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Au/Cr Sputter Coating for the Protection of Alumina During Sliding at High Temperatures

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Work performed for
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ABSTRACT

A sputter deposited bilayer coating of gold and chromium was investigated as a potential solid lubricant to protect alumina substrates in applications involving sliding at high temperature. The proposed lubricant was tested in a pin-on-disk tribometer with coated alumina disks sliding against uncoated alumina pins. Three test parameters; temperature, load, and sliding velocity were varied over a wide range in order to determine the performance envelope of the Au/Cr solid lubricant film. The tribo-tests were run in an air atmosphere at temperatures of 25 to 1000 °C, under loads of 4.9 to 49.0 N and at sliding velocities from 1 to 15 m/s. Post test analyses included surface profilometry, wear factor determination and SEM/EDS examination of worn surfaces.

Compared to unlubricated Al_2O_3 sliding, the use of the Au/Cr film reduced friction by 30 to 50 percent and wear by one to two orders of magnitude. Increases in test temperature resulted in lower friction and the Au/Cr film continued to provide low friction, about 0.3, even at 1000 °C. Pin wear factors and friction were largely unaffected by increasing loads up to 29.4 N. Sliding velocity had essentially no effect on friction, however, increased velocity reduced coating life (total sliding distance). Based upon these research results, the Au/Cr film is a promising lubricant for moderately loaded, low speed applications operating at temperatures as high as 1000 °C.

INTRODUCTION

Ceramics, such as alumina, with their low thermal conductivity and high temperature stability, are attractive materials for high temperature applications. However, if they are to be used in sliding contacts, an appropriate lubrication scheme must be implemented. Thermal breakdown renders typical liquid lubricants ineffective above temperatures of approximately 350 °C. Therefore, we have chosen to investigate solid lubrication, using a sputter deposited bilayer coating of Au and Cr, for the protection of alumina substrates during sliding at elevated temperature.

The use of thin soft coatings of materials such as gold to protect hard substrates is not new technology (ref. 1). While soft coatings, such as gold, lead, copper, silver, and others have historically been used to protect harder substrates, using soft metallic films to protect ceramic surfaces is a rather recent development. The underlying principle remains the same whether the substrate is metallic or ceramic; under a sliding load the soft overlayer shears rather than abrades, protecting the hard substrate which in turn actually carries the load.

The following simple equation, based on the junction theory of friction, can be used to estimate the coefficient of friction for hard substrates lubricated by thin soft solid films;

$$\mu = \tau_i / p_o$$

Where τ_i is the shear strength of the interfacial thin solid and p_o is the indentation pressure of the substrate material (ref. 2).

A number of studies have been done recently utilizing thin silver films to lubricate ceramics at temperatures up to 570 °C (refs. 3 to 5). In the present study, a thin gold layer was chosen as a solid lubricant for high temperature applications. Gold was selected because of its chemical stability and relatively high melting temperature, 1073 °C. Unfortunately, because it is nonreactive, gold adheres poorly to ceramic substrates (ref. 6) The contact angle, Θ, which characterizes the wettability of two substances, is 140° between gold and monocrystalline alumina, indicating that gold is nonwetting on aluminum oxide (ref. 7).

This difficulty can be overcome by using a binder layer of a more reactive metal between the gold and the alumina substrate. The electronics industry typically uses a bond layer of chromium between gold electrical contacts and underlying silicon substrates (ref. 8). Although silicon lacks the mechanical strength for load bearing applications, the use of a chromium underlayer can be applied to more likely bearing substrates such as alumina. During preliminary testing described in a previous publication, it was demonstrated that the Au/Cr material combination successfully lubricated alumina at temperatures to 800 °C (ref. 9).

The current investigation was undertaken to further characterize the operating limits and performance of the Au/Cr high temperature solid lubricating system. Au/Cr sputter coated alumina disks were tested in a high temperature pin-on-disk tribometer at temperatures of 25, 250, 500, 800, and 1000 °C under normal loads ranging from 4.9 N to

49.0 N and sliding velocities from 1 to 15 m/s. Post test wear measurements and microscopy were used to analyze the specimens and help to elucidate the wear process.

EXPERIMENTAL

Materials/Specimen Preparation

Disks and pins were made from sintered α-alumina (99.4 percent pure). Complete pin and disk property and fabrication data may be found in a previous publication (ref. 10). The sputter targets of gold and chromium had purities of 99.999 and 99.95 percent respectively.

The alumina disks were ultrasonically cleaned for 15 min in acetone followed by 15 min in methanol to remove any surface contamination. Immediately prior to the coating deposition, the Al_2O_3 disks were backsputter etched with argon to remove adsorbed surface contaminants. The disks were then RF Magnetron sputter coated with 1000 Å of Cr, and finally overcoated with 2 μm of Au. Details of the sputtering technique have been described previously (ref. 9).

During the preliminary testing, a Cr thickness of 500 Å was used. At high temperature (800 °C) this coating suffered sporadic delamination failures (ref. 9). Doubling the Cr layer to 1000 Å eliminated the delamination failures. The 2μm Au coating thickness was not optimized but was chosen based upon previous investigations by others such as Maillat et al. (ref. 5), who, in their investigation of silver films found that a 2μm thickness was optimal. Future research may be conducted to investigate film thickness effects but this is beyond the scope of the current study.

After sputter coating, the disk specimens were heat treated in air for 6 hr at 800 °C. This heat treatment, developed during the initial test program, was found to produce a more tenacious coating. EDS analysis of post-heat treated disk specimens revealed Cr diffusion through the 2 μm Au layer. In addition, the disk surfaces appeared gray-green after heat treatment, consistent with chromium oxide (ref. 11).

A 2.54 cm radius of curvature was machined on 9.55 mm diameter alumina rods to act as pins during the tribo-testing. The pin surfaces were then polished to a finish of 0.2 μm rms. The pins were prepared for testing by rinsing with ethyl alcohol, scrubbing with levigated alumina powder and water, rinsing with de-ionized water, and finally drying with compressed air.

Experimental Apparatus/Method

Pin on disk testing was performed in a high temperature tribometer (ref. 12) shown schematically in figure 1. Both the pin and disk were enclosed in a resistance-heated furnace. Each alumina disk was aligned in the tribometer prior to tribo-testing to reduce total indicated run-out to less than 12.2 μm. The pin was loaded on the rotating disk surface using dead-weights. Rotational speed was varied from 370 to 5640 rpm, corresponding to nominal tangential velocities of 1 to 15 m/s. The majority of tests were run for a period of 1 hr and at 1 m/s sliding velocity in order to make comparisons to earlier data. Testing at higher speeds (>1 m/s) was limited to 30 min duration due to rapid coating wear through and subsequent substrate disk wear. A computer acquisition system recorded the load, friction force, temperature and speed at 30 sec intervals throughout each test. In addition, friction force and load were recorded continuously on a strip chart recorder.

After tribo-testing, a stylus profilometer was utilized to measure the depth of the disk wear scar at four locations on each wear track. Pin wear volume was calculated based on pin wear scar diameter as measured with an optical microscope. Pin wear factors were then determined using the equation:

$$k = V/LD \ (mm^3/Nm)$$

where

V = wear volume in mm^3
L = applied load in Newtons
D = total sliding distance in meters

Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) analyses were performed on selected pin and disk specimens to further elucidate the wear process.

RESULTS AND DISCUSSION

Effect of Test Temperature:

Compared to unlubricated alumina in sliding contact, the use of the Au/Cr film reduced friction by 30 to 50 percent and pin wear by one to two orders of magnitude over the entire temperature range tested (from 25 to 1000 °C). Test temperature significantly affected friction but produced no obvious effect on wear. As temperature was increased, the friction coefficient decreased by almost 50 percent averaging 0.49 at 25 °C and 0.27 at 1000 °C, as shown in Table I and figure 2.

The observed decrease in friction coefficient is consistent with the decrease in hardness that occurs in gold as the temperature is increased. Figure 3 shows both the friction coefficient of the Au/Cr film and Vicker's hardness of Au plotted as a function of temperature. Since hardness is a measure of the ability of a material to resist permanent deformation, it follows that the shear strength of the gold lubricating layer and hence the friction would decrease as hardness decreased. The reduction in friction coefficient with increased temperature is, of course, limited by the melting point of the gold lubricant layer (≈ 1073 °C). Based upon these results, it appears plausible that the test temperature is affecting friction through a shear strength reduction mechanism. Test temperatures in excess of 1000 °C were not attainable because the Al_2O_3 disk specimens repeatedly failed by fracture.

Pin wear, given in Table II, showed no consistent trends with changing test temperature. Differences in the average pin wear factors measured at various test temperatures were generally within data scatter. For example, at a load of 4.9 N, the pin wear factor at 1000 °C was $17.4 \pm 13 \times 10^{-8}$ mm^3/Nm while at 25 °C it was $7.0 \pm 0.8 \times 10^{-8}$ mm^3/Nm indicating that although the average values differed, they were still within the scatter band of one standard deviation. The similarity of the pin wear factors may be due, in part, to the fact that compared to the alumina pin surface, the Au/Cr coating is very soft at all of the test temperatures and thus has a small effect on pin wear.

Disk wear is not reported in this study. Preliminary testing suggested that disk wear measurements for Au/Cr coated specimens may not be meaningful (ref. 9). The primary function of a thin film lubricant is to protect the substrate and reduce friction. Since the tests conducted here generally ended before the films completely wore through all of the disk wear depths were comparable, ≈2 μm. Therefore, direct comparison of Au/Cr lubricated disk wear to unlubricated alumina disk wear was not made. For characterizing the overall performance of the lubricant coating, only friction and counterface wear are considered.

Effect of Load

The effect of load on friction is given by the data in Table I and is illustrated graphically in figure 4. The data show that, within scatter, load has little or no effect on friction. At room temperature, 25 °C, a tenfold increase in load, from 4.9 N to 49 N, produced only a 15 percent increase in friction coefficient, from 0.49 to 0.57. Similar behavior was observed at 800 °; a fivefold increase in load, from 4.9 N to 29.4 N, resulted in a 30 percent increase in friction coefficient, from 0.30 to 0.39. At all loads, 4.9 to 49 N, the friction coefficients of coated disks remained 30 to 50 percent less than those of uncoated disks tested under a 4.9 N load.

Test load had little or no effect on pin wear at lower test loads as shown in Table II. The pin wear factors remained essentially constant as load was increased up to 19.6N, but at higher loads, pin wear factors increased. For example, at 25 °C, the pin wear factor was approximately 8×10^{-8} mm^3/Nm for loads between 4. 9 and 19.6 N. The wear factor increased to approximately 26×10^{-8} mm^3/Nm at 29.4 N and further increased to 47×10^{-8} mm^3/Nm at 49 N. Despite this increase, the pin wear factors produced under even the most severe load conditions (49 N) with coated disks remained an order of magnitude lower than those produced under the mildest load conditions (4.9 N) on uncoated disks.

SEM and EDS analyses were conducted on disk wear tracks after testing to identify possible wear mechanisms and to better understand the wear process. Figure 5(a to d) are electron micrographs at increasing magnifications, of a wear track after sliding under a 9.8 N load. Three types of morphological regions characterized the wear track surface: (1) large predominant regions of intact Au/Cr which provide continued lubrication; (2) small localized regions showing gradual thinning out or wearing through of the Au/Cr film; and (3) small faceted surface regions which appear to be grain pullout of the Al_2O_3 substrate.

EDS analysis, shown in figure 6(a) of the predominant region detects the presence of Au and Cr indicating that the film is indeed intact. Where the Au/Cr layer has been removed a faceted Al_2O_3 substrate is revealed suggesting a grain pullout mechanism. This is shown by callout in figure 5(d). In other adjacent areas the coating appears to be simply wearing through, suggestive of a more gradual wear mechanism as also shown in figure 5(d). The corresponding EDS analyses, given in figure 6(b), show that these areas are largely depleted of gold but still contain chromium, further suggesting a gradual wear process.

Thus, it appears that even after substantial sliding has taken place, the Au/Cr film continues to provide lubrication. Furthermore, the wear process is typified by gradual film wear not catastrophic film delamination or failure.

Effect of Velocity

The effect of sliding velocity on friction and wear is given in Tables III and IV respectively. The effect on friction is also illustrated graphically in figure 7. At 25 °C, friction was not affected by sliding velocity. This may be due to the high thermal conductivity of the gold film which prevented significant thermal heating (and temperature rise) and softening of the film which might have resulted in lower fiction. At 500 and 800 °C, however, increases in sliding velocity caused measurable decreases in friction of approximately 50 percent. In these cases, the elevated ambient temperature may have softened the film to the point where additional frictional heating, due to higher sliding velocities, caused further shear strength reduction resulting in reduced friction. This appears to be a plausible explanation since the hardness of Au is sharply reduced at about 500 °C as shown in figure 3.

Despite the reduction in friction at elevated temperatures and sliding velocity, coating wear life was significantly reduced at velocities higher than 1m/s. In fact, the test time had to be reduced from 60 to 30 min to minimize disk damage. The measured wear lives (in total sliding at distance) at velocities in excess of 1 m/s were substantially lower than those routinely achieved during low speed sliding. Pin wear factors increased during high speed testing, as shown in Table IV although no general trends were observed. The results of these tests suggest that, under these sliding conditions, the velocity must be 1 m/s or less to ensure prolonged life.

CONCLUDING REMARKS

At 800 °C extended life was achievable at a load of 4.9 N with a sliding velocity of 1 m/s; failure did not occur even after sliding for 9 hr (32 km). Although it was not possible to quantify coating life exactly, due to the large number of tests and samples that would have been required, the service envelope of the Au/Cr solid lubricant can be quantified as follows: The Au/Cr coating reduces friction and wear across the wide temperature range of 25 to 1000 °C. Temperature had a dramatic effect on friction. Increasing the temperature from 25 to 1000 °C resulted in friction reduction of 50 to 60 percent. No definite trends of wear with temperature were observed. Coating life was significantly reduced and pin wear increased when the load exceeded 19.6 N or the sliding velocity was raised above 1 m/s. Even under the most severe sliding conditions, i.e., high load and temperature the wear mechanism was not catastrophic delamination, but rather a localized failure of the protective layer. Based upon the results of these tests it appears that the Au/Cr film can provide adequate lubrication to moderately loaded low speed sliding contacts at temperatures as high as 1000 °C.

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TABLE I.—FRICTION COEFFICIENT SUMMARY
(Test conditions: 1 m/s sliding velocity and air atmosphere, 60 to 120 min test)

°C	Uncoated disks	Au/Cr Sputter coated disks					
	4.9 N	4.9 N	7.4 N	9.8 N	19.6 N	29.4 N	49.0 N
25	0.85±.03	0.49±.06	(A)	0.52±.03	0.52±.08	0.58±.09	0.57±.04
250	(A)	(A)	(A)	0.41±.03	0.43±.04	0.49±.04	(A)
500	0.69±.05	0.34±.05	0.32±.02	0.49±.06	(A)	(A)	(A)
800	0.76±.02	0.30±.02	0.31±.02	0.35±.05	0.28±.04	0.39±.06	(A)
1000	0.67±.16[B]	0.27±.02	(A)	0.4±.13	(A)	(A)	(A)

Notes: Values with no error indicated are based on single data points, all others represent multiple (typically 6) data points with one standard deviation uncertainty.
[A]Test not run.
[B]9.8 N load.

TABLE II.—LOW SPEED PIN WEAR FACTOR SUMMARY ($mm^3/N\text{-}m*10^{-8}$)
(Test conditions: 1 m/s sliding velocity and air atmosphere, 60 to 120 min test)

°C	Uncoated disks	Au/Cr Sputter coated disks					
	4.9 N	4.9 N	7.4 N	9.8 N	19.6 N	29.4 N	49.0 N
25	232±7	7.0±.8	(A)	8.1	7.8	25.6±16	46.8±18
250	(A)	(A)	(A)	9.3±3.9	13.5	19.3	(A)
500	1400±150	9.5	4.2±.7	4.8±1.2	(A)	(A)	(A)
800	450±50	2.6±.8	6.3±4.1	4.8±1.7	11.8±10	72	(A)
1000	32±3[B]	17.4±13	(A)	0.4±.13	(A)	(A)	(A)

Notes: Values with no error indicated are based on single data points, all others represent multiple (typically 6) data points with one standard deviation uncertainty.
[A]Test not run.
[B]9.8 N load.

TABLE III.—HIGH SPEED FRICTION COEFFICIENT SUMMARY
(Test conditions: 4.9 N load, air atmosphere, 30 min test duration)

°C	Uncoated disks	Au/Cr Sputter coated disks			
	1 m/s	1 m/s	5 m/s	10 m/s	15 m/s
25	0.85±.03	0.49±.06[A]	0.46	0.54	0.49
500	0.69±.05	0.346±.05	0.41	0.38	0.31
800	0.76±.02	0.30±.02	0.31	0.26	0.22

Notes: Values with no error indicated are based on single data points, all others repreent multiple (typically 6) data points with one standard deviation uncertainty.
[A]60 min test.

TABLE IV.—HIGH SPEED PIN WEAR FACTOR SUMMARY ($mm^3/N\text{-}m*10^{-8}$)
(Test conditions: 4.9 N load, air atmosphere, 30 min test duration)

°C	Uncoated disks	Au/Cr Sputter coated disks			
	1 m/s	1 m/s	5 m/s	10 m/s	15 m/s
25	232±7	7.0±.8[A]	18.9	488	998
500	1400±150	9.5	891	756	123
800	450±50	2.6±.8	637	1250	1070

Notes: Values with no error indicated are based on single data points, all others represent multiple (typically 6) data points with one standard deviation uncertainty.
[A]60 min test.

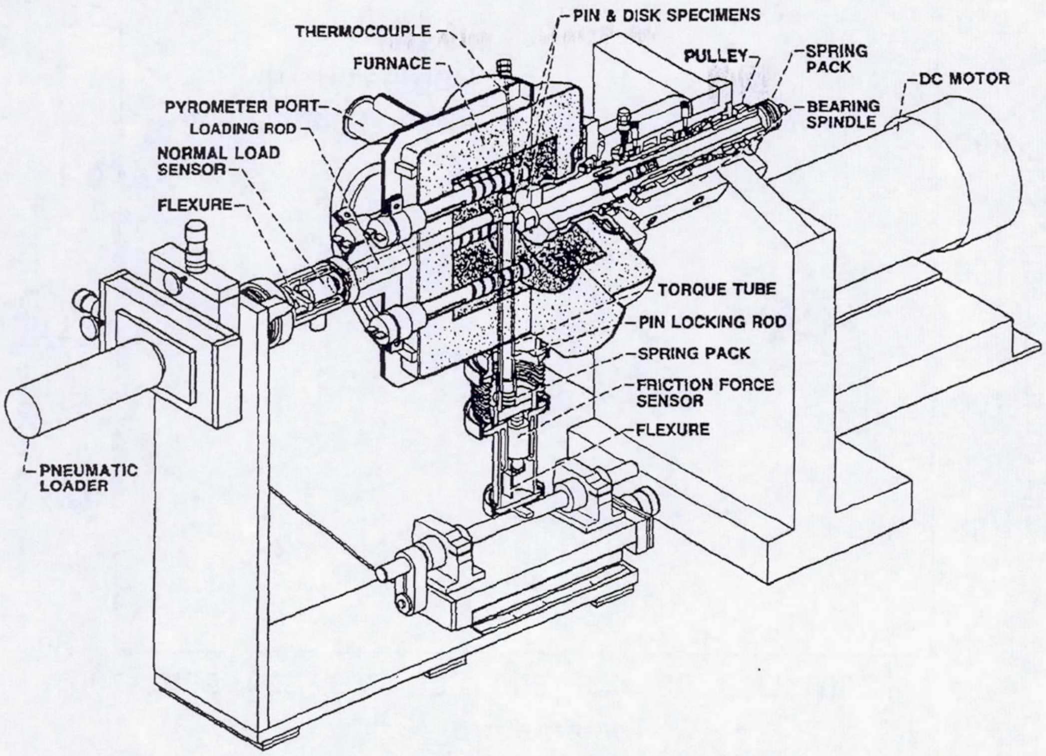


Figure 1.—High-temperature pin on disk tribometer.

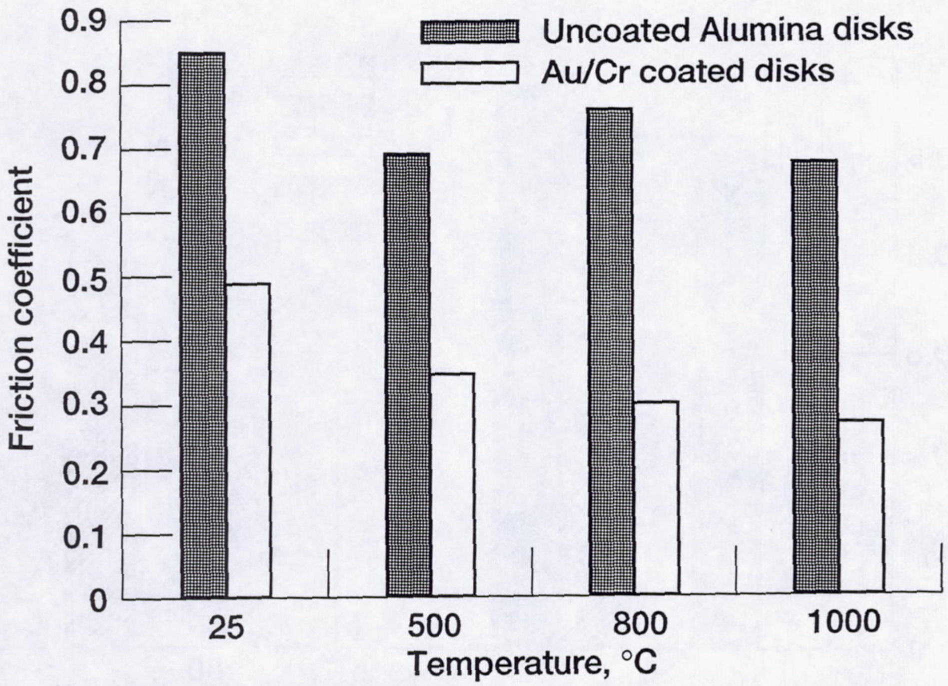


Figure 2.—Friction coefficient at various test temperatures for both Au/Cr coated and uncoated Al_2O_3 disks (4.9 N load, 1 m/s sliding velocity, air atmosphere).

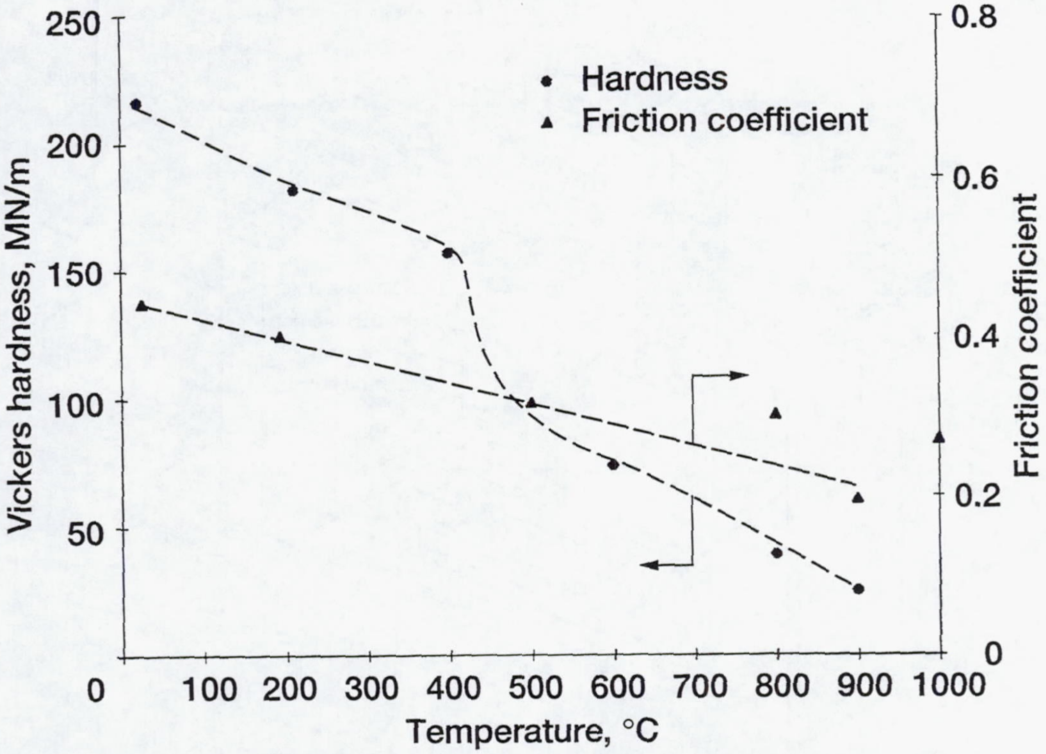


Figure 3.—Vickers hardness of gold and Au/Cr friction coefficient as a function of temperature (hardness data from reference 13).

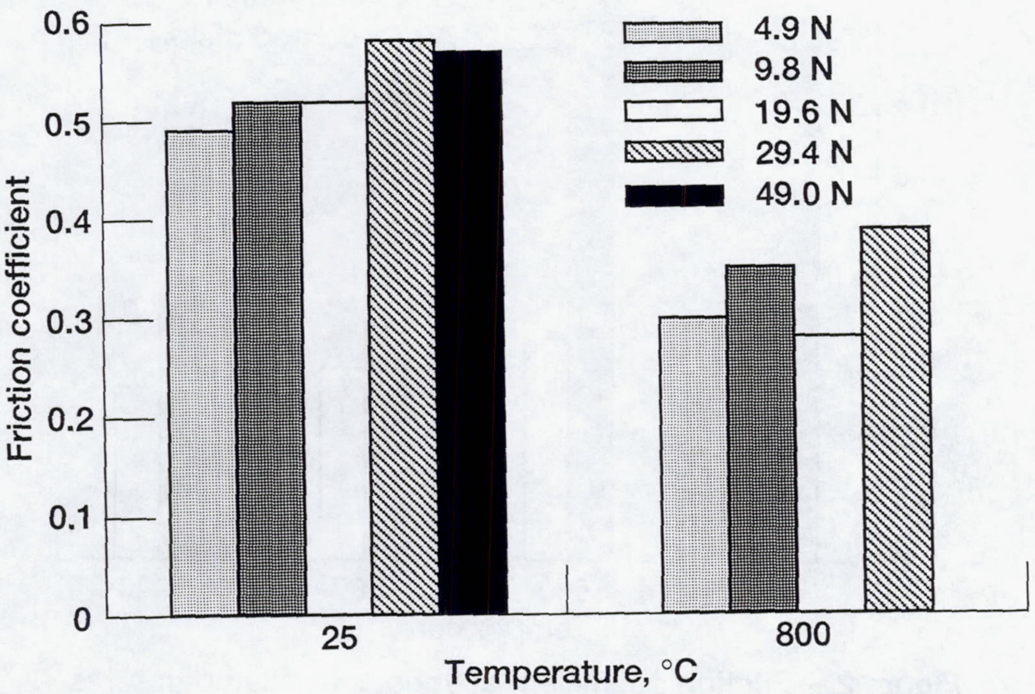


Figure 4.—Friction coefficient of Au/Cr coated alumina as a function of load at 25 and 800 °C (1 m/s sliding velocity, air atmosphere).

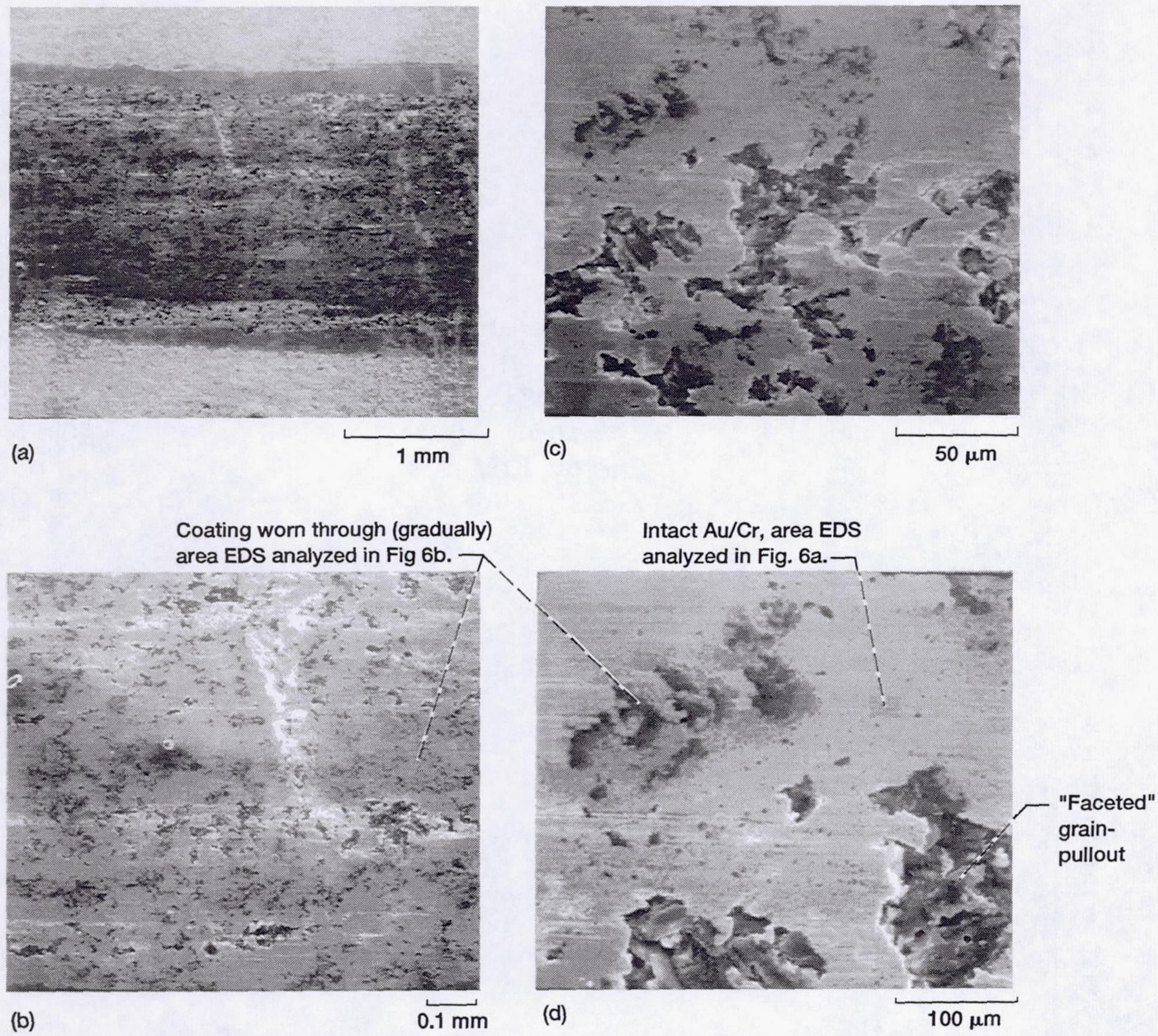
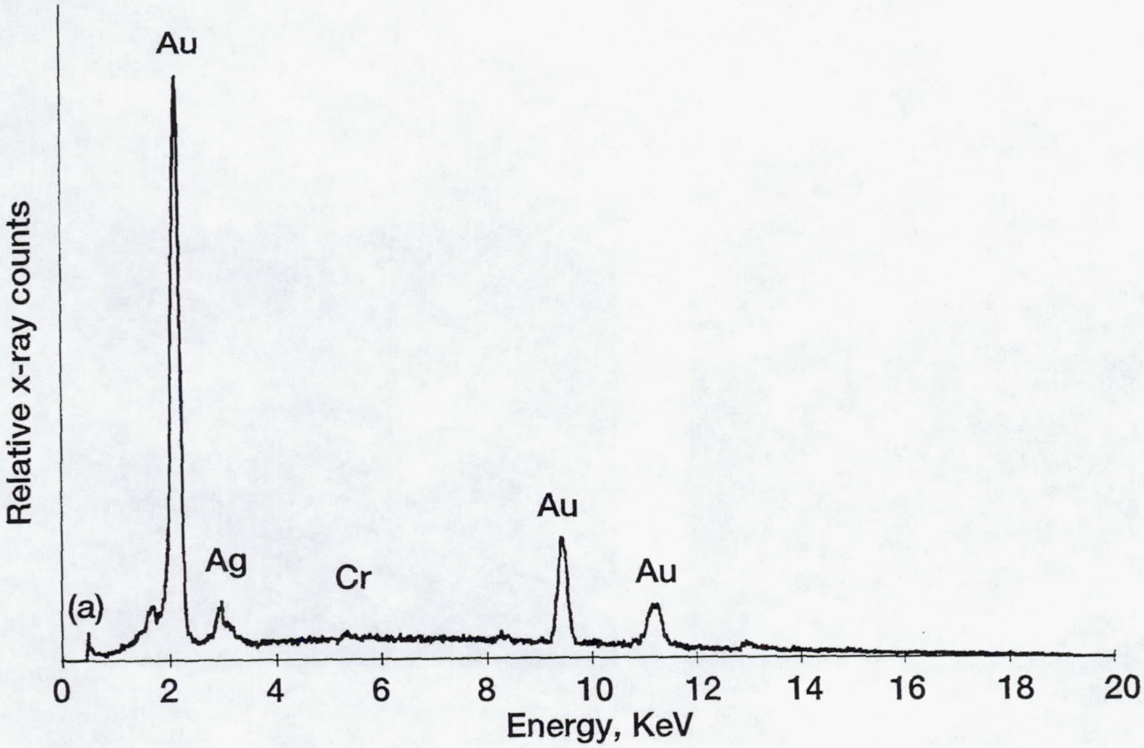


Figure 5.—Electron photomicrographs of Au/Cr wear track at various magnifications. Test conditions: 9.8 N load, 1 m/s sliding velocity, 250 °C, air atmosphere.



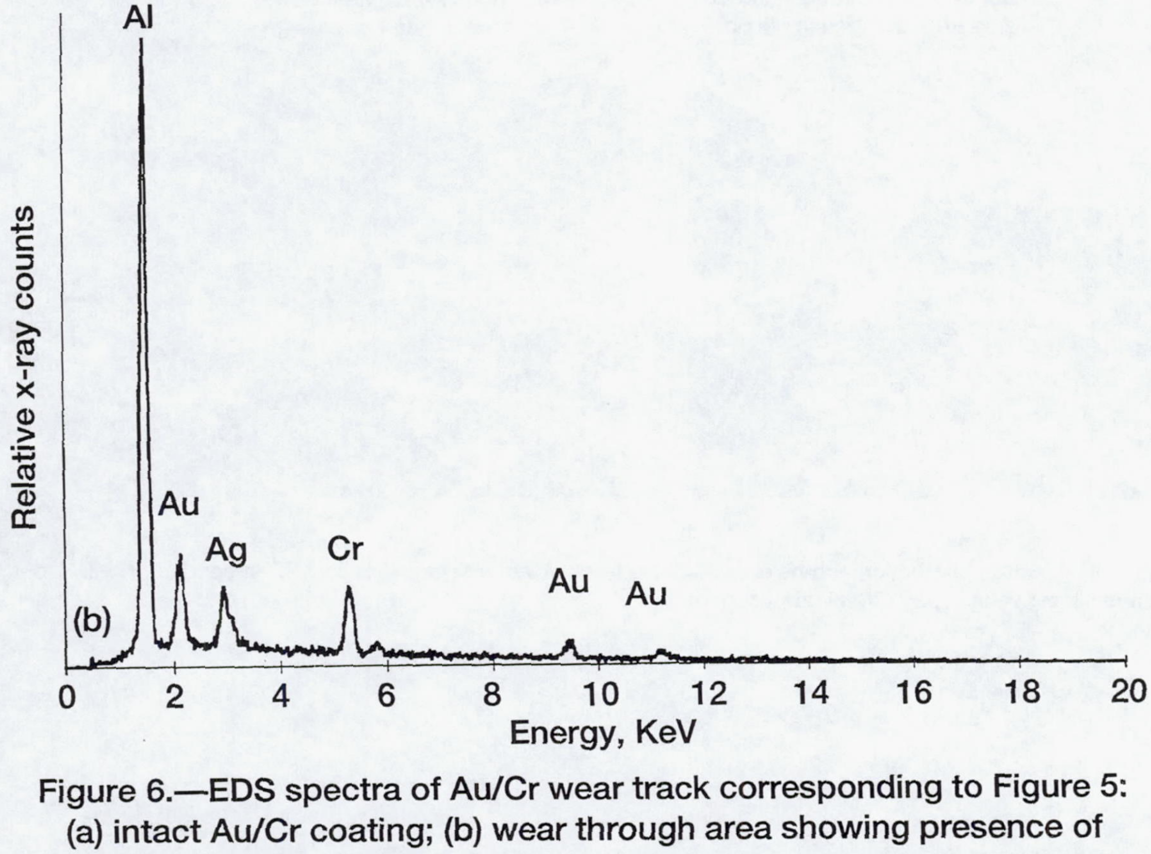


Figure 6.—EDS spectra of Au/Cr wear track corresponding to Figure 5: (a) intact Au/Cr coating; (b) wear through area showing presence of Cr and Al_2O_3 (Al peak) substrate (Ag peak from overlay coating used to prepare specimen for EDS analyses).

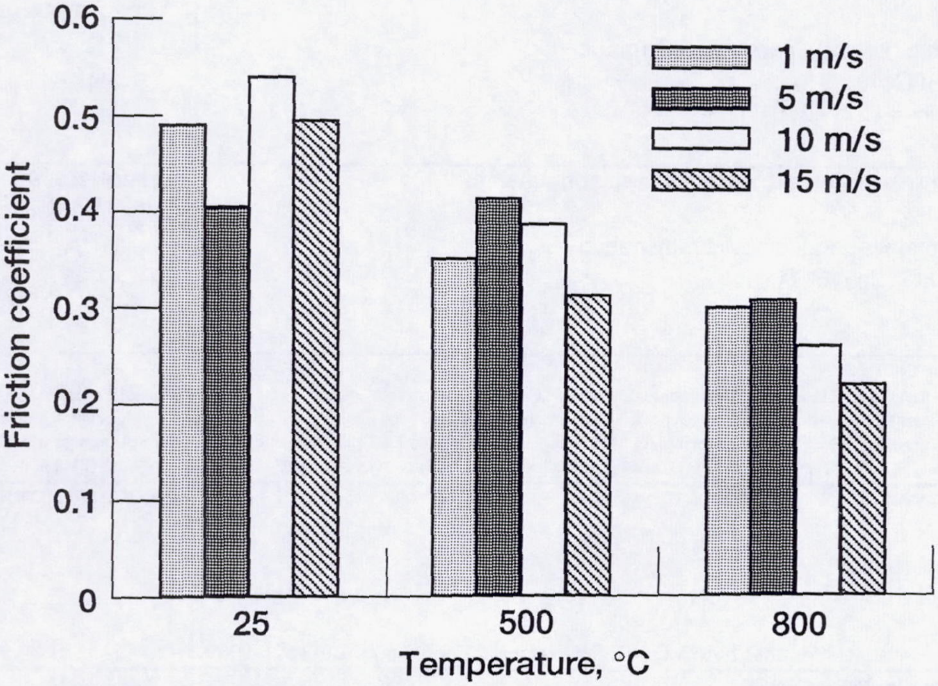


Figure 7.—Friction coefficient as a function of temperature and sliding velocity (4.9 N load, air atmosphere).

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

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