

ANL/ET/CP--89828
CONF-970429--2

SOLID/LIQUID LUBRICATION OF CERAMICS AT ELEVATED TEMPERATURES*

A. Erdemir, R. A. Erck, and G. R. Fenske
Energy Technology Division
Argonne National Laboratory
Argonne, IL 60439

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H. Hong
The Lubrizol Corporation
Wickliffe, OH 44092

April 1996

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MASTER

For presentation at 11th International Conference on Wear of Materials, San Diego, CA, April 21-24, 1997.

*Work supported by the Tribology Program, Office of Transportation Technologies, U.S. Department of Energy, under Contract W-31-109-Eng-38.

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Energy Technology Division
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ABSTRACT

This study investigates the effect of solid and liquid lubrication on friction and wear performance of silicon nitride (Si_3N_4) and cast iron. The solid lubricant was a thin silver film ($\approx 2 \mu\text{m}$ thick) produced on Si_3N_4 by ion-beam-assisted deposition. A high-temperature polyol-ester-base synthetic oil served as the liquid lubricant. Friction and wear tests were performed with pin-on-disk and oscillating-slider wear test machines at temperatures up to 300°C . Without the silver films, the friction coefficients of $\text{Si}_3\text{N}_4/\text{Si}_3\text{N}_4$ test pairs were 0.05 to 0.14, and the average wear rates of Si_3N_4 pins were $\approx 5 \times 10^{-8} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$. The friction coefficients of $\text{Si}_3\text{N}_4/\text{cast iron}$ test pairs ranged from 0.08 to 0.11, depending on test temperature. The average specific wear rates of cast iron pins were $\approx 3 \times 10^{-7} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$. However, simultaneous use of the solid-lubricant silver and synthetic oil on the sliding surfaces reduced friction coefficients to 0.02 to 0.08. Moreover, the wear of Si_3N_4 pins and silver-coated Si_3N_4 disks was so low that it was difficult to assess by a surface profilometer. The wear rates of cast iron pins were $\approx 7 \times 10^{-9} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$ up to 250°C , but showed a tendency to increase slightly at much higher temperatures. In general, the test results demonstrated that the solid/liquid lubrication of ceramic and/or metallic components is both feasible and effective in controlling friction and wear.

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INTRODUCTION

Advanced structural ceramics (e.g., silicon nitride (Si_3N_4), silicon carbide (SiC), zirconia (ZrO_2), and aluminum titanate ($\text{Al}_2\text{O}_3\cdot\text{TiO}_2$) etc.) combine excellent thermal and chemical stability with high mechanical strength, making these materials good prospects for demanding tribological applications [1-7]. Experimental prototypes of engine parts (e.g., piston pins, intake and exhaust valves, camshafts, cam lobes, roller followers, high-temperature seals, etc.) have already been fabricated from Si_3N_4 in recent years and tested for their tribological performance and reliability [2,4-6]. Furthermore, ceramic coatings (e.g., Cr_2O_3 , ZrO_2 , CrN , $\text{Al}_2\text{O}_3\text{-ZrO}_2$) and cermets (e.g., Ni or Co base- Cr_3C_2 , - TiC , - TaC , and - WC) have also been contemplated for advanced engine uses, and their potential usefulness has recently been demonstrated under a wide range of test conditions [8,9].

Despite their attractive mechanical, thermal, and chemical properties, most ceramics and their coatings are not expected to replace traditional iron-base alloys in the near future [10]. This is because the long-term reliability of most ceramic components in actual applications is not well established. Ceramics are inherently brittle and their fabrication into useful shapes may be rather expensive. Also, recent field and laboratory tests have revealed that the tribological performance and reliability of most ceramics are not that impressive, especially under dry or marginally lubricated sliding conditions and at high temperatures [10-16]. The present consensus is that without the development of effective lubricants and new lubrication concepts, large-scale utilization of ceramic materials and their coatings for friction and wear applications appears highly unlikely [2,9].

In an effort to reduce friction and wear, a series of liquid [1,3,17-20] and solid lubricants [21-23] have been developed and tried on ceramic components. Initial results with synthetic lubricants appeared highly promising [18,24], but the test results from solid-lubricated ceramics were somewhat disappointing. A finite lifetime and gradual thermal or chemical degradation of these lubricants appeared to be serious drawbacks for their use in actual applications [21,25]. However, recent laboratory tests have proved that combining liquid and solid lubricants on sliding ceramic surfaces may

be effective in reducing friction and wear of ceramic materials [19,26-28].

This study investigates the effects of thin silver films and a high-temperature synthetic oil on the tribological performance of Si_3N_4 at temperatures up to 300°C . Specifically, both the solid-lubricant silver film and the polyol-ester-base synthetic oil were used simultaneously on the sliding surfaces of Si_3N_4 and cast iron to reduce friction and increase wear resistance. Test conditions were selected to create a boundary lubrication regime, so that the effectiveness of silver as a backup lubricant could be clarified, especially at elevated test temperatures where liquid lubricants lose much of their viscosity and, thus, their load-bearing capacity.

EXPERIMENTAL PROCEDURES

Test Material

The pin and flat specimens used in this study were fabricated from Si_3N_4 ceramic. Further details of the structure and mechanical properties of the test material can be found in Refs. 24, 27, and 28. Disk specimens, 75 mm in diameter and 8 mm thick, were surface-finished by diamond-wheel grinding to a roughness of $0.2 \pm 0.02 \mu\text{m}$ centerline average (CLA). The rectangular Si_3N_4 flats used in the reciprocating wear tester were 3.2 mm thick, 76.2 mm long, and 15.9 mm wide.

The counterface pins were made of Si_3N_4 and gray cast iron. The Si_3N_4 pins were 8 mm in diameter and 15 mm long. One end of each pin was hemispherically finished to a radius of curvature of ≈ 127 mm, with a surface roughness of $\approx 0.03 \mu\text{m}$ CLA. The cast iron pins were 25.4 mm long and 4.8 mm in diameter and had a hemispherically finished tip with a radius of 101.6 mm. All specimens were ultrasonically cleaned before ion-beam-deposition or wear testing by following the procedures described in Refs. 24 and 28.

Silver Deposition

Thin silver films, 1.5 to 2 μm thick, were deposited on Si_3N_4 substrates at room temperature in an ion-beam-deposition system equipped with an electron-beam-heated evaporation source. A mixture of oxygen and argon (10 O_2 /4 Ar ratio) was fed through a hot-cathode Kaufman-type ion source to create an ion flux composed of the ions of O_2 and Ar. This ion flux was then used to bombard and remove the adsorbed contaminants from the surfaces of Si_3N_4 substrates for a period of 5 to 10 min at an acceleration voltage of 0.5 keV and an ion current density of 0.1 mA/cm^2 . Next, the ion beam was turned off, the gas flow through the ion source was stopped, and a thin niobium or titanium bond layer (20 to 50 nm thick) was deposited on the Si_3N_4 substrate, mainly to promote adhesion. Finally, silver was thermally evaporated from the electron-beam-heated evaporation source and deposited on the surface of substrates. The thickness of the growing silver film was controlled with the aid of a quartz-crystal rate monitor. A schematic depiction of the deposition system is shown in Fig. 1. Further details of this system and resultant films can be found in Refs. 27 and 29.

Friction and Wear Tests

Friction and wear were measured with the pairs of Si_3N_4 / Si_3N_4 and cast iron/ Si_3N_4 with and without silver film, in a pin-on-disk machine and a reciprocating wear tester under lubricated sliding conditions. The liquid lubricant used in this study was a formulated polyol-ester-base oil. A recent paper by Hong and Stadnyk provides details on this lubricant and its characteristics at temperatures up to 400°C [24]. Some thermal and physical properties of this oil are given in Table 1.

Pin-on-disk tests were performed at 0.1 m/s sliding velocity, in open air of 30 to 40% relative humidity and at room temperature, 100, 200, and 300°C. The Si_3N_4 disks were firmly clamped on a lubricant cup filled with the oil. The dead weight applied to the pin specimens was 50 N, which created an initial mean Hertzian contact pressure of ≈ 166 MPa between the Si_3N_4 pin and the Si_3N_4 flat. Because of the increasing wear-scar diameter, nominal bearing pressures fell to a fraction of this initial value by the end of the tests. The final nominal pressures for each test pair are noted in parentheses in a graph

showing the wear data. Frictional force was monitored by a load cell and recorded on chart papers, as well as on diskettes by a data acquisition system. In a few cases, the ambient temperature was allowed to increase steadily up to 300°C while the sliding test continued and then gradually returned to room temperature.

The reciprocating pin-on-flat tests were performed with cast-iron/ Si_3N_4 pairs with and without a silver film under a 27.5-N load that created an initial mean Hertzian contact pressure of ≈ 149 MPa between the cast iron pin and the Si_3N_4 flat. The average sliding velocity was ≈ 0.1 m/s and the sliding distance was 200 m. These experiments were carried out at bulk specimen temperatures of 23, 250, and 300°C. The lubricant was heated to test temperature before being injected onto the sliding surface. It was fed continuously to the lubricant cup through an oil inlet and drained through an outlet. In an earlier study, friction and wear coefficients of cast iron/ Si_3N_4 test pairs remained essentially constant during reciprocating tests at temperatures up to 250°C [24]. Therefore, This study focused on the higher test temperatures (250° and 300°) which would create much more severe test conditions. Further details of the reciprocating wear test machine and procedures are given in Ref. 24.

To check the accuracy and reproducibility of friction test results, two or three tests were run at a specific temperature. Results were quite reproducible, and deviations from the mean friction values were $\pm 10\%$ to $\pm 20\%$, depending on test pairs and configuration.

RESULTS

Friction

Figure 2 shows the friction coefficients of Si_3N_4 pins during sliding against the uncoated and silver-coated Si_3N_4 disk at room temperature (23°C) under lubricated sliding contact. The friction coefficient of the test pair without a silver film is ≈ 0.14 , suggesting that a boundary lubrication regime was established between the pair. The initial friction coefficient of the Si_3N_4 /silver-coated Si_3N_4 test pair is ≈ 0.1 , but tends to decrease steadily and stabilize at ≈ 0.07 . Note that this value is a factor of 2 lower

than that observed on the $\text{Si}_3\text{N}_4/\text{Si}_3\text{N}_4$ test pair.

The friction coefficients of the Si_3N_4 pins during sliding against Si_3N_4 and silver-coated Si_3N_4 at 100°C are shown in Fig. 3. Without a silver film, the friction coefficient of test pairs is ≈ 0.13 at steady state. However, with a silver film on Si_3N_4 disk, the initial friction coefficient is ≈ 0.07 , but decreases to ≈ 0.02 after a sliding distance of ≈ 130 m and remains essentially constant afterward.

Figure 4 shows the friction coefficients of $\text{Si}_3\text{N}_4/\text{Si}_3\text{N}_4$ and $\text{Si}_3\text{N}_4/\text{silver-coated } \text{Si}_3\text{N}_4$ test pairs at 200°C . Without the silver film, the friction coefficient is ≈ 0.1 at steady-state, significantly lower than those recorded at 23 and 100°C . The friction coefficient of the Si_3N_4 pin sliding against the silver-coated Si_3N_4 disk is, however, much lower, ≈ 0.03 .

Figure 5 shows the friction coefficients of the Si_3N_4 pins during sliding against Si_3N_4 and silver-coated- Si_3N_4 disks at 300°C . Without the silver coating, the friction coefficient of test pairs is initially ≈ 0.09 , but decreases to 0.03 to 0.04 after a sliding distance of ≈ 30 m. The friction coefficient of the Si_3N_4 pin sliding against the silver-coated Si_3N_4 disk is initially below 0.01 but increases to ≈ 0.01 as sliding distance increases, then remains constant for the remainder of the sliding test. After these tests, a layer of black deposit had formed on the sliding surfaces of both the uncoated and silver-coated samples.

Figure 6 shows the friction coefficients of $\text{Si}_3\text{N}_4/\text{Si}_3\text{N}_4$ and $\text{Si}_3\text{N}_4/\text{Ag-coated } \text{Si}_3\text{N}_4$ test pairs as a function of continuously increasing temperature. In general, during the heating cycle of the sliding test, the friction coefficients of both pairs decreased with increasing temperature. However, during the cooling cycle, they showed a reverse trend. Overall, these friction coefficients, obtained as a function of continuously increasing temperatures, were essentially consistent with those recorded at constant temperatures (see Figs. 2-5). Test results at both fixed and continuously increasing temperatures generally demonstrated that the friction coefficients of pairs without a silver film were markedly higher than those of pairs with a silver film.

Figure 7 shows the range of friction coefficients of cast iron/Ag-coated Si_3N_4 test pairs as a function

of increasing temperature. Friction coefficients of these pairs were distinctly lower than those of pairs without a silver film when tested at room temperature and 250°C. However, at 300°C, a slight increase in friction was noticed.

Wear

Figure 8 shows wear rates of the Si_3N_4 pins during sliding against uncoated and silver-coated Si_3N_4 disks at various test temperatures. The average specific wear rate of pins slid against the uncoated disks is $5.5 \times 10^{-8} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$ at room temperature and increases to $7.3 \times 10^{-8} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$ at 200°C. At 300°C, the wear rate of pins was somewhat comparable to that observed at room temperature.

The wear of Si_3N_4 pins sliding against the silver-coated Si_3N_4 disks was so slight that it was difficult to measure. A surface profilometer at vertical magnifications up to 50,000x did not indicate a measurable wear scar on Si_3N_4 pins after they were slid against the silver-coated Si_3N_4 disks. Some of the Si_3N_4 pins appeared to have a small wear scar, especially after tests at 200 and 300°C. However, after the oil and some silver that was transferred from the disk side were removed, no measurable wear could be verified on these Si_3N_4 pins. The wear of silver-coated disks was also very difficult to assess. The surface profilometer could not detect measurable wear losses on the Si_3N_4 substrates. The only wear track formed was due to the easy deformation of soft silver films of $\approx 2\text{-}\mu\text{m}$ thickness, as is evident in an SEM micrograph and a 3-D surface profile map in Fig. 9. The electron microscopy inspection revealed that the silver films were largely intact on the wear tracks of Si_3N_4 disks. They were flattened to provide a very smooth surface finish as shown in Fig. 9. Disks tested at 300°C appeared to have been covered with a layer of oil that was viscous in nature and quite adherent to the sliding surfaces.

Figure 10 shows the average specific wear rates of cast iron/ Si_3N_4 and cast iron/Ag-coated Si_3N_4 as a function of temperature. The wear rates of cast iron pins sliding against Ag-coated Si_3N_4 flats were significantly lower than those of pins slid against uncoated flats during tests at room temperature and 250°C. However, at 300°C, rates exceeded the wear rates of pins slid against the uncoated flats.

Figure 11 shows the surface topography of cast iron pins slid against uncoated and Ag-coated Si_3N_4 flats. The cast iron tested against Ag-coated Si_3N_4 at 250°C showed a smooth wear scar containing some Ca and S. Some transferred silver flakes were also observed on this scar (see Fig. 11a). The cast iron that was rubbed against Si_3N_4 at 250°C showed a much larger wear scar with a smooth surface finish. EDS analysis of spots A and B on this wear scar revealed the presence of some sulfur on these rubbing areas. The cast iron pins tested at 300°C exhibited smooth wear scars, and the EDS survey of these scars confirmed the presence of some sulfur. The cast iron tested against Ag-coated Si_3N_4 at 300°C showed a mixture of smooth and reacted areas. EDS analysis of this scar revealed that a thin sulfur-containing layer was present on the smooth areas (regions A and C in Fig. 11c) and a sulfur- and calcium-containing layer was on the reacted area (region B in Fig. 11c).

Visual inspection of the silver-coated Si_3N_4 revealed some discolored areas outside the wear tracks after tests at high temperatures. X-ray photoelectron spectroscopy (XPS) of these areas resulted in binding energies of 368.1 and 161.2 eV, which corresponded to Ag 3d and S 2p, respectively, in the compound Ag_2S , suggesting the formation of silver sulfide.

DISCUSSION

The results presented above indicate that combining a liquid lubricant and a solid-lubricant silver film has a beneficial synergistic effect on the friction and wear performance of sliding ceramic/ceramic and metal/ceramic interfaces. For the excellent friction-and wear-reducing capability of silver films in the presence of a synthetic oil, we propose the following explanation. Because of its low shear strength, silver deforms easily under normal and tangential forces of sliding contact and fills in the valleys between surface asperities of relatively rough (e.g., $\approx 0.2 \mu\text{m}$ CLA) Si_3N_4 surfaces. Consequently, the sliding contact surface becomes very smooth, as manifested by the three-dimensional (3-D) surface map and scanning electron microscopy (SEM) photomicrograph in Figs. 9a and 9b, respectively. At elevated test temperatures, shear deformation of silver becomes much easier, mainly because of thermal softening. As a result, the sliding-contact surfaces become much smoother. As elaborated in lubrication textbooks [30,31], a smooth surface finish imparts an increased λ ratio (the ratio of the minimum

elastohydrodynamic [EHD] film thickness [h] to the composite surface roughness of the contacting bodies, $\lambda = h/s$). When the λ ratio increases, the effectiveness of the lubricant oil also increases, as explained below.

For a fully-flooded point contact under isothermal, steady-state conditions, the λ ratio increases as the composite surface roughness (s) decreases. As a consequence, the effectiveness of the liquid lubricant improves and the friction coefficient tends to decrease. This is particularly true for cases where the λ ratio is equal to or greater than unity, which means that the lubricant film thickness is greater than the composite surface roughness of the contacting bodies; hence asperity/asperity interactions through the lubricant film are minimized. The most ideal case is a λ ratio of >3 so that the sliding surfaces are completely separated and essentially no asperity/asperity contacts occur. This lubrication regime is known as full-film lubrication, and the friction coefficients are determined by the shear rheology of the liquid lubricant.

To demonstrate the severity of asperity/asperity interactions during these sliding tests, we attempted to estimate the λ ratios for an undisturbed area and a sliding contact area of a silver-coated Si_3N_4 flat at 100°C . An SEM photomicrograph of this particular surface and its 3-D surface map are shown in Fig. 9. A surface profilometer measured an average surface roughness of $\approx 0.02 \mu\text{m}$ inside and $\approx 0.2 \mu\text{m}$ outside the wear track. From an empirical equation proposed by Hamrock and Dowson [30], we estimated the λ ratios to be ≈ 0.4 for the undisturbed (outside the track) and ≈ 0.9 for the sliding-contact areas (inside the track) of this flat. It is clear that with the use of a thin silver film, the λ ratio increased by a factor of >2 . This is mainly because of the easy shear deformation of silver film that results in a smooth surface finish.

Therefore, it is postulated that the observed decrease in the friction coefficients of all test pairs containing a silver film was primarily due to the creation of a smooth wear track that in turn led to an increase in λ ratio. Furthermore, because the silver film effectively reduced the extent of asperity/asperity contacts across the sliding interfaces and carried a significant portion of the contact stresses, it can be regarded as an effective boundary film or back-up lubricant. At 300°C , the silver film

tended to react with oil additives, particularly with sulfur (Fig. 11). XPS analysis confirmed that a thin Ag_2S film was present on the surfaces of both the friction-tested and undisturbed areas of the initial silver films. Also, after tests at 300°C , some patches of black deposits formed on the sliding surfaces of both the uncoated and silver-coated Si_3N_4 disks. It is conceivable that relatively low friction coefficients, i.e., ≈ 0.04 , of uncoated test pairs at 300°C (Figs. 5 and 6a) may have been associated with the presence of these deposit layers, essentially acting as a quasi-solid lubricant film with easy-shear capability. For the Si_3N_4 /silver-coated Si_3N_4 test pairs (Figs. 5 and 6b), the friction coefficients at 300°C were ≈ 0.01 , a much lower value than the 0.04 recorded on uncoated pairs. The significantly lower friction coefficients of silver-coated pairs at 300°C is attributed to (a) a much smoother surface finish that results from the easy deformation of silver film and (b) the formation of an Ag_2S reaction film on sliding surfaces. This film may have been acting as a low shear boundary film.

To elucidate this hypothesis further, we performed three more sliding tests at 300°C : one with a pure silver plate (0.8 mm thick) and a formulated oil, and two others with uncoated and silver-coated disks and an unformulated polyol-ester-base oil (the base stock with no additives). The results of these tests are shown in Fig. 12. The friction coefficient of an Si_3N_4 pin sliding against silver plate in formulated oil is ≈ 0.02 (Fig. 12a) and is comparable to that (i.e., 0.01) of an Si_3N_4 /silver-coated Si_3N_4 test pair tested in the same oil at 300°C (see Fig. 5). However, the friction coefficients of the Si_3N_4 pins sliding against uncoated and silver-coated Si_3N_4 disks in base-stock oil are high, i.e., ≈ 0.05 and 0.04, respectively (see Fig. 12b). These values are significantly higher than those obtained with the formulated oil.

From the results of these additional tests, it can be deduced that silver alone is not that effective in reducing the friction coefficients of test pairs. The sulfur-containing additives impart a significant reduction in the friction coefficients. These findings suggest that perhaps the formation of Ag_2S on sliding surfaces has a beneficial effect on friction. Obviously, more analytical work should be done to further elucidate the exact role of sulfur-bearing additives in reduced friction.

CONCLUSIONS

- * The simultaneous use of liquid and solid-film lubricants at sliding interfaces of ceramic/ceramic and steel/ceramic pairs can significantly reduce friction and wear.
- * The results show that when adherent-silver films are used at oil-lubricated sliding interfaces, the wear rates of both pins and flats are markedly reduced. The friction coefficients are also substantially reduced (i.e., by factors of 2 to 10).
- * Beneficial synergistic effects of silver films on the boundary-lubrication behavior of ceramic interfaces were more pronounced at elevated test temperatures than at room temperature.
- * Silver films undergo easy shear during oil-lubricated sliding contact and provide an increasingly smooth surface finish. Consequently, the λ ratio increases, leading to a more efficient lubrication regime in which asperity/asperity interactions through the lubricant film are minimized.
- * Surface analytical studies revealed that silver reacts with sulfur in formulated oil to form Ag_2S . This compound seems to act as a boundary film and help lower friction.

ACKNOWLEDGMENTS

This work was supported by the Tribology Program, Office of Transportation Technologies, U.S. Department of Energy, under Contract W-31-109-Eng-38. The authors thank the Lubrizol Corporation for permission to publish this paper. Also, the technical support of Dr. O. O. Ajayi (Argonne) and Dr. E. Badasz and Mr. N. M. Stadnyk (Lubrizol) is greatly appreciated.

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FIGURE CAPTIONS:

Figure 1. Schematic depiction of IBAD system used in this study.

Figure 2. Friction coefficient of Si_3N_4 pin during sliding against uncoated and silver-coated Si_3N_4 disks with synthetic lubricant at 23°C.

Figure 3. Friction coefficient of Si_3N_4 pin during sliding against uncoated and silver-coated Si_3N_4 disks with synthetic lubricant at 100°C.

Figure 4. Friction coefficient of Si_3N_4 pin during sliding against uncoated and silver-coated Si_3N_4 disks with synthetic lubricant at 200°C.

Figure 5. Friction coefficient of Si_3N_4 pin during sliding against uncoated and silver-coated Si_3N_4 disks with synthetic lubricant at 300°C.

Figure 6. Variation of oil-lubricated friction coefficients of Si_3N_4 pins during sliding against (a) uncoated and (b) silver-coated Si_3N_4 disks as a function of increasing temperature.

Figure 7. Variation of friction coefficients of cast iron pins during sliding against uncoated and silver-coated Si_3N_4 flats as a function of increasing temperature.

Figure 8. Average wear rates of Si_3N_4 pins during sliding against uncoated and silver-coated Si_3N_4 disks at various test temperatures. Values in parentheses indicate final nominal contact pressures (MPa). Initial mean Hertzian pressure was 166 MPa.

Figure 9. (a) Low-magnification SEM photomicrograph and (b) three-dimensional surface map of wear track formed on silver-coated Si_3N_4 disk during oil-lubricated sliding test against uncoated Si_3N_4 pin at 100°C.

Figure 10. Average wear rates of cast iron pins during sliding against uncoated and silver-coated Si_3N_4 disks at various test temperatures.

Figure 11. SEM photomicrographs of wear scars formed on cast iron pins during sliding at 250°C against (a) silver coated Si_3N_4 , (b) uncoated Si_3N_4 at 250°C; and at 300°C (c) against silver-coated Si_3N_4 and (d) uncoated Si_3N_4 .

Figure 12. Friction coefficients of Si_3N_4 pins during sliding against (a) silver plate and (b) uncoated and silver coated Si_3N_4 disks at 300°C.

Table 1. Properties of polyol-ester-base lubricant.

Property		Specific Value
Viscosity at	40°C	61.8 cSt.
	100°C	9.6 cSt.
Pour Point		-51°C
Flash Point		282°C

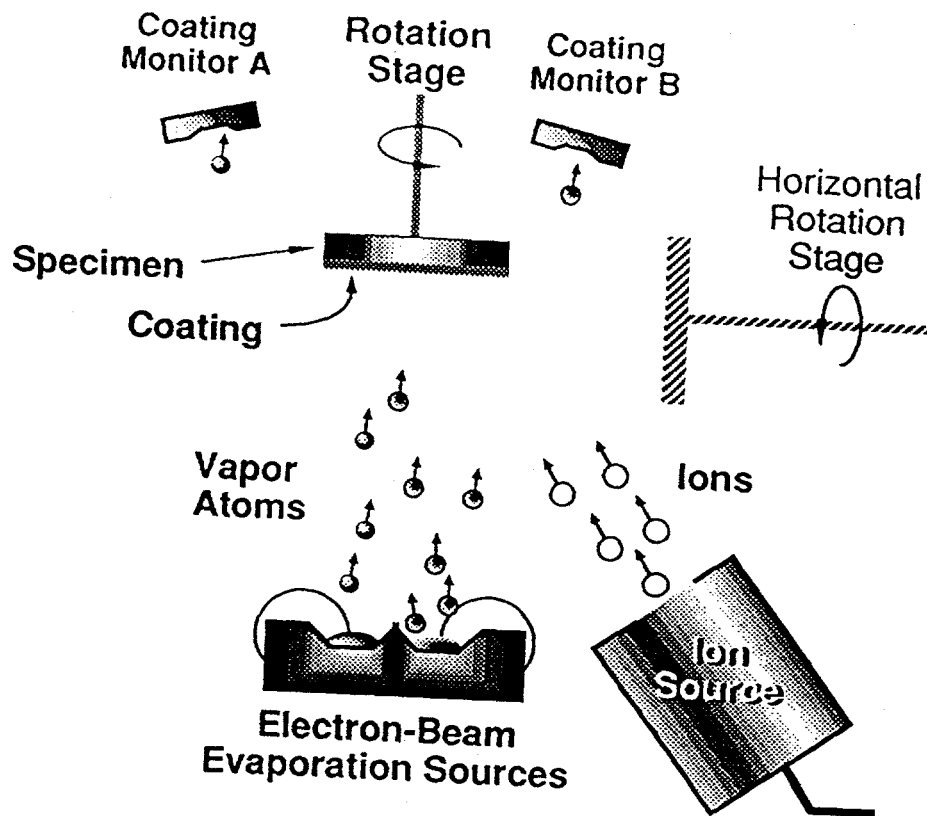


Figure 1.

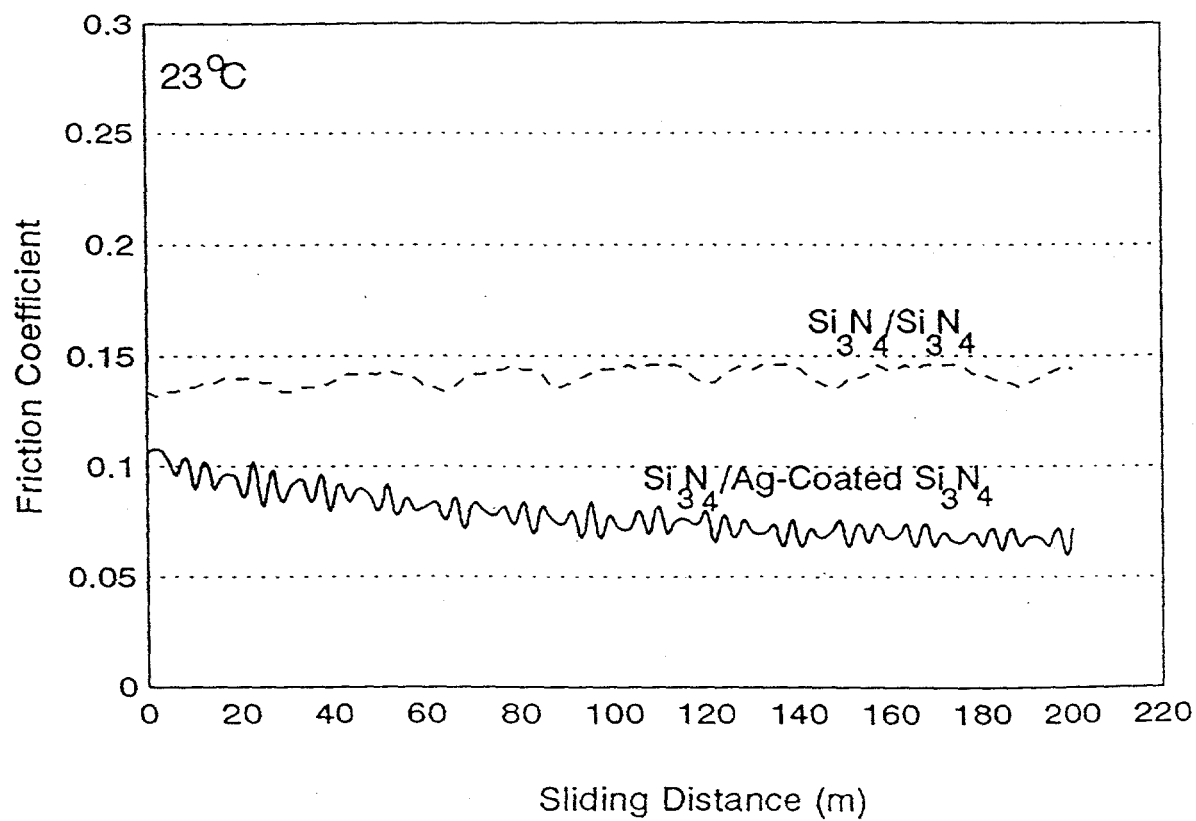


Figure 2.

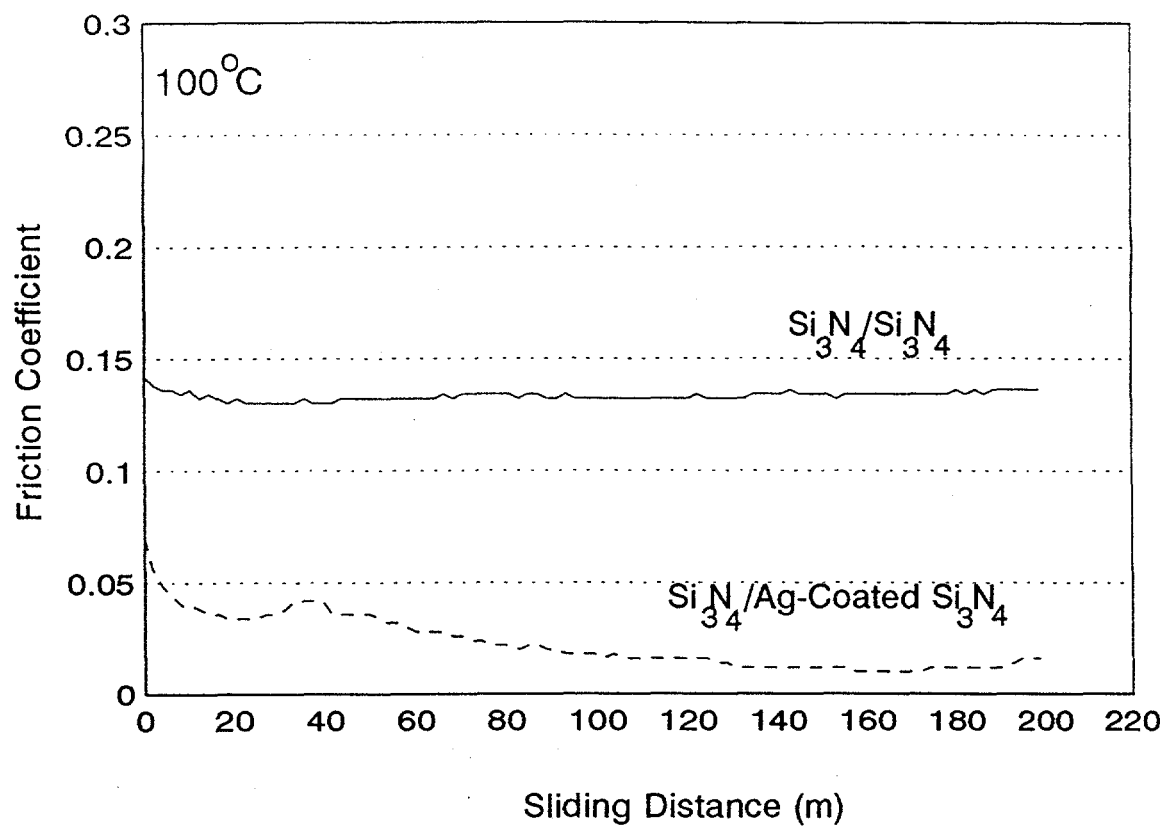


Figure 3.

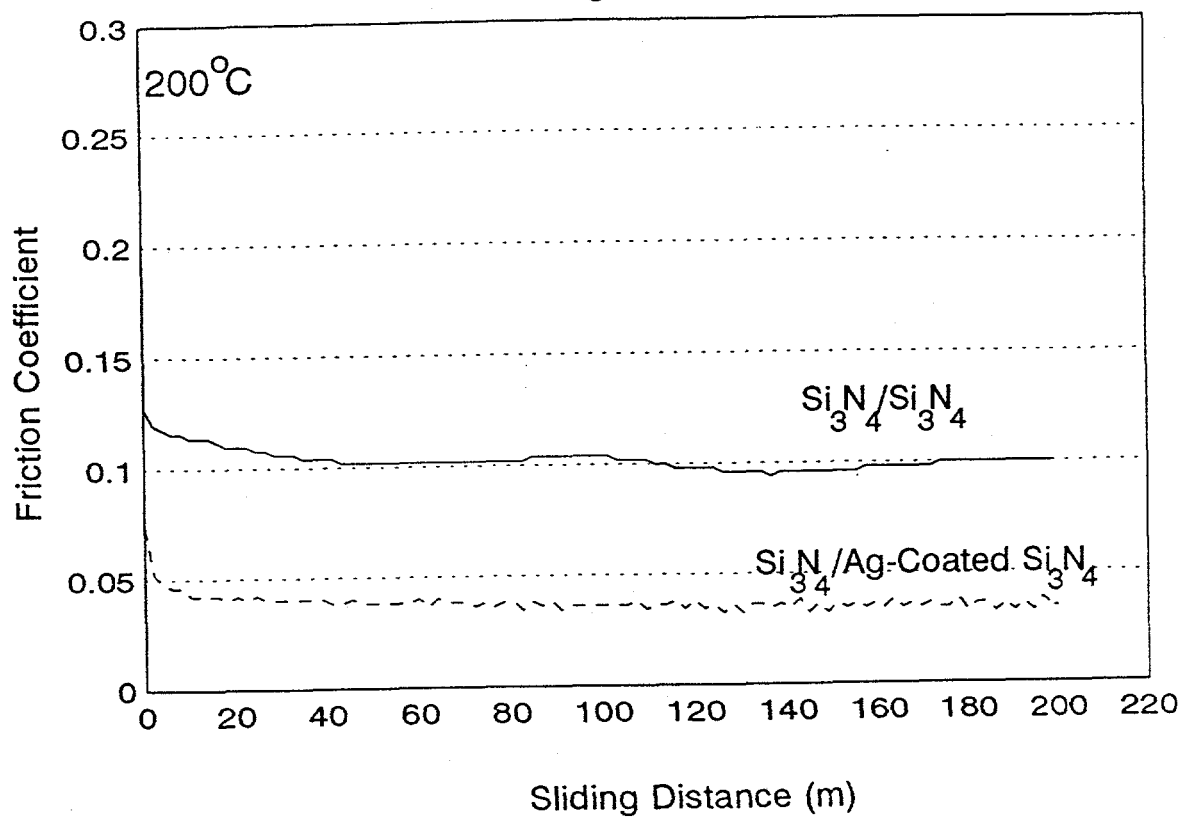


Figure 4.

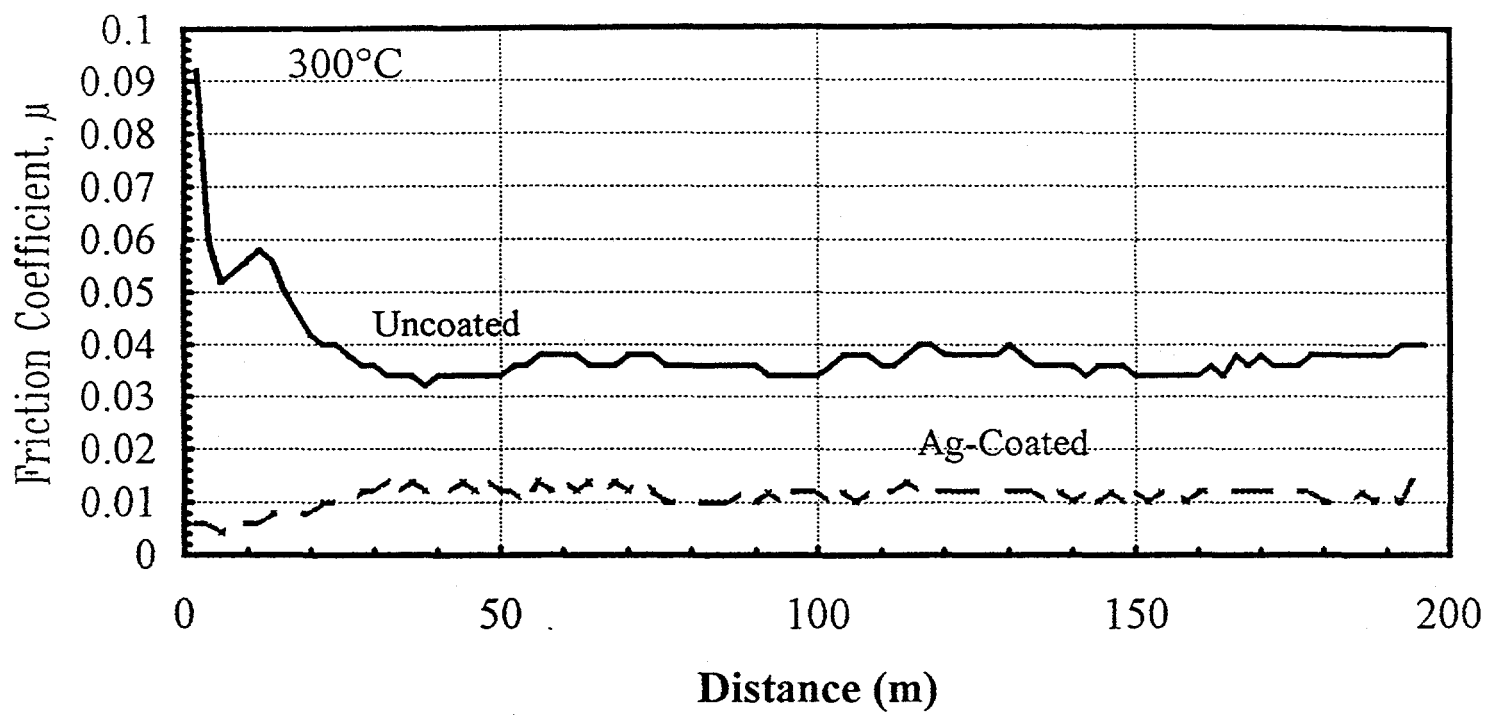
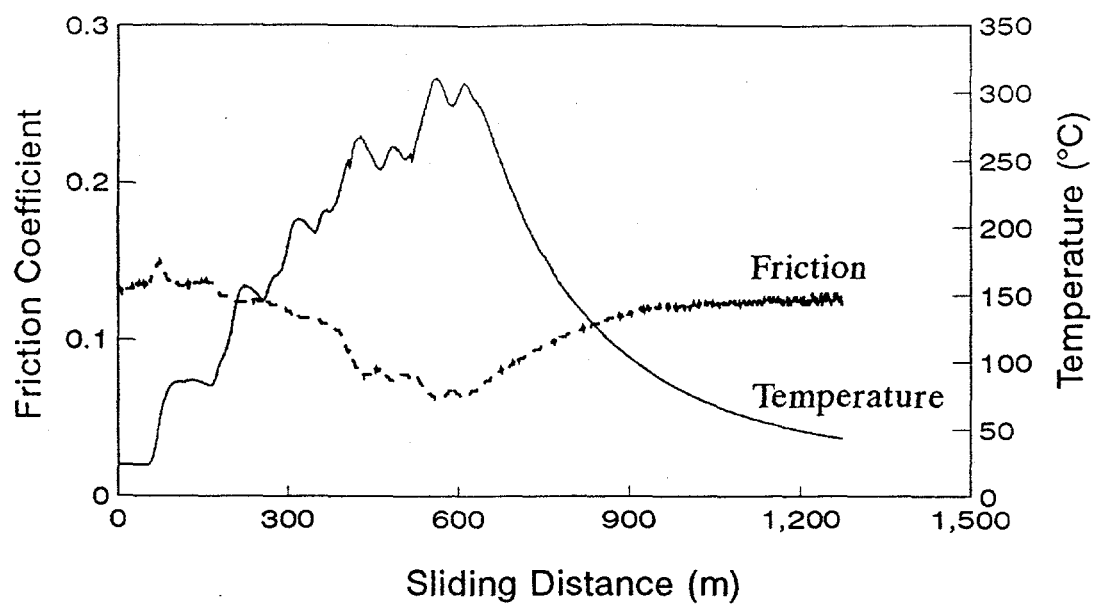
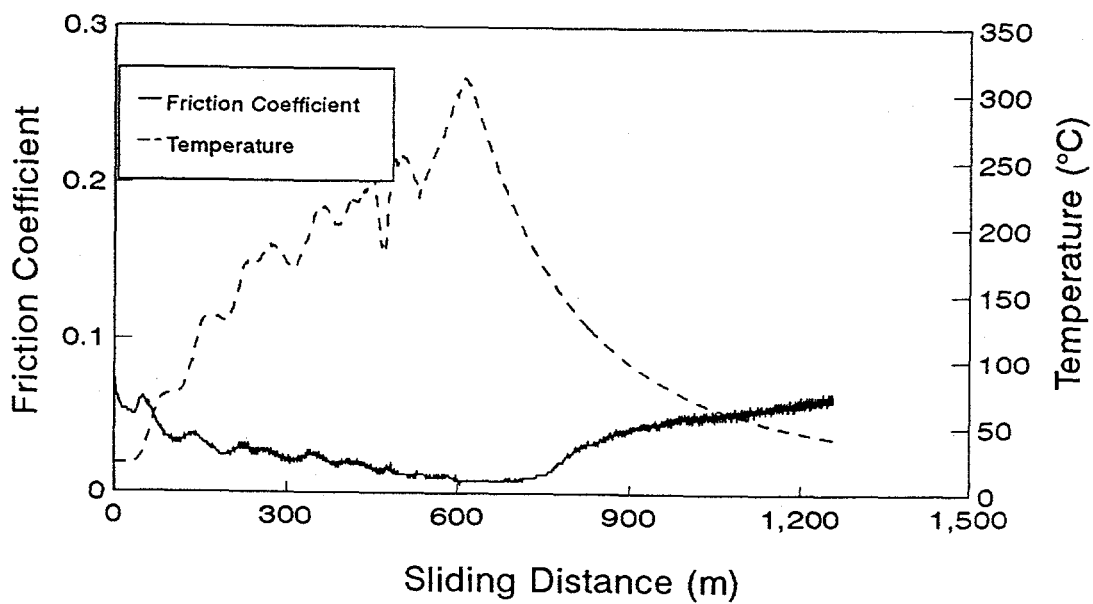


Figure 5.



(a)



(b)

Figure 6.

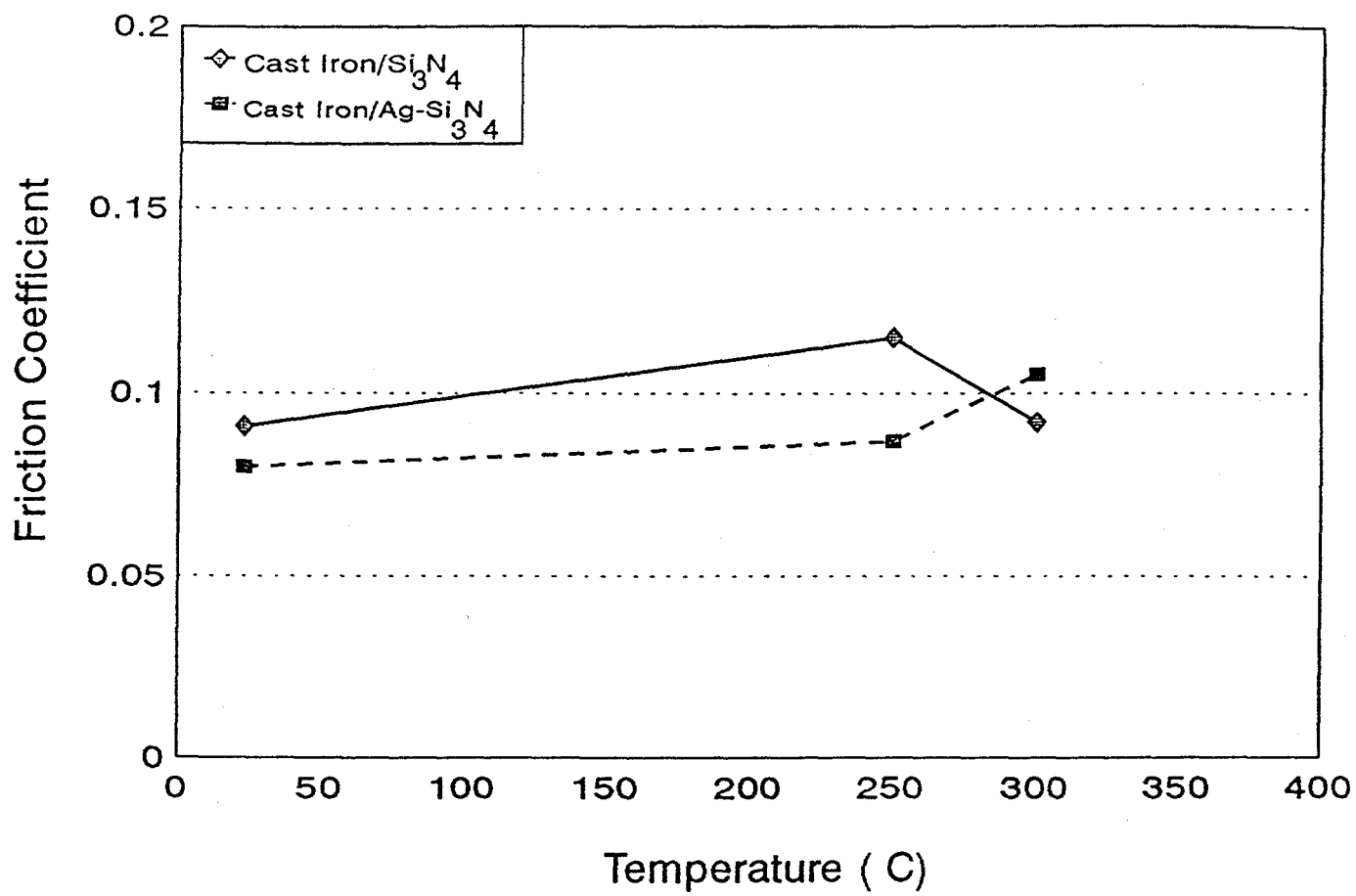


Figure 7.

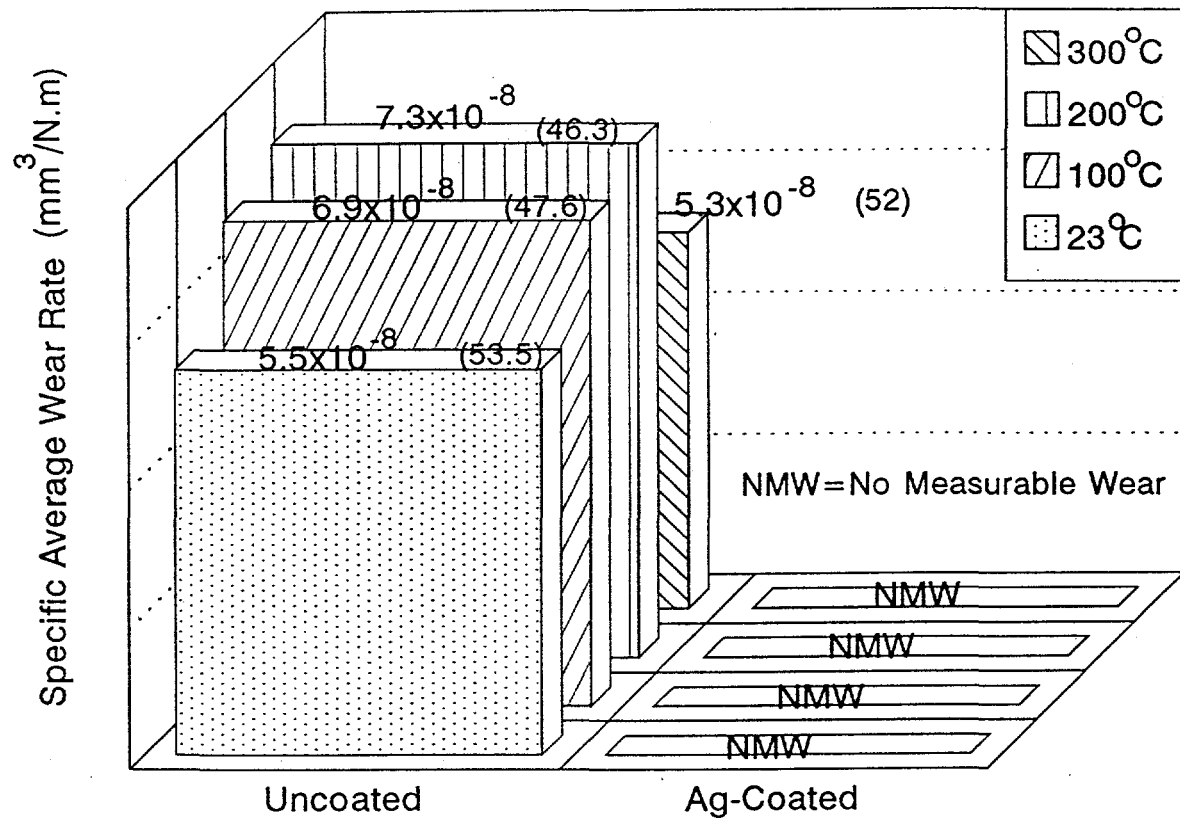
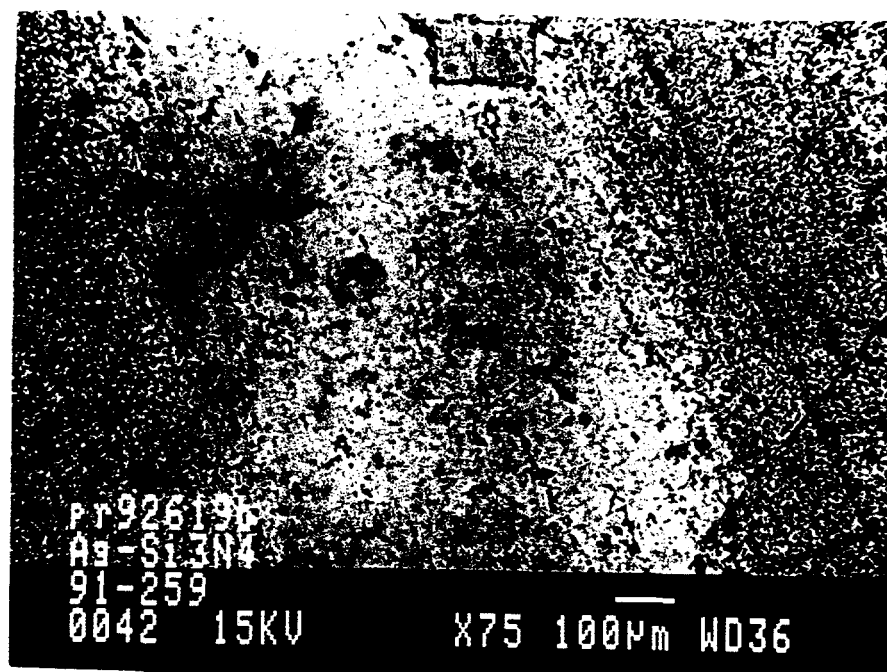
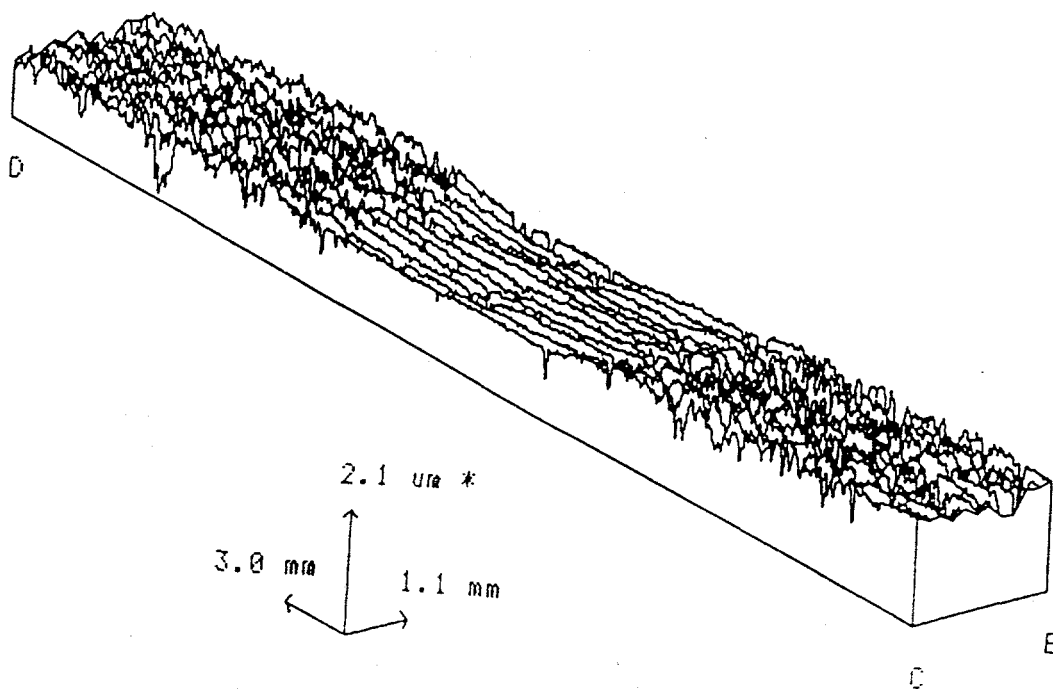


Figure 8.



(a)



(b)

Figure 9.

Pin Wear

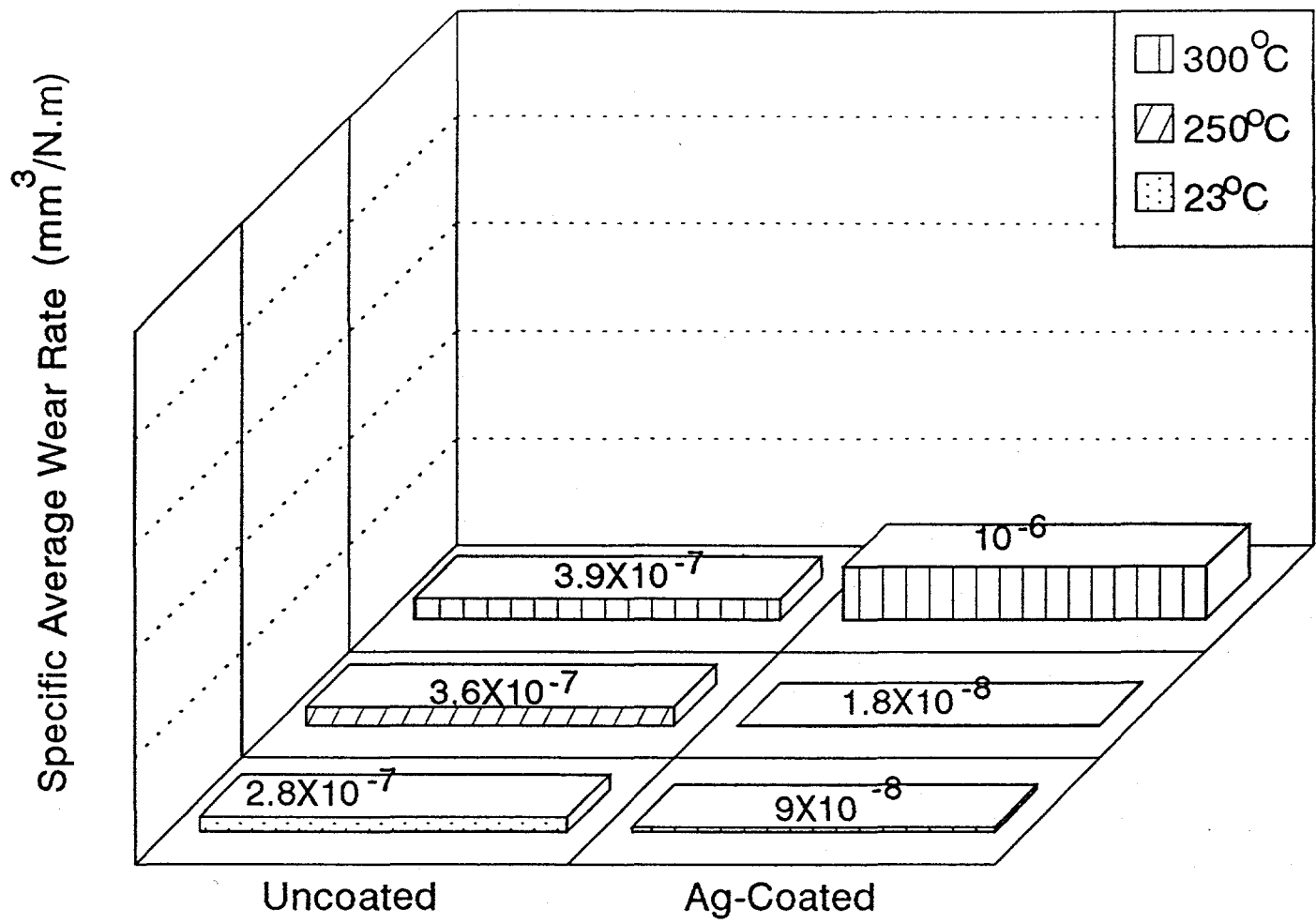
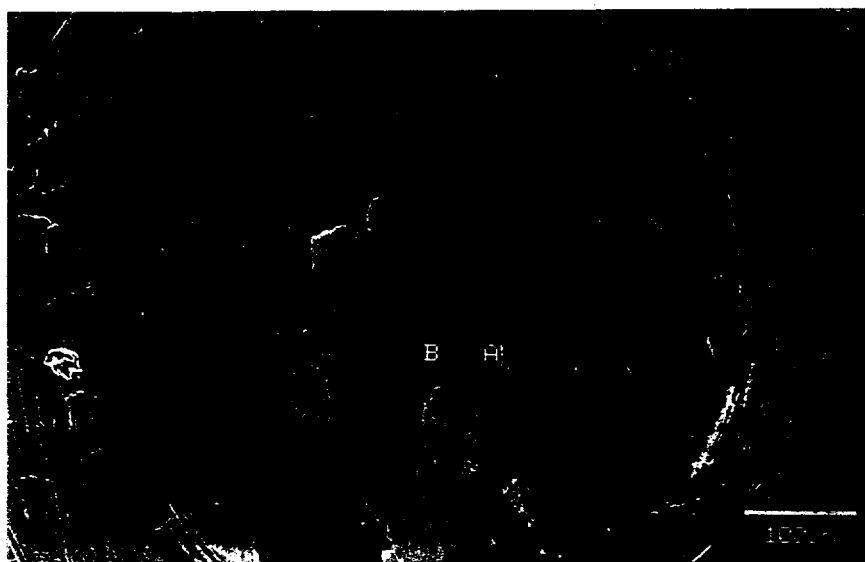


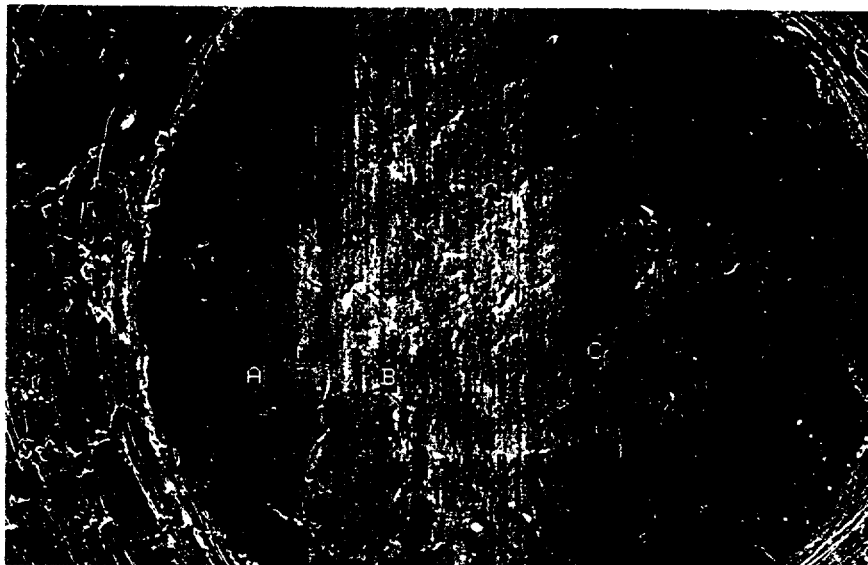
Figure 10.



(a)



(b)

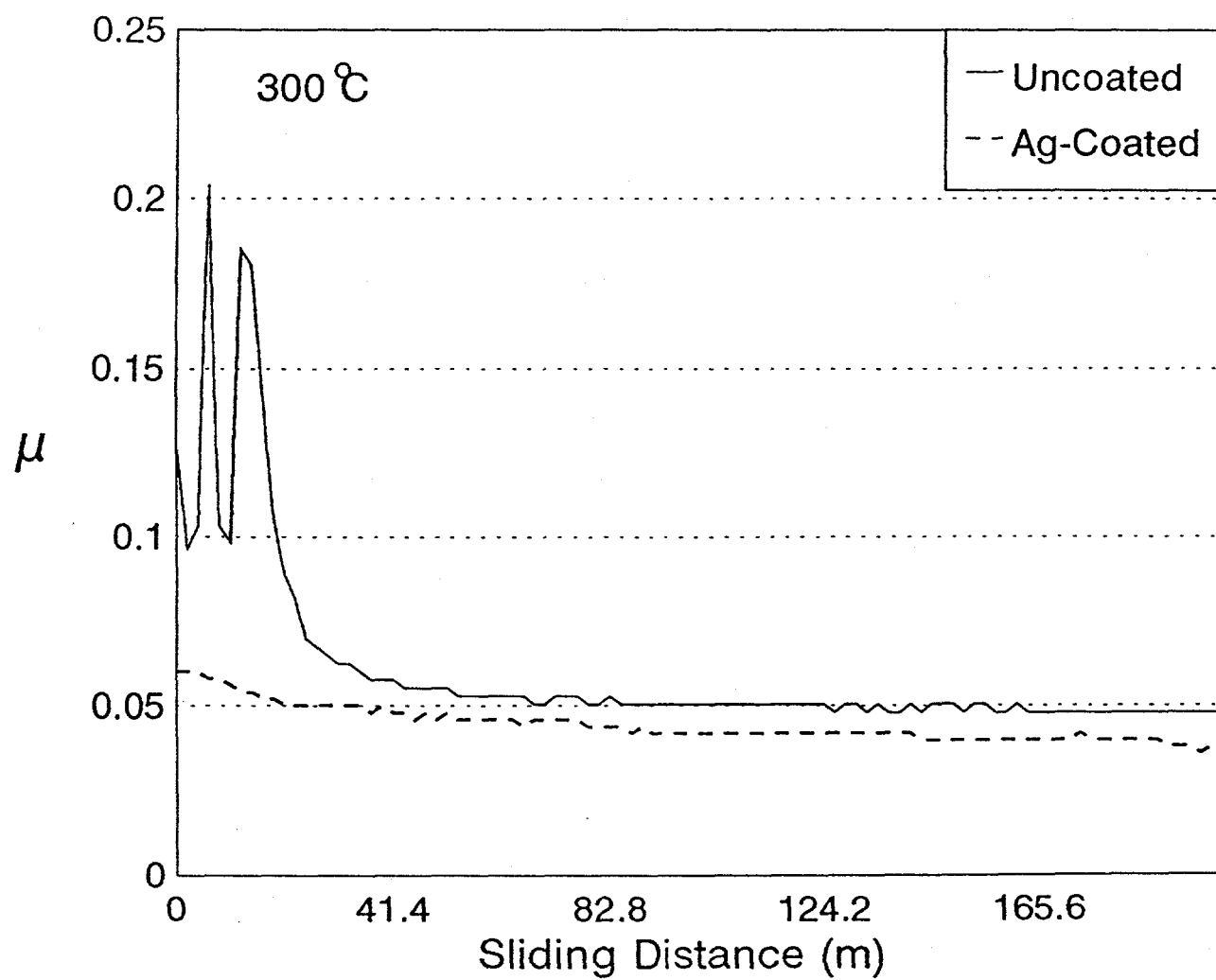


(c)



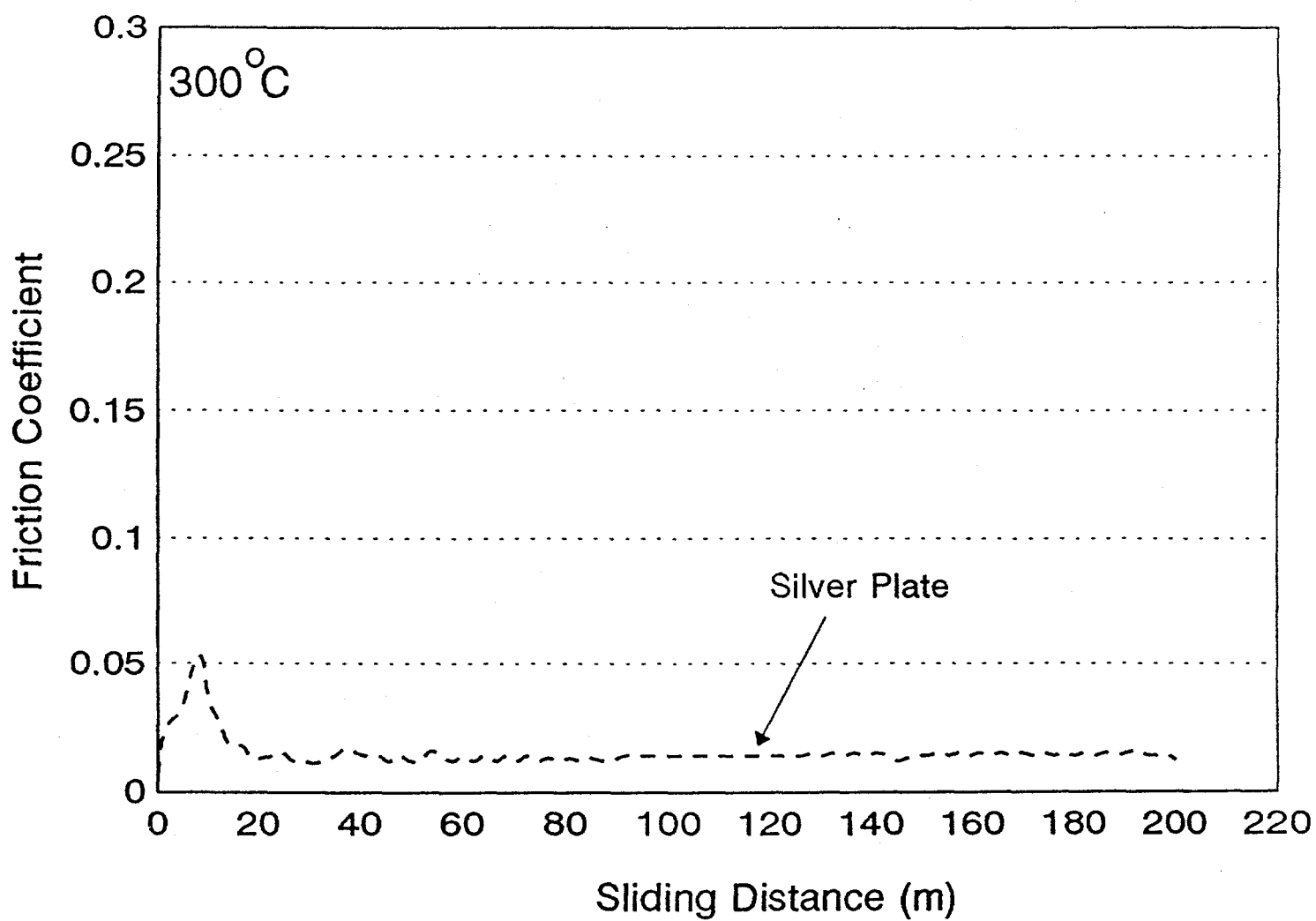
(d)

Figure 11.



(b)

Figure 12.



(a)