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C. DellaCorte
National Aeronautics and Space Administration
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J.A. Laskowski
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Tribological Evaluation of PS300: A New Chrome Oxide Based Solid Lubricant Coating Sliding

Against Al_20_3 from 25 to 650 °C

by

C. DellaCorte, NASA LeRC
J.A. Laskowski, Parks College
for Joint Tribology Conference

ABSTRACT

This paper presents the tribological characteristics of Al_20_3 sliding against PS300; a chrome oxide based self lubricating coating. Al_20_3 pins were slid against PS300 coated superalloy disks in air, under a 4.9N load at velocities of 1 to 8 m/s. At a sliding velocity of 1 m/s, friction ranged from 0.6 at 25 °C to 0.2 at 650 °C. Wear factors for the Al_20_3 pins were in the 10^{-7} mm^3/N-m range and for the PS300 coating were in the 10^{-5} mm^3/N-m range. The test results suggest that increased surface temperature resulting from either frictional heating, generated by increased sliding velocity, or ambient heating caused a reduction in friction and wear of the sliding couple. Based upon these results, the tested material combination is a promising candidate for high temperature wear applications.

Key Words

Tribology, Friction, Wear, Ceramics, High temperature, Solid lubricants, Coatings

INTRODUCTION

The need to provide lubrication to ceramics, especially at elevated temperatures is a challenging and ongoing technical problem (Ref. 1). PS300, a new coating developed at the author’s laboratory, has shown promise in lubricating superalloys at temperatures as high as 800 °C (Ref. 2). In the following paper PS300 is considered as a candidate lubricant for ceramics.

PS300 is a metal bonded-chrome oxide based plasma sprayed coating with silver and fluoride lubricant additions. PS300 is a follow-on coating to PS200 (Ref. 3), a chrome-carbide based coating, and has been shown to provide comparable friction and wear properties to PS200 with several key benefits (Ref. 2).

One benefit is that the substitution of chrome oxide for chrome carbide eliminates the need for diamond grinding finishing processes. Chrome oxide itself has been shown to be a good high temperature lubricant (Refs. 4 and 5). Since it is already oxidized, it will not degrade upon exposure to air at elevated temperatures. Another benefit is that the raw materials for PS300 are less costly than those for PS200. In addition, though the reasons are not known, the plasma spray process was found to be less sensitive to deposition parameters for the new coating and the deposition efficiency was higher compared to the PS200 (Ref. 2).

PS300 has been evaluated in sliding against a variety of materials under low sliding velocity conditions (Ref. 2). The following paper describes a research program to further develop and evaluate the PS300 coating. In particular, its tribological performance in sliding contact with the ceramic, aluminum oxide, is investigated over a wide temperature and sliding velocity spectrum. A goal of the research is to determine if PS300 can be used to lubricate ceramics effictively at high temperatures up to 650 °C and sliding velocities up to 8 m/s. Sliding tests were conducted using Al_20_3 pins loaded against the rotating face of a PS300 coated superalloy disk in a high temperature tribometer.

Experimental: Materials and Specimen Properties

The detailed compositions, by weight and volume percent, of PS300 and PS200 are given in Table I. PS300 is comprised of a wear resistant matrix of nickel-chrome bonded chromium oxide, silver, a low temperature lubricant and BaF_2/CaF_2 eutectic, a high temperature lubricant. The PS300 coating is formed by plasma spraying a simple powder blend of the constituents. Powder particle sizes range from 20 to 150 mm and the plasma spray parameters used to apply the coating are given in Table II. The coating is characterized using cross section metallography, electron microscopy and x-ray fluorescence (for bulk composition analysis). Figure 1 shows some representative cross-section micrographs of the coating. The plasma spray process produces a fairly uniform "splat" type coating with some residual low level porosity. In the optical photomicrographs shown in Fig. 1, the bright areas are NiCr and Ag; the grey areas are Cr_2O_3 and the black areas are voids and BaF_2lCaF_2.

To prepare a disk sample, a 0.5 mm thick PS300 coating mixture was plasma sprayed onto a grit blasted disk surface which has been previously coated with a 0.1mm thick NiCr (80/20) bond coat layer. the resulting coating was then ground to a final thickness of about 0.3mm. The ground roughness was about 0.5mm rms. 600 grit silicon carbide abrasive paper and water was used to lightly polish the surface to a finish of 0.1 to 0.2 mm rms.

The pin specimens were 2.5 cm long, 0.95 cm diameter cylinderical rods with both ends finished to a 2.5 cm radius of curvature. The pins were prepared by diamond grinding from 99.4% purity Al_20_3. The pin surface was polished to less than 0.2 mm rms surface roughness. Prior to testing, the pins and disks were rinsed with ethyl alcohol, scrubbed with a paste of levigated Al_20_3 and water, rinsed with deionized water and dried with lab air.

Experimental: Tribological Testing

The specimens were tested in a pin-on-disk test rig described in detail in Ref. 3 and shown in Fig. 2. The pin wears a 51 mm diameter track into the rotating test disk which was inductively heated to the desired test temperature. The air atmosphere was controlled and maintained at 50% R.H. at 25 °C. Selected test temperatures were 25,500, and 650 °C. These temperatures represent the anticipated application temperature for foil bearings. The initial test velocity was 370 rpm (1 m/s) and the load was 4.91N. These conditions were chosen to simulate the start-up/shut-down conditions for foil air bearing applications. Additional tests were conducted at higher sliding velocities (1000 rpm, 2000 rpm and 3000 rpm) to assess the effect of sliding speed on friction and wear. For these tests, the total sliding distance was kept constant (3.6 km) and equal to 60 minutes of sliding at 370 rpm.

Friction was monitored continuously during the tests which typically last 30 minutes. Wear was measured using optical microscopy (for pin wear scars) and stylus surface profilometry. For tests conducted at 1 m/s, new pin and disk specimen pairs were used for each test temperature and typically three repeats were run on each pair. For this condition (1 m/s velocity) the data uncertainties presented are one standard deviation. For tests conducted at higher velocities, two repeat tests were conducted on each pin and disk pair tested at each test temperature and velocity. Thus, for these higher velocity tests, the data set is too small for valid statistical analysis and the data presented represents the average and the range.

RESULTS AND DISCUSSIONS

The test results are summarized in Table III. Friction and wear at 1m/s sliding velocity are shown in Figs. 3 and 4. The coefficient of friction for the Al_20_3/PS300 sliding pair was high at 25 °C, about 0.62, and decreased significantly to 0.19 at

650 °C. Figure 5 plots three typical test runs and illustrates the uniformity and smoothness of the friction coefficient over a 60 minute test interval.

The friction at 25 °C is much higher when PS300 is sliding against Al_20_3 rather than Inconel X-750. As reported previously under the same test conditions (Ref. 2), the friction coefficient for PS300 versus Inconel X-750 was about 0.3. The increased friction when sliding against Al_20_3 may be due to the poor wetability of silver (Ref. 6), which is the low temperature lubricant in PS300, to the Al_20_3 counterface inhibiting the development of a sufficient lubricating transfer film.

At elevated temperatures, however, the friction was significantly reduced. This may be due to the lubricating effect of PS300's high temperature lubricant additive, BaF_2/CaF_2 eutectic. The eutectic has been shown to be an effective solid lubricant above 400 °C (Ref. 7). The friction reduction with temperature may also be affected by the presence of the Cr_20_3 which is an intrinsic solid lubricant above 500 °C (Refs. 4 and 6). From an engineering standpoint, however, low friction (<0.30) at full operating temperatures, for a gas turbine seal for example, is of significant value since most machinery runs at low temperatures only during brief start-up periods.

Sliding velocity has an interesting effect on friction which appears to be complimentary to the effect of temperature. Figure 6 shows the friction as a function of sliding velocity for all three test temperatures. As discussed, at 25 °C the friction at 1 m/s is high, approximately 0.6. This value is reduced to about 0.3 at 8.1 m/s. At elevated temperatures, the effect of velocity is very small. This response to velocity suggests that frictional heating of the sliding surfaces contributes to reduced friction as does ambient heating of the surfaces.

The wear of both the Al_20_3 and the PS300, shown in Figs. 7 and 8, exhibits the same trend with temperature and velocity as the trend observed for friction. Reference 3 defines low wear as 10^{-7} mm^3/N-m, moderate wear as 10^{-5} to 10^{-6} mm^3/N-m and high wear as greater than 10^{-4} mm^3/N-m. At low speed (1 m/s), the wear factors for the PS300 coating range from 10^{-4} mm^3/N-m to 10^{-6} mm^3/N-m as the test temperature increased from 25 to 650 °C. At high sliding velocities coating wear also decreased with increasing ambient temperature but the trend was less dramatic. Similarly, pin wear factors exhibited a large temperature dependence at 1 and 2.7 m/s sliding velocity, but at higher speeds the temperature dependence was not uniformally observed or as significant.

In previous testing of PS300 against the nickel based superalloy Inconel X-750 the opposite trends with ambient temperature were observed (Ref. 2). At room temperature, the friction was 0.23 and at 650 °C it increased to 0.31. Similarly, coating wear increased an order of magnitude from 7×10-5 to 7×10-4 mm^3/N-m when the test temperature was increased from 25 to 650 °C under the same sliding conditions. Clearly, the tribological properties are sensitive to the counterface material.

This phenomena may be due to transfer film development and has been referred to as tribological compatibility (Ref. 8). It has been shown in work with carbide based coatings using the same lubricants (Ag + BaF_2/CaF_2) that transfer of the solid lubricant additives play a key role in reducing friction and wear (Ref. 3). Since noble metals like Au and Ag do not spontaneously wet ceramics (e.g. Al_20_3), room temperature performance is likely to be poor (Refs. 6 and 9). However, as the ambient temperature is increased, the fluorides soften and are able to develop a lubricous transfer film on the Al_20_3 counterface since fluorides do wet oxide ceramics (Ref. 10).

EDS analysis of the worn Al_20_3 pins corroborates this hypothesis. Little or no detectable transfer of silver occurs during room temperature sliding. Noticeable x-ray signals from the lubricants Ba, Ca and Ag are, however, detected on tribological specimens from high temperature tests (see Fig. 9).

These results suggest that room temperature performance of the Al_20_3/PS300 sliding couple may be improved by making the counterface a composite of metal and ceramic.

CONCLUDING REMARKS

The friction and wear of PS300 was evaluated in sliding against Al_20_3 over a wide temperature range. Friction and wear were noticeably higher at room temperature than at 650 °C. At higher sliding velocities, friction and wear remained low over the entire test temperature range exhibiting very little sensitivity with temperature. On the basis of these results it is apparent that ambient or self generated surface temperatures may be playing a role in reducing friction and wear.

Analyses of the worn pin surfaces suggest that the higher temperatures promote the development of a lubricous fluoride transfer film. At low speed and low temperature, the sliding couple must rely on the low temperature lubricant, silver, which; perhaps due to is inability to wet oxide ceramics, fails to provide adequate lubrication.

Nonetheless, Al_20_3 is a practical engineering counterface for the PS300 coatings. Wear during sliding against Al_20_3 was an order of magnitude less than for the PS300 coating sliding against a superalloy. In addition, high temperature friction was observed to be significantly lower. Many intended applications for this type of solid lubricant coating (e.g. foil bearings, turbine seals) operate almost exclusively at high sliding speeds and elevated temperatures, making room temperature, low speed friction performance a less ctitical issue.

REFERENCES

1. Benoy, P.A. and DellaCorte, C. , “Au/Cr Sputter Coating for the Protection of Al_20_3 During Sliding at High Temperatures”, NASA TM–106853, March, 1995.

2. DellaCorte, C. and Edmonds, B.J., “Preliminary Evaluation of PS300: A New Self-Lubricating High Temperature Composite Coating for Use to 800 °C”, NASA TM–107056, November 1995.

3. DellaCorte, C. and Sliney, H.E., “Composition Optimization of Self-Lubricating Chromium-Carbide Based Composite Coatings for Use to 760 °C”, ASLE Transactions, Vol. 30, 1, pp 77–83, 1987.

4. Liu, G.H., Gras, R., and Blovet, J., “Characterization of Composite Tribological Coatings: Composition, Microstructure and Mechanical Properties”, Surface Coatings and Technology, 58 (1993) pp. 199–203.

5. Liu, G. H., Rubbevalloire, F., and Blovet, J., “Improvement in Tribological Properties of Chrome Oxide Coating at High Temperature by Solid Lubricants”, Wear, 160 (1993) pp. 181–189.

6. Erck, R.A., Erdemir, A., and Fenske, G.R., “Effect of Adhesion on Tribological Properties of Ag-Coated Alumina”, Surface and Coatings Technology, 43–44, pp. 577–578, 1990.8. H.E. Sliney, T.N. Strom and G.P. Allen, “Fluoride Solid Lubricants for Extreme Temperatures and Corrosive Environments”, ASLE Transactions, 8 (1965) pp. 307–322.

7. Sliney, H.E., Strom, T.N., and Allen, G.P., "Fluoride Solid Lubricants for Extreme Temperatures and Corrosive Environments", ASLE Transactions, 8 (1965) pp. 307–322.

8. Fukuda, K., Sato, Y., Sato, T., and Veki, M., "Wear Properties of SiC-Whisker Reinforced Ceramics Against Bearing Steel" in Tribology of Composite Materials, ASM International, Metals Park, Ohio, 1990 pp. 323–328.

9. DellaCorte, C., Pepper, S.V. and Honecy, F.S., "Tribological Properties of Ag/Ti Films on $Al_2 0_3$ Ceramic Substrates", Surface Coatings Technology, Vol. 52, pp. 31–37, 1992.

10. Levin, E.M., Robbins, C.R., and McMurdic, H.F., Phase Diagrams for Ceramists #1673, pp. 470, American Ceramic Society, 1984.

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

Coating Designation	Density P, g/cm^3	Constituent, wt% (vol%)			
		*NiCr-Cr_2O_3	+Ni-Co-Cr_3C_2	Ag	BaF_2/CaF_2
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_2O_3, 16Ni, 4Cr.

+By wt% contains 54 Cr_3Cr_2, 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 Arc gas flow rate	35 sl/min
Powder flow rate	≈1 kg/hr
Powder gas flow rate	0.4 m^3/hr

TABLE III.—FRICTION AND WEAR DATA SUMMARY
(4.9N load, air atmosphere)

Temperature, °C	rpm (velocity), m/s	μ	Kpin, mm^3/N-m	Kdisk, mm^3/N-m	Number repeats
25	376 (1)	0.62±0.06	$3.3\pm2.1\times10^{-6}$	$2.3\pm0.4\times10^{-4}$	7
25	1000 (2.7)	0.52±0.02	$1.3\pm0.9\times10^{-5}$	$6.9\pm3.5\times10^{-4}$	2
25	2000 (5.4)	0.34±0.02	$6.9\pm3.1\times10^{-7}$	$4.5\pm3.0\times10^{-5}$	2
25	3000 (8.1)	0.31±0.01	$1.4\pm0.1\times10^{-7}$	$1.8\pm0.5\times10^{-5}$	2
500	376 (1.0)	0.32±0.07	$2.6\pm1.9\times10^{-7}$	$2.5\pm1.0\times10^{-5}$	9
500	1000 (2.7)	0.33±0.02	$2.7\pm1.9\times10^{-7}$	$3.2\pm1.1\times10^{-5}$	2
500	2000 (5.4)	0.25±0.01	$8.1\pm7.1\times10^{-7}$	$1.8\pm0.4\times10^{-5}$	2
500	3000 (8.1)	0.28±0.03	$5.6\pm1.6\times10^{-6}$	$4.5\pm2.3\times10^{-5}$	2
650	376 (1.0)	0.19±0.02	$2.1\pm1.3\times10^{-7}$	$7.8\pm5.3\times10^{-6}$	7
650	1000 (2.7)	0.19±0.02	$0.85\pm0.35\times10^{-7}$	$1.3\pm0.2\times10^{-5}$	2
650	2000 (5.4)	0.28±0.02	$6.3\pm1.5\times10^{-7}$	$2.8\pm0.2\times10^{-5}$	2
650	3000 (8.1)	0.26±0.01	$9.1\pm5.3\times10^{-8}$	$2.7\pm1.8\times10^{-5}$	3

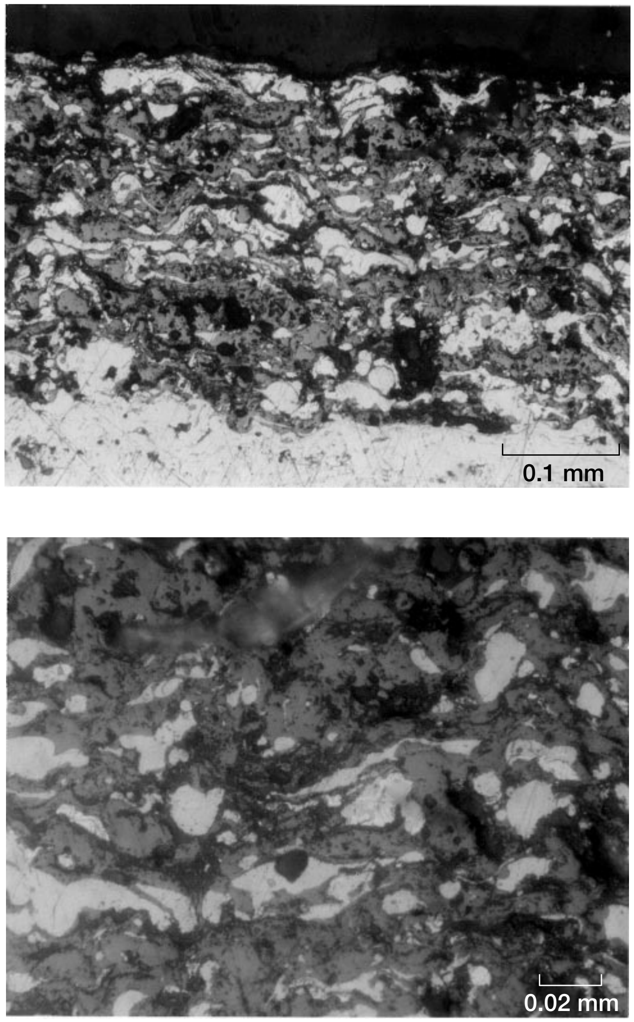


Figure 1.—Cross-sectional optical micrographs of PS300 showing plasma sprayed composite coating structure. Bright areas are NiCr and Ag; gray areas are Cr_2O_3 and black areas are porosity and BaF_2/CaF_2.

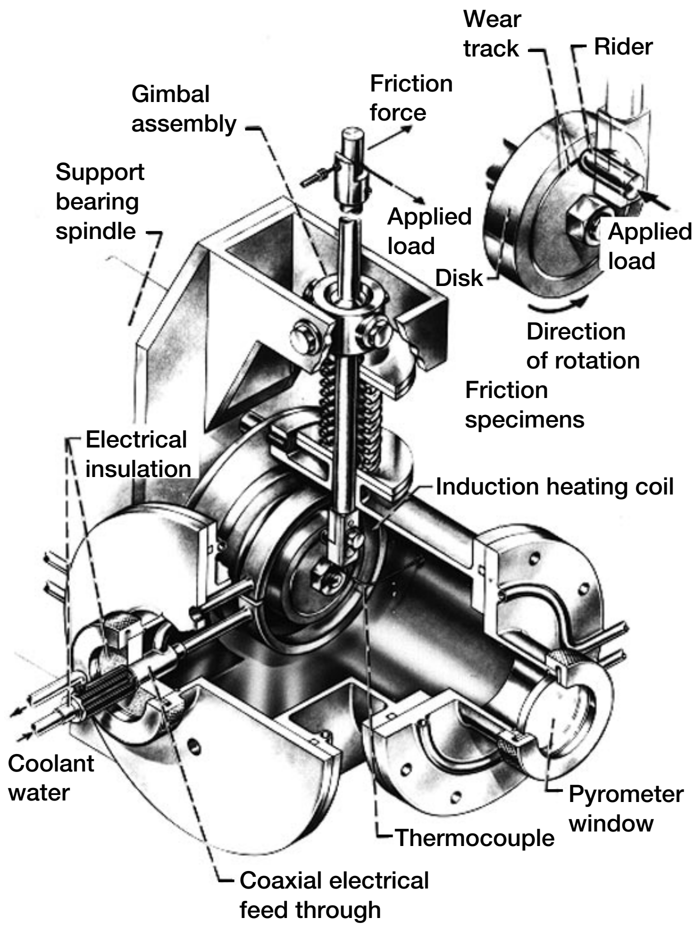


Figure 2.—Induction heated pin-on-disk rig.

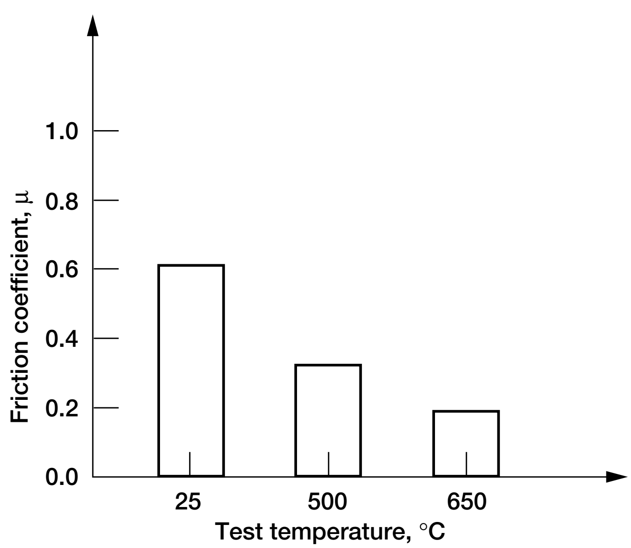


Figure 3.—Friction coefficient for Al_2O_3 pins sliding against PS300 at 1 m/s, 4.9-N load, in air.

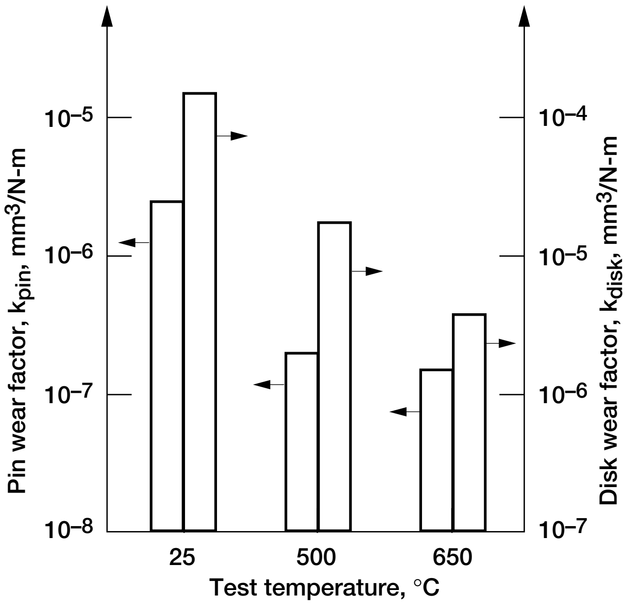


Figure 4.—Specimen wear for Al_2O_3 pins sliding against PS300 at 1 m/s, 4.9-N load, in air.

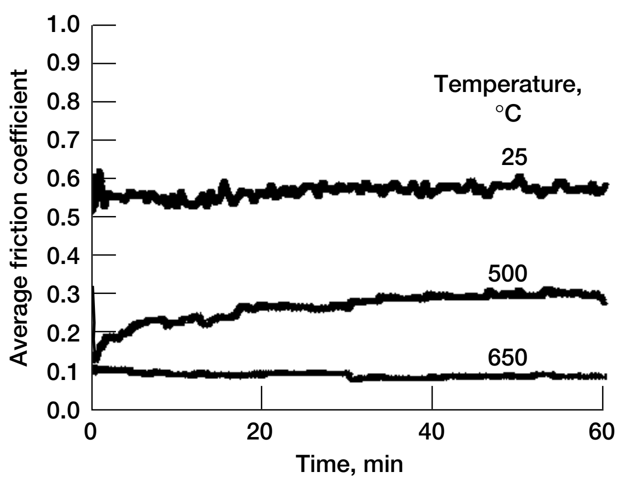


Figure 5.—Typical friction vs. sliding time traces for Al_2O_3 pins sliding against PS300 at 1 m/s.

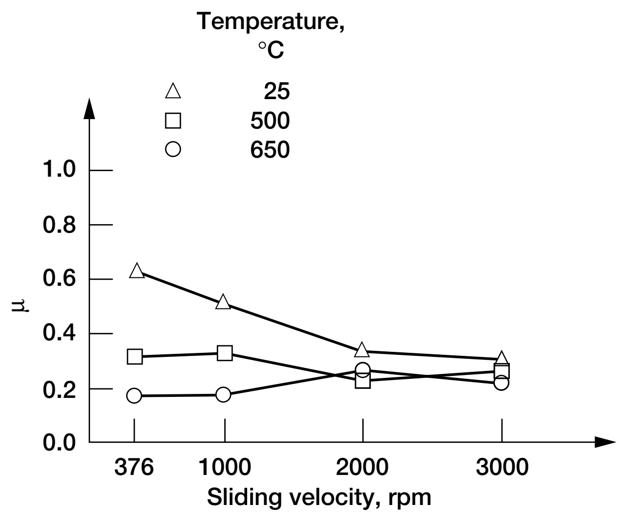


Figure 6.—Friction vs. sliding velocity.

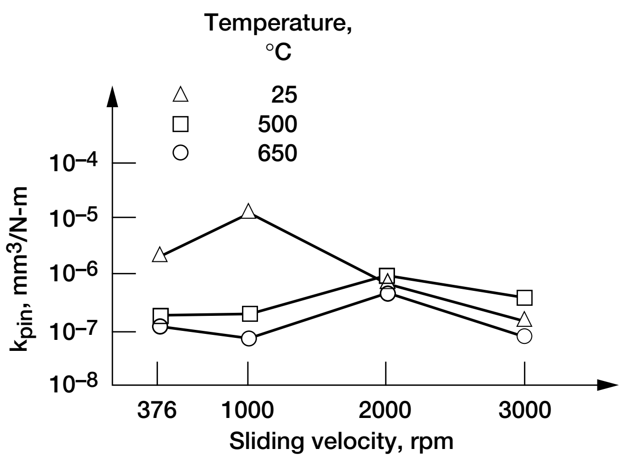


Figure 7.—Pin wear factor vs. sliding velocity.

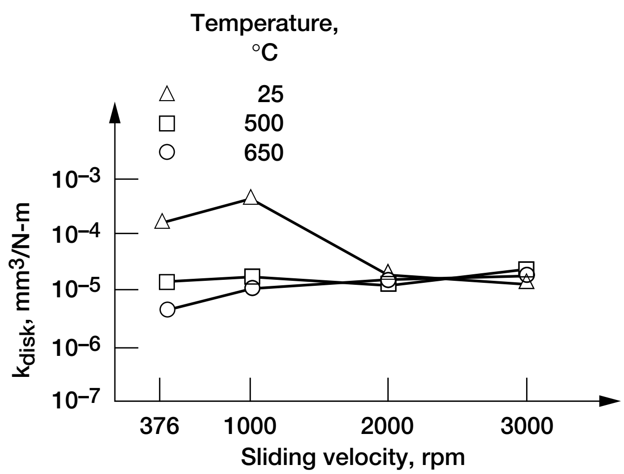
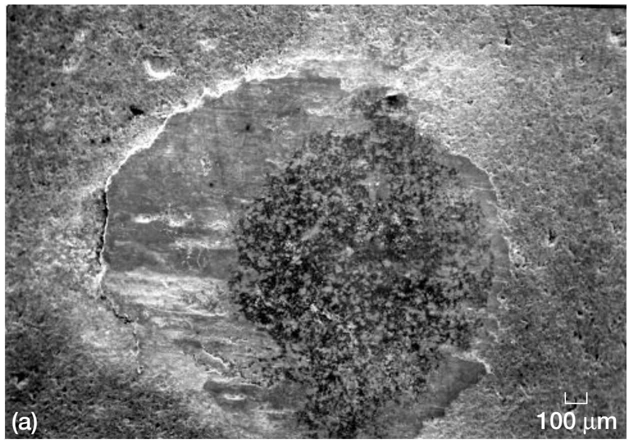


Figure 8.—Disk wear factor vs. sliding velocity.



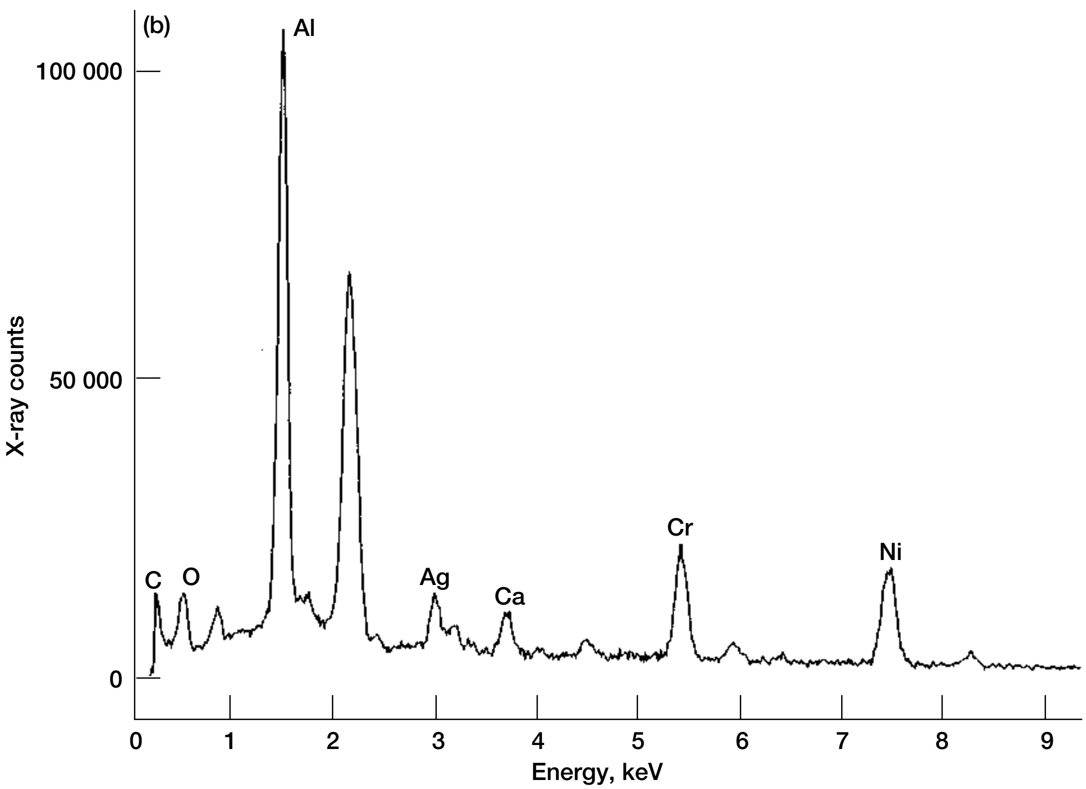


Figure 9.—(a) SEM micrograph of Al_2O_3 pin surface after sliding against PS300 at 650 °C, 1 m/s. (b) Corresponding EDS X-ray spectrum of Al_2O_3 wear surface showing lubricant (Ba, Ca, Ag) peaks, 20 kV accelerating energy.

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13. ABSTRACT ***(Maximum 200 words)***

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