological compatibility (ref. 5).

In this paper, the effect of counterface selection on the performance of solid lubricants will be studied by examining the tribological properties of a Ag/BaF<sub>2</sub>-CaF<sub>2</sub> composite in sliding against metal and ceramic counterfaces using a pin-on-disk tribometer. In addition to comparing tribological behavior, wear surface morphology will be observed using optical and electron micrographic techniques. The tribological performance will be considered in the context of relevant research cited in the literature.

## MATERIALS

The tribological materials studied include either Al<sub>2</sub>O<sub>3</sub> or Inconel X-750 pins in sliding against a disk coated with a composite coating. The composite coating studied is designated PS300. PS300 is a plasma sprayed nichrome bonded Cr<sub>2</sub>O<sub>3</sub> coating with silver and BaF<sub>2</sub>/CaF<sub>2</sub> eutectic solid lubricant additions (ref. 6). This coating is a follow-on or next generation composition to PS200, a chrome-carbide based coating which has been shown to provide good tribological performance at temperatures as high as 900 °C (refs. 7 and 8).

Several key benefits are realized by basing the coating composition on chrome oxide rather than chrome carbide. Unlike chrome carbide, which because of its extreme hardness requires diamond grinding,

chrome oxide is readily machinable by low cost carbide grinding techniques. Furthermore, chrome oxide is a known high temperature lubricant which cannot further oxidize upon exposure to high temperature air (ref. 9).

Silver is added as the low temperature solid lubricant. It has been used to lubricate bearings, seals, fasteners and other components and exhibits good thermochemical stability over a wide temperature range (ref. 10).

$\text{BaF}_2/\text{CaF}_2$  eutectic is added as the high temperature solid lubricant. The fluoride eutectic undergoes a brittle to ductile transition at about 400 °C resulting in a reduction in shear strength; increasing its effectiveness as a lubricant (refs. 11 and 12).

Sliney pioneered the combination of silver and fluorides in composite coatings three decades ago as a way to provide reduced friction and wear from 25 to 650 °C over repeated temperature cycling (ref. 13). The PS300 coating is an extension to this concept.

### Coating Preparation

The coating composition of PS300 is as follows: 60 wt%  $\text{Cr}_2\text{O}_3$ , 20 wt% NiCr (80/20) binder, 10 wt% silver and 10 wt%  $\text{BaF}_2/\text{CaF}_2$  eutectic. More complete details and volumetric composition figures are given in Table I. The coatings were prepared by plasma spraying a simple powder blend of the constituents onto superalloy test disks which have been pre-coated with a 0.1 mm thick nichrome (80/20) bond coat. Table II shows the plasma spray parameters used to apply the coatings which were deposited in multiple passes until a thickness of 0.5 mm was achieved. Figure 1 shows cross-section micrographs of the PS300 coating. Following deposition, the coatings were ground to a final thickness (PS300 + NiCr bond coat) of 0.5 mm. Air atomized water spray was used as the grinding coolant/lubricant to prevent surface contamination with oil. The ground surface roughness was typically about 0.5 mm rms. 600 grit SiC abrasive paper was used to lightly hand polish the coating surface to a finish of 0.1 to 0.2 mm rms.

### Counterface Materials

One metal (Inconel X-750) and one ceramic ( $\text{Al}_2\text{O}_3$ ) were tested as pins in sliding against the PS300 coated disks in this study. Inconel X-750 is a nickel based superalloy with excellent high temperature strength, toughness and oxidative stability.  $\text{Al}_2\text{O}_3$  is a well known wear resistant ceramic which has been shown to perform well as a high temperature tribological material (refs. 14 and 15).

The Inconel pins are machined from precipitation hardened 9.5 mm diameter rod. The pins are 25 mm long and hemispherically tipped with a 25.4 mm radius of curvature. The pin hardness is RC32-36.

The  $\text{Al}_2\text{O}_3$  pins are prepared by diamond grinding a 25.4 mm radius of curvature on the end of sintered rod stock 9.5 mm in diameter. The material is 99.4 percent pure, fully densified alumina. Table III gives its properties as supplied by the manufacturer. The pin surfaces were polished to less than 0.2 mm rms surface roughness.

Prior to testing, the pins and disks were rinsed with pure ethyl alcohol, scrubbed with a paste of levigated  $\text{Al}_2\text{O}_3$  powder and water, rinsed with deionized water and air dried.

### Tribological Testing

The specimens are tested in a pin-on-disk test rig described in detail in reference 16 and shown in figure 2. The pin wears a 51 mm diameter track into the rotating test disk which is inductively heated to the desired test temperature. The air atmosphere is controlled and maintained at 50 percent R.H. at 25 °C. Selected test temperatures were 25, 500 and 650 °C. Test velocity was 370 rpm (1 m/s) and the load was 4.91N. These conditions were chosen to simulate foil air bearings which experience sliding contact during low journal rotation speeds during start-up/shut-down conditions.

Friction was monitored continuously during the test which typically lasts 30 min. Wear was measured using optical microscopy (for pin wear scars) and stylus surface profilometry. At least six tests were conducted for each test condition. Data uncertainties presented are one standard deviation. The tribological data for the Inconel/PS300 combination have recently been reported in reference 6 and have been incorporated in this paper for comparative purposes.

## RESULTS AND DISCUSSION

The friction and wear results are summarized in Table IV and shown graphically in figures 3 to 5. When sliding against the metal, Inconel X-750, friction and coating wear increase markedly with test temperature. Pin wear factors fluctuated but were about the same at 25 °C as at 650 °C;  $\approx 3 \times 10^{-5}$  mm<sup>3</sup>/N-m. Disk coating wear factors increased an order of magnitude over this temperature range from  $6.6 \times 10^{-5}$  mm<sup>3</sup>/N-m at 25 °C to  $7.1 \times 10^{-4}$  mm<sup>3</sup>/N-m at 650 °C. Friction at 25 °C was low, 0.23, and increased to a moderate value of 0.31 at 650 °C.

When sliding against the ceramic, Al<sub>2</sub>O<sub>3</sub>, friction and wear of both the ceramic pin and the coating decreased with test temperature. For example, at 25 °C, the friction coefficient was quite high at 0.62. Friction dropped by a factor of two to 0.32 at 500 °C and was further reduced to 0.19 at 650 °C. Wear factors for both the pin and disk coating followed the same trend. At elevated temperatures, pin wear decreased over an order of magnitude from  $10^{-6}$  to  $10^{-7}$  mm<sup>3</sup>/N-m compared to room temperature. Disk wear factors, which were  $2.3 \times 10^{-4}$  mm<sup>3</sup>/N-m at 25 °C were reduced to  $2.5 \times 10^{-5}$  mm<sup>3</sup>/N-m at 500 °C and  $7.8 \times 10^{-6}$  mm<sup>3</sup>/N-m at 650 °C.

Clearly, the trend of friction and wear was reversed for Al<sub>2</sub>O<sub>3</sub> sliding against the composite coatings. Previous research experience by the author with the solid lubricants used in PS300 and relevant information from the literature offer some plausible explanations for the tribological behavior observed.

For instance, at room temperature, the alumina pin was poorly lubricated by the PS300 coating as evidenced by very high friction. In sharp contrast, the Inconel pin exhibited low friction in sliding against the coating under identical test conditions. This may be due to the inability of the silver in PS300 to form an adequate transfer film on the ceramic pin surface needed to reduce friction.

In research conducted by Erdemir et al. (ref. 14), silver was used to lubricate Al<sub>2</sub>O<sub>3</sub>. In their work, to achieve sufficient adhesion and reduce friction, it was necessary to apply Ag to the Al<sub>2</sub>O<sub>3</sub> surface using Ion Beam Assisted Deposition (IBAD). Their coating provided lubrication at room temperature. At elevated temperatures, however, the silver spontaneously dewetted the Al<sub>2</sub>O<sub>3</sub> surface.

Subsequent work conducted by the author showed a similar result (ref. 17). Silver was unsuccessful as a lubricant for Al<sub>2</sub>O<sub>3</sub> unless an active metal (Ti) bond layer was sputter deposited between the Al<sub>2</sub>O<sub>3</sub> and the silver to promote adhesion. Without the bond layer, the silver coating rapidly delaminated causing friction levels to rise to 0.7, approximately equal to that measured in the present study for the Al<sub>2</sub>O<sub>3</sub>/PS300 combination.

The inability of silver to transfer from the PS300 coating and adhere to the Al<sub>2</sub>O<sub>3</sub> counterface may be the reason for the high friction observed. Since silver is the only material in the composite with low temperature lubrication properties (i.e. low shear strength), the friction is high. Clauss writes that obtaining good adhesion of soft metals is a major problem and that oxide films on metals reduce adhesion and film life (ref. 4). Clearly, silver is not a functional lubricant for Al<sub>2</sub>O<sub>3</sub> unless effective measures are taken to improve transfer film formation and adhesion. SEM/EDS analyses of Al<sub>2</sub>O<sub>3</sub> pin surfaces after sliding against the coating at 25 °C exhibit only small localized pockets of wear debris containing essentially all the components of PS300. No lubricant-like film is detected. Serendipitous Ag film formation on Al<sub>2</sub>O<sub>3</sub> when sliding against PS300 obviously does not occur; helping to explain the poor room temperature performance of the Al<sub>2</sub>O<sub>3</sub>/PS300 combination.

The situation was reversed when sliding against Inconel pins. At 25 °C, sliding friction is low and post test surface analyses using SEM/EDS show a more or less uniform layer of silver (and some fluorides). This behavior is very similar to results published in reference 16. In this work, transfer films of silver formed on a chromium-cobalt alloy pin in sliding against a carbide composite coating which also contained silver and BaF<sub>2</sub>/CaF<sub>2</sub> eutectic as its lubricants.

Silver alone has been shown by Bowden and Tabor to be an effective lubricant for Inconel X-750 in a ball-on-flat geometry (ref. 18). In their work, friction coefficients of 0.1 to 0.4 were obtained using silver films to lubricate the sliding contact.

In related applications, silver films are used to lubricate rolling element bearings used for x-ray tubes (ref. 4). In this case, the bearings are made from 440 C stainless steel and operate in a vacuum from 25 to 600 °C. Despite the development of this lubrication method some five decades ago (ref. 19), the use of silver films, now applied by ion-plating, persist in this application.

Additionally, silver is used throughout the aircraft and turbine industry as an antiseize and thread lubricant for superalloy fasteners (ref. 20). In this case, good adhesion under high contact stresses is essential. There is concern about silver sulphide/sulfuric acid, formed through a reaction of the silver with sulphur found in fuel, attacking fasteners and turbine components but no significantly improved lubricants have yet been identified.

Clearly, the results from the experimental work presented here as well as those reviewed from the literature show that silver is a suitable lubricant for Inconel, but not for  $\text{Al}_2\text{O}_3$ . This tribological "compatibility" helps to explain the dramatic difference in the triboperformance between the metal and ceramic counterpart.

The tribological characteristics and performance are somewhat reversed when the test temperature is increased. In fact, the lowest friction, pin wear factor and disk wear factors are measured for the  $\text{Al}_2\text{O}_3$  pins sliding against the PS300 coating at 650 °C. To understand this behavior it is useful, once again, to consider the counterface/lubricant compatibility.

The high temperature lubricant in PS300 is  $\text{BaF}_2/\text{CaF}_2$  eutectic.  $\text{BaF}_2/\text{CaF}_2$  eutectic has been shown to be a good solid lubricant above 400 °C as a thin film coating (ref. 13) and as a component in composites (refs. 16, 21, and 22). The fluoride eutectic functions by providing a low shear strength film between the sliding surfaces to mitigate friction and wear.

Sliney's early work with fluorides centered on thin, fused coatings deposited on nickel and cobalt based superalloys (refs. 23 and 13). These coatings were adherent and provided adequate life for limited sliding applications. Later work on composites offer more insight towards understanding the tribological performance of the PS300/ $\text{Al}_2\text{O}_3$ -Inconel sliding couples.

Sliney briefly conducted research on composites formed by infiltrating a porous Inconel composite with fluorides (ref. 24). In that work, he noted that when vacuum infiltration of the porous metal shell with molten fluorides was attempted, poor infiltration was exhibited if the metal (Inconel) matrix was bright, clean and free from surface oxides. If the matrix was lightly preoxidized, surface wetting of the fluorides occurred producing a strong "wicking" action. These experimental observations lend insight into the PS300 tribological results.

When sliding the PS300 against  $\text{Al}_2\text{O}_3$  at high temperatures, the fluorides readily "wet" the  $\text{Al}_2\text{O}_3$  forming a lubricous film to reduce friction and wear. Figure 6 is an SEM/EDS analysis showing that such transfer occurs. In sliding against Inconel, however, the wear process minimizes the persistence of surface metal oxides inhibiting the growth of a lubricous fluoride transfer film. Thus, against Inconel X-750 pins, high temperature friction and wear is not reduced. In fact, wear is higher. This may be due, in part, to the softening of the superalloy which occurs at these temperatures.

In any case, it is clear that at high temperatures, the high temperature lubricant ( $\text{BaF}_2/\text{CaF}_2$ ) is more functional for the  $\text{Al}_2\text{O}_3$  counterface. This concept is further corroborated by the fact that  $\text{Al}_2\text{O}_3$  readily forms compounds with  $\text{BaF}_2$  and  $\text{CaF}_2$  especially at elevated temperatures (ref. 25). In general, transfer film formation and adherence are enhanced by some sort of reaction at the interface (refs. 26 and 17).

## CONCLUDING REMARKS

The tribological characteristics of PS300 presented in this paper dramatically illustrate the important role the counterface plays in determining tribological performance. This concept is often overlooked in solid lubrication perhaps because the chemical reactivity often observed in liquid lubricated systems is not obvious for solid state systems.

For PS300, or other Ag and fluoride lubricated composites, the choice of a counterface must be carefully made taking into consideration use conditions, cost and required tribological properties. If the component to be lubricated will spend most of its operating life at low temperature with only occasional exposure to high temperatures, superalloys, like Inconel X-750, would be a good counterface choice. However, if high temperature performance is paramount,  $\text{Al}_2\text{O}_3$  may be a practical choice despite its brittle nature and, generally, higher manufacturing costs.

Since many advanced engine applications, such as gas turbine bearings, bushings and seals operate predominately at elevated temperatures, PS300 versus  $\text{Al}_2\text{O}_3$  may be a sliding combination which warrants further study and serious consideration. Furthermore, using the concepts of counterface selection and solid lubricant addition; new composites may be tailored for desirable performance in a wide variety of high temperature tribological applications.

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TABLE I.—COMPOSITION BY WEIGHT AND VOLUME PERCENT OF PS300 AND PS200

Coating Designation	Density P, g/cm <sup>3</sup>	Constituent, wt% (vol%)			
		*NiCr-Cr <sub>2</sub> O <sub>3</sub>	+Ni-Co-Cr <sub>3</sub> C <sub>2</sub>	Ag	BaF <sub>2</sub> /CaF <sub>2</sub>
PS300	5.81	80 (80.3)	-----	10 (5.5)	10 (14.2)
PS200	6.75	-----	80 (77.1)	10 (6.4)	10 (16.5)

\*By wt% contains 80 Cr<sub>2</sub>O<sub>3</sub>, 16Ni, 4Cr.

+By wt% contains 54 Cr<sub>3</sub>Cr<sub>2</sub>, 28Ni, 12Co, 2Mo, 2Al, 1B, 1 Si.

TABLE II.—PLASMA SPRAY  
PARAMETERS

[Used to apply PS300 coatings]

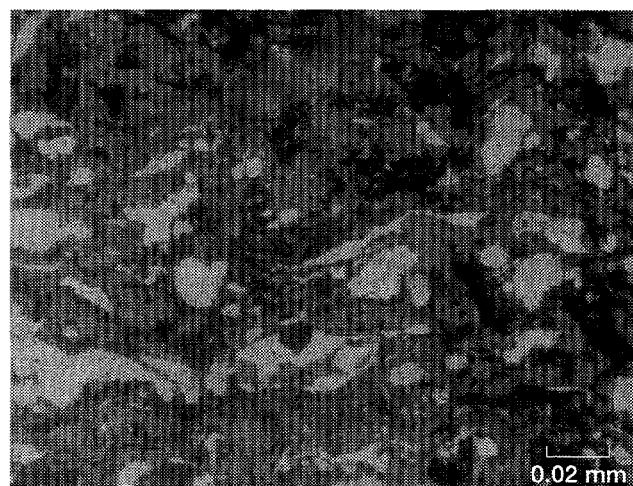
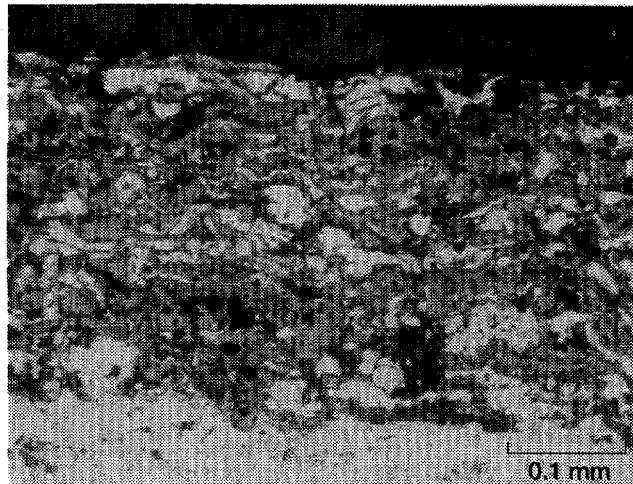
Parameter	Value
Current	600 amps
Voltage	30-32 volts
Standoff distance	8-10 cm
Argon	35 sl/min
Arc gas flow rate	≈1 kg/hr
Powder flow rate	0.4 m <sup>3</sup> /hr

TABLE III.—PROPERTIES OF  $\text{Al}_2\text{O}_3$  MATERIAL TESTED

Property	Value
Composition	99.4 wt% $\text{Al}_2\text{O}_3$ and trace Fe, Si
Density	$3.9 \text{ g cm}^{-3}$
Young's modulus	386 Gpa
Vickers hardness	$2000 \text{ kgf mm}^{-2}$
Toughness	$4.2 \text{ Mpa m}^{-1/2}$
Thermal expansion coefficient	$8.0 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$
Four-point bend strength	344 Mpa
Poisson's ratio	0.23
Thermal conductivity	$22 \text{ W m}^{-1} \text{ }^{\circ}\text{C}^{-1}$

TABLE IV.—FRICTION AND WEAR SUMMARY  
[1m/s sliding velocity, 4.9N load, 30 min test, air atmosphere at 50% R.H. at 25 °C]

Disk coating	Pin material	Temperature, °C	Friction coefficient	$K_{pi}$ , $\text{mm}^3/\text{N}\cdot\text{m}$	$K_{disk}$ , $\text{mm}^3/\text{N}\cdot\text{m}$
PS300	INCX750	25°C	$0.23 \pm 0.05$	$3.9 \pm 0.5 \times 10^{-5}$	$6.6 \pm 2.5 \times 10^{-5}$
PS300	INCX750	500°C	$0.29 \pm 0.04$	$1.3 \pm 0.3 \times 10^{-5}$	$3.9 \pm 0.3 \times 10^{-4}$
PS300	INCX750	650°C	$0.31 \pm 0.01$	$3.1 \pm 0.8 \times 10^{-5}$	$7.1 \pm 1.6 \times 10^{-4}$
PS300	$\text{Al}_2\text{O}_3$	25°C	$0.62 \pm 0.06$	$3.3 \pm 2.1 \times 10^{-6}$	$2.3 \pm 0.4 \times 10^{-4}$
PS300	$\text{Al}_2\text{O}_3$	500°C	$0.32 \pm 0.07$	$2.6 \pm 1.9 \times 10^{-7}$	$2.5 \pm 1.0 \times 10^{-5}$
PS300	$\text{Al}_2\text{O}_3$	650°C	$0.19 \pm 0.02$	$2.1 \pm 1.3 \times 10^{-7}$	$7.8 \pm 2.3 \times 10^{-6}$



**Figure 1.—Cross-sectional optical micrographs of PS300 showing plasma sprayed composite coating structure.**

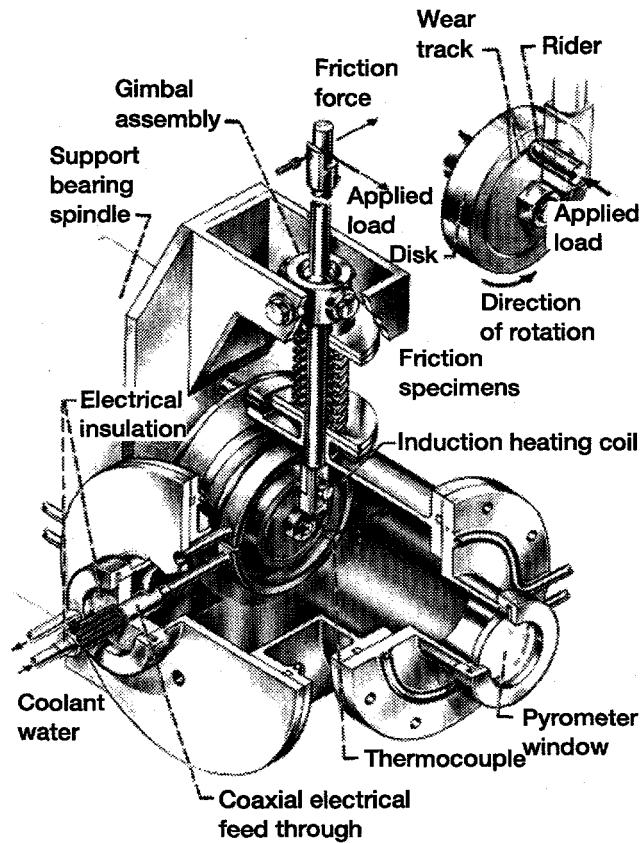


Figure 2.—High temperature friction and wear test rig.

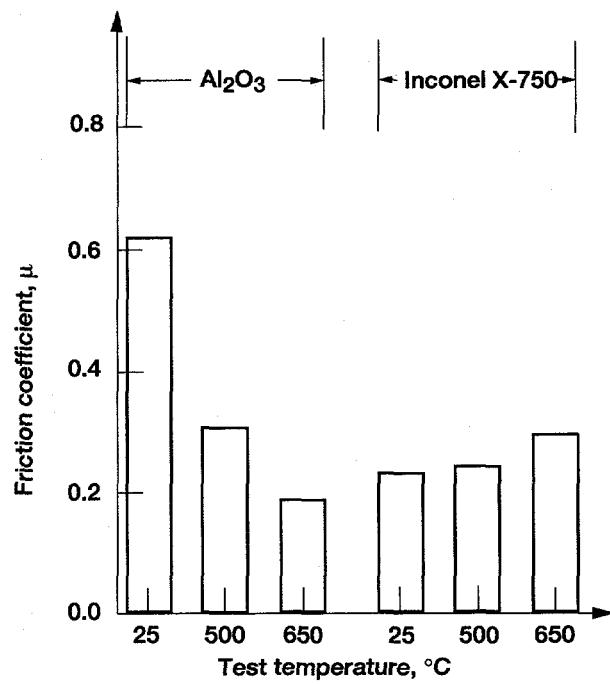


Figure 3.—Friction coefficient,  $\mu$ , vs. test temperature for both the ceramic ( $\text{Al}_2\text{O}_3$ ) and superalloy (Inconel X-750) pins sliding against PS300 under a 4.9N load, 1m/s sliding velocity, in air.

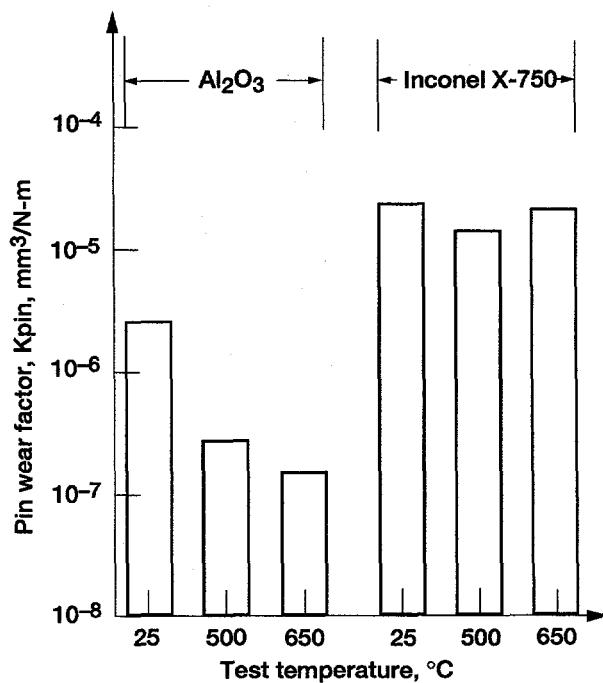


Figure 4.—Pin wear factor,  $K_{pin}$ , vs. test temperature for both the ceramic ( $\text{Al}_2\text{O}_3$ ) and superalloy (Inconel X-750) pins sliding against PS300 under a 4.9N load, 1m/s sliding velocity, in air.

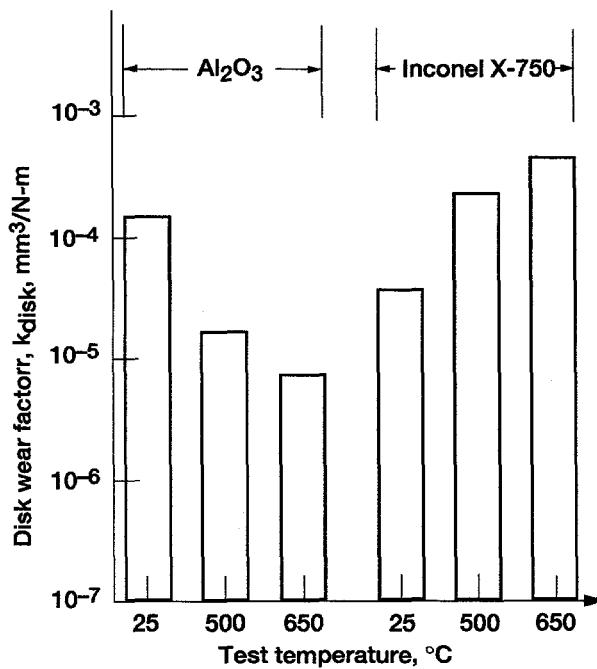


Figure 5.—PS300 coating wear factor,  $K_{disk}$  vs. test temperature for both the ceramic ( $\text{Al}_2\text{O}_3$ ) and superalloy (Inconel X-750) pins sliding against PS300 under a 4.9N load, 1m/s sliding velocity, in air.

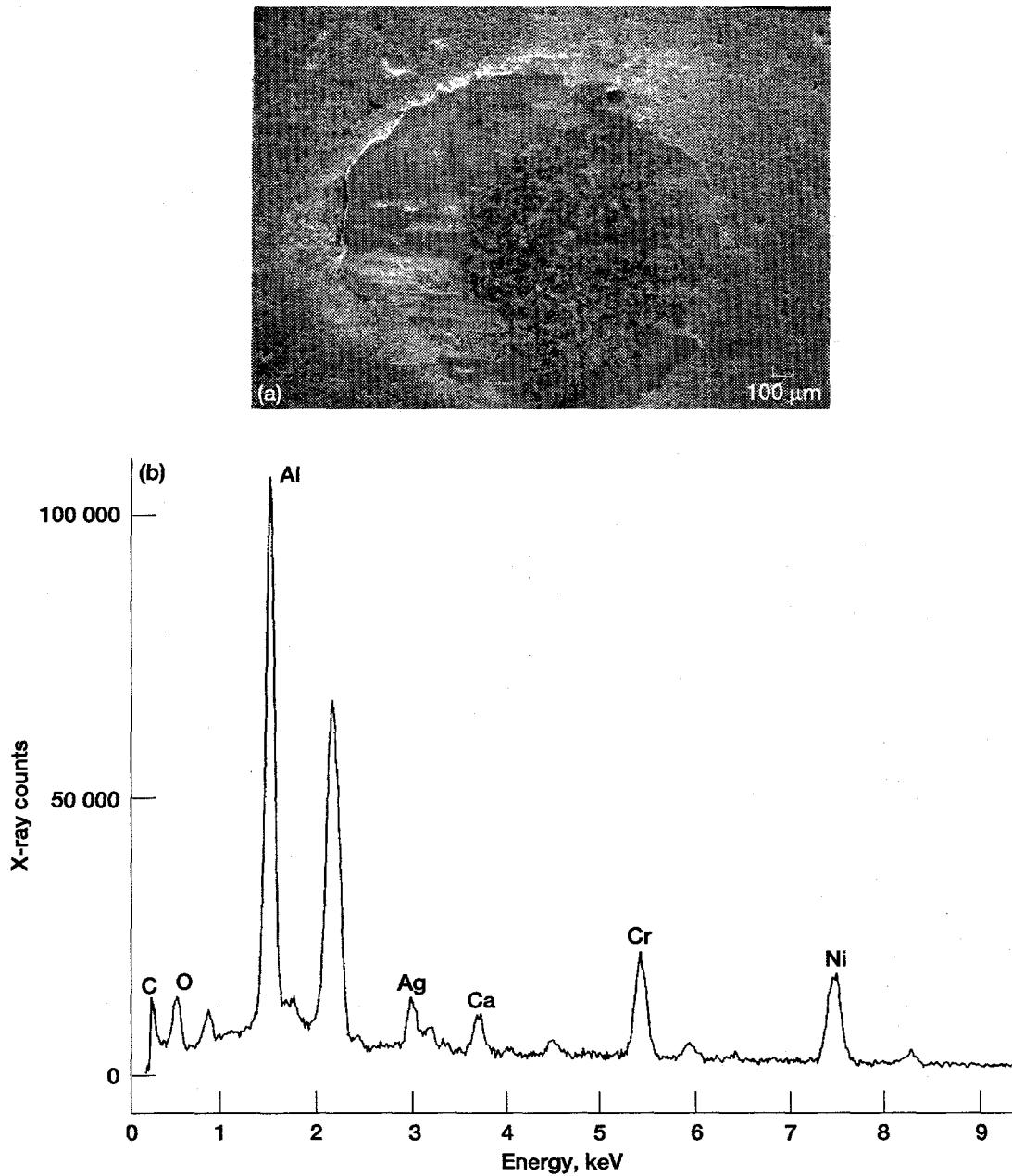


Figure 6.—(a) SEM micrograph of  $\text{Al}_2\text{O}_3$  pin surface after sliding against PS300 at  $650^\circ\text{C}$ ,  $1\text{ m/s}$ .  
 (b) Corresponding EDS X-ray spectrum of wear surface showing lubricant (Ba, Ca, Ag) peaks,  $20\text{ kV}$  accelerating energy.

# REPORT DOCUMENTATION PAGE

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<b>13. ABSTRACT (Maximum 200 words)</b> <p>The effect of counterface selection on the tribological performance of a Ag/BaF<sub>2</sub>-CaF<sub>2</sub> containing composite coating is studied. Ceramic (Al<sub>2</sub>O<sub>3</sub>) and metal (Inconel X-750) pins are slid against PS300 (a metal bonded chrome oxide coating with Ag and BaF<sub>2</sub>/CaF<sub>2</sub> lubricant additives) in a pin-on-disk tribometer at 25 500 and 650 °C. Compared to the ceramic counterface, the metal counterface generally exhibited lower friction and wear at 25 °C but higher friction and wear at 650 °C. Friction coefficients, for example, for the Al<sub>2</sub>O<sub>3</sub>/PS300 combination at 25°C were 0.64 compared to 0.23 for the Inconel/PS300 sliding couple. At 650 °C the ranking was reversed. The Al<sub>2</sub>O<sub>3</sub>/PS300 combination gave a friction coefficient of 0.19 while the friction for the metal counterface increased slightly to about 0.3. Based upon these tribological results and other information found in the literature, it appears that the performance of each counterface/PS300 combination is affected by the ability of the solid lubricant additives to form an adequate transfer film. The effects of surface wettability and tribological compatibility are discussed in relation to the observed tribological results.</p>				
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