

# SYNERGISTIC EFFECTS OF SILVER FILMS AND SYNTHETIC LUBRICANTS ON BOUNDARY-LUBRICATION BEHAVIOR OF CERAMICS\*

A. Erdemir, O. O. Ajayi, R. A. Erck, G. R. Fenske, and  
F. A. Nichols

Tribology Section  
Materials and Components Technology Division  
Argonne National Laboratory  
Argonne, IL 60439

ANL/MCT/CP--76367

DE93 007895

J. M. Ockers  
University of Illinois at Urbana-Champaign  
Urbana, IL 61801

K. K. Kar and T. A. Morgan  
The Dow Chemical Corporation  
Midland, MI 48667

The submitted manuscript has been authored by a contractor of the U. S. Government under contract No. W-31-109-ENG-38. Accordingly, the U. S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U. S. Government purposes.

RECEIVED  
FEB 18 1993  
OSTI

November 1992

## DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

To be published in the Proceedings of the 1992 Coatings for Advanced Heat Engines Workshop, August 2-7, 1992, Monterey, CA

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

**MASTER**

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

*clb*

# SYNERGISTIC EFFECTS OF SILVER FILMS AND SYNTHETIC LUBRICANTS ON BOUNDARY-LUBRICATION BEHAVIOR OF CERAMICS\*

A. Erdemir, O. O. Ajayi, R. A. Erck, G. R. Fenske, and  
F. A. Nichols

Tribology Section  
Materials and Components Technology Division  
Argonne National Laboratory  
Argonne, IL 60439

J. M. Ockers  
University of Illinois at Urbana-Champaign  
Urbana, IL 61801

K. K. Kar and T. A. Morgan  
The Dow Chemical Corporation  
Midland, MI 48667

## ABSTRACT

In a study seeking to achieve low friction and low wear on ceramic materials, we investigated a new lubrication concept that explores the synergistic effect of a silver film and a recently developed synthetic oil on the boundary-lubrication behavior of silicon nitride ( $\text{Si}_3\text{N}_4$ ) ceramics. Friction and wear tests were performed on a wear test machine at temperatures up to  $380^\circ\text{C}$ . Under the test conditions explored, we found that the friction coefficients of  $\text{Si}_3\text{N}_4/\text{Si}_3\text{N}_4$  test pairs during oil-lubricated sliding tests ranged from 0.1 to 0.35, and the average wear rates of ceramic pins were between  $3 \times 10^{-7}$  and  $10^{-6} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$ , depending on test temperature. Concurrent use of lubricant oil with a silver film had a synergistic effect on both friction and wear. Specifically, it was observed that when silver films are used at oil-lubricated sliding interfaces, the wear rates of both pins and flats were reduced to unmeasurable levels and the friction coefficients were reduced by factors of two to ten below those of the test pairs without silver films. Beneficial synergistic effects of silver films and synthetic oil on the boundary-lubrication behavior of ceramics were more pronounced at elevated test temperatures than at room temperature.

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

## INTRODUCTION

Stringent operating conditions of advanced heat and turbine engines require the use of new materials and lubricants that can endure high operating temperatures, corrosive environments, and high contact pressures. For the materials needs of these transportation systems, certain ceramics ( $\text{Si}_3\text{N}_4$ ,  $\text{SiC}$ ,  $\text{ZrO}_2$  etc.) and ceramic coatings (e.g.,  $\text{CrN}$ ,  $\text{Cr}_2\text{O}_3$  and  $\text{ZrO}_2$ ) are being considered. Owing to their high mechanical strength and hardness, and excellent thermal and chemical properties, these monolithic ceramics and ceramic coatings hold promise for advanced engine applications [1-5]. Unlike traditional (mostly iron-base) engine materials, they can retain their hardness, and other desirable properties at fairly high temperatures. Furthermore, excellent thermal insulation afforded by some of the ceramics (especially  $\text{ZrO}_2$ -base ceramics) allows their use as thermal barrier coatings in turbine and low-heat-rejection engines. However, despite their excellent mechanical, thermal, and chemical properties, recent tribological experiences with these and other candidate ceramics have demonstrated that the friction and wear performance of most ceramics and their coatings is rather poor, especially under dry and high-temperature sliding conditions [6-10].

To achieve lower friction and wear, a series of liquid [1,3,11] and solid lubricants [12-14] have recently been developed and are now being tested on ceramic surfaces. Initial test results with synthetic lubricants appear highly promising [15,16], whereas the test results from solid-lubricated ceramics are not as favorable. Recently, tests by our group at Argonne demonstrated that when used simultaneously with liquid lubricants, solid-lubricant coatings can also be very effective in controlling friction and wear in metallic and/or ceramic tribosystems [17-19].

Over the years, we have accumulated a great deal of knowledge and experience in the fabrication and testing of solid-lubricant coatings in our tribology laboratory. Specifically, we have demonstrated that adherent silver films can greatly reduce the wear of sliding  $\text{ZrO}_2$ ,  $\text{Si}_3\text{N}_4$ , and  $\text{Al}_2\text{O}_3$  ceramics [20-22]. We found that thin silver films, 1 to 2  $\mu\text{m}$ -thick, can virtually eliminate the wear of rotating ceramic disks, while reducing the wear rates of stationary counterface (uncoated) pins by one to three orders of magnitudes below those of pins slid against uncoated flats.

Silver exhibits low shear strength, high thermal conductivity, and excellent chemical inertness which are important for improved tribological behavior and long durability. However, we noticed that the reduction in friction through the use of silver alone was somewhat marginal, e.g., 40 to 60%. Consequently, in a series of recent studies aimed at achieving not only low wear but also low friction, we directed our effort to a new lubrication concept; combined solid and liquid lubrication of ceramics. Specifically, we explored the synergistic effects of silver films and advanced synthetic oils on the boundary-lubrication behavior of silicon nitride ( $\text{Si}_3\text{N}_4$ ) and other advanced ceramics. In this study, we report on the synergistic effect of silver films and a synthetic lubricant (Dow X-1P) on the sliding friction and wear of  $\text{Si}_3\text{N}_4$  at temperatures up to 380°C.

We believe that because of its high chemical inertness, relatively high melting point (961°C), low shear strength, and very high thermal conductivity (429 W.m<sup>-1</sup>.K<sup>-1</sup> at room temperature), silver can act as a back-up lubricant at oil-lubricated sliding interfaces, especially at elevated temperatures where liquid lubricants become rather ineffective and silver becomes more easily shearable due to thermal softening. In previous studies, we showed that under high-load/pressure and high-speed sliding conditions (where high frictional heat is produced and dissipated to sliding surfaces), silver effectively dissipates heat from contact zones and thereby minimizes thermal and/or thermomechanical instabilities otherwise leading to severe wear [13,21,22].

## EXPERIMENTAL DETAILS

### Test Materials

The pins and disks of this study were fabricated from Si<sub>3</sub>N<sub>4</sub> powders by sintering at 1800°C. According to the technical data provided by the manufacturer [23], the principal constituent, Si<sub>3</sub>N<sub>4</sub> powder, contained some residue of W and Y<sub>2</sub>O<sub>3</sub> sintering aid, resulting in grain boundaries containing amorphous yttrium silicates and a crystalline phase mainly made up of Y<sub>2</sub>Si<sub>2</sub>O<sub>7</sub>. Volume porosity of the end products was specified to be less than 1%. Scanning electron microscopy of a molten-NaOH-etched sample revealed that the sintered Si<sub>3</sub>N<sub>4</sub> consisted of elongated β-Si<sub>3</sub>N<sub>4</sub> grains with a length-to-width ratio of ≈4. The disk specimens, 75 mm in diameter by 8 mm thick, were surface-finished by diamond-wheel grinding to an average roughness of 0.2 ± 0.02 μm center-line average (CLA). Other mechanical and thermal properties of the Si<sub>3</sub>N<sub>4</sub> used in this study are shown in Table 1.

The counterface pins were 8 mm in diameter by 15 mm long. One end of each pin was hemispherically finished to a radius of curvature of about 127 mm with a surface roughness of about 0.03 μm CLA. They were firmly secured on a pin holder to assume the vertical pin-on-horizontal-disk configuration. All specimens were ultrasonically cleaned sequentially in hexane + 10 vol.% toluene, acetone, deionized water containing 2 wt.% laboratory detergent, and deionized water for about 1 min each, then dried in an oven at 110°C for 20 min. This cleaning sequence was shown in Ref. 24 to remove most of the organic contaminants from the ceramic surfaces.

Table 1. Selected room-temperature mechanical and thermal properties of  $\text{Si}_3\text{N}_4$  ceramics (Kyocera SN-220 grade) used in this study [23].

Property	$\text{Si}_3\text{N}_4$
Fracture Toughness (Mpa√m)	5.7
Hardness (Gpa)	17.4
Flexural Strength (Mpa)	590
Young's Modulus (Gpa)	294
Poisson Ratio	0.28
Density ( $\text{kg.m}^{-3}$ )	3200
Thermal	
Conductivity ( $\text{W.m}^{-1}.\text{K}^{-1}$ )	21
Coefficient of Thermal Expansion ( $10^{-6}.\text{K}^{-1}$ )	2.6

### **Ion-Beam-Assisted Deposition (IBAD) of Silver**

The IBAD of thin silver films (e.g.,  $\approx 2 \mu\text{m}$  thick) was performed at room temperature in a vacuum chamber equipped with an electron-beam-heated evaporation source. A mixture of argon and oxygen gas was fed through the ion source to create an ion flux composed of the ions of these gases. To remove the adsorbed contaminants from the surface, the substrates were subjected to Ar + O ion bombardment at an acceleration voltage of 1 keV before deposition of the silver. To achieve better adhesion, a titanium bond layer, 10 to 20 nm thick, was first deposited on the substrate surfaces. The thickness of the growing film was controlled with the aid of a quartz-crystal rate monitor. Ion bombardment of the growing films was achieved with a hot-cathode Kaufman-type ion gun. Acceleration voltage and ion-current density were 1 keV and  $25 \mu\text{A.cm}^{-2}$ , respectively, during silver deposition. The ions were neutralized by a hot-wire filament, thereby reducing the charging of insulating ceramic substrates. After a 1000-Å-thick film was produced under ion bombardment, the ion beam was turned off and the gas flow through the ion source was stopped. The balance of the film thickness was obtained by conventional electron-beam evaporation. A schematic depiction of the IBAD system is shown in Figure 1. Further details of this system, together with the structural characteristics of resultant films, were elaborated in previous publications [13,20-22].

### **Friction and Wear Tests**

Friction and wear tests were performed with the pairs of  $\text{Si}_3\text{N}_4$  pins and IBAD-silver-coated  $\text{Si}_3\text{N}_4$  disks in a pin-on-disk tribometer. Specifically, flat disks were placed in a lubricant cup and oil was supplied to the sliding interface by means of drip-feeding at rates of 1 to 2 drops per 10 seconds, depending on test temperature. The liquid lubricant was an experimental fluid developed by The Dow Chemical Co, Midland, Mi, with a code name X-1P. This fluid was previously tested on various ceramic and metallic surfaces at temperatures up to  $\approx 460^\circ\text{C}$  and was found to evaporate at high

temperatures and leave very little deposit on sliding surfaces [16]. Table 2 presents some of the thermal and physical properties of this lubricant. A more detailed description of X-1P can be found in ref. 25.

Tests were performed in open air of 30 to 40 % relative humidity. The dead weight applied on top of the ball specimens was 50 N which created an initial mean Hertzian contact pressure of approximately 166 MPa between the  $\text{Si}_3\text{N}_4$  pin and the  $\text{Si}_3\text{N}_4$  flat (ignoring any effects of the Ag film). Because of the formation of a flat wear-scar during sliding contacts, by the end of the tests nominal bearing pressures fell to a small fraction of this initial Hertzian value. The final nominal contact pressures for each test pair are given in the graphs showing the wear data in the results section. The elevated test temperatures, 100, 200, and 300°C, were created by quartz heaters positioned beneath the rotating disk specimens. In a few tests, temperature was allowed to go up to 380°C while sliding was in progress. The sliding velocity was fixed at 0.1 m/s and the total sliding distance was 200 m. Frictional force was monitored by means of a load cell and recorded on a chart paper and a floppy disk by means of a data-acquisition system.

Wear-volume calculations on the balls used a mathematical expression given in an ASTM Standard [26]. The wear of disk specimens was estimated from the traces of surface profiles across the wear tracks. To check the accuracy and reproducibility of test results, some of the tests were repeated two to three times under each condition.

Table 2. Properties of X-1P lubricant oil.

Property	Value
Viscosity,	
40°C	$312 \times 10^{-3}$ Pa.s
100°C	$15.6 \times 10^{-3}$ Pa.s
200°C	$2.32 \times 10^{-3}$ Pa.s
Specific Gravity	
40°C	1.46
Pour Point	-15 °C
Flash Point	>275°C

## RESULTS AND DISCUSSION

### FRICITION

As shown in Fig. 2, with the use of X-1P lubricant alone, the friction coefficient of the  $\text{Si}_3\text{N}_4/\text{Si}_3\text{N}_4$  test pairs at room temperature (23°C) fluctuates between 0.1 and 0.14 which is typical for boundary-lubrication regimes in most oil-lubricated systems. The lack of extreme pressure and other additives in this base-stock oil may have been the reason for erratic friction traces as evident from Fig. 2. However, when X-1P is used on silver-coated  $\text{Si}_3\text{N}_4$  surfaces, the steady-state friction coefficient was markedly lower,  $\approx 0.09$ , and the erratic frictional behavior was less pronounced.

Fig. 3 shows the friction coefficients of the  $\text{Si}_3\text{N}_4/\text{Si}_3\text{N}_4$  test pairs with and without a silver film at  $100^\circ\text{C}$ . Without silver, the friction coefficient of pairs fluctuates between 0.13 and 0.22. However, when silver is present on the sliding surface, the friction coefficient drops to a value of 0.05 at steady-state and the frictional trace is much less erratic.

Fig. 4 presents the frictional performance of  $\text{Si}_3\text{N}_4/\text{Si}_3\text{N}_4$  test pairs with and without a silver film at  $200^\circ\text{C}$ . As is clear, without a silver film, the friction coefficient is  $\approx 0.18$  at steady-state. Sometimes, it becomes unstable and fluctuates between 0.13 and 0.24. In contrast, the friction coefficient of the pair with a silver film is essentially constant at  $\approx 0.04$ .

Fig. 5 shows the friction coefficients of the  $\text{Si}_3\text{N}_4/\text{Si}_3\text{N}_4$  test pairs with and without a silver film at  $300^\circ\text{C}$ . The friction coefficient of the silver-coated  $\text{Si}_3\text{N}_4$  disk was in the range of 0.05 to 0.1, whereas the friction coefficient of an uncoated disk was highly unstable. As shown in Fig. 5, it fluctuates between 0.16 and  $\approx 0.4$  during sliding contact at  $300^\circ\text{C}$ .

Fig. 6 shows the friction coefficients of (a) the  $\text{Si}_3\text{N}_4/\text{Si}_3\text{N}_4$  and (b)  $\text{Si}_3\text{N}_4/\text{Ag-coated Si}_3\text{N}_4$  test pairs as a function of increasing temperature. In general, these results are consistent with those presented in Figs 2 -5. They show that without the presence of a silver film at the sliding interface, the friction coefficient is high, e.g., 0.08 - 0.38, and becomes highly unstable as temperature increases, whereas the friction coefficient of pairs with a silver film varies between  $\approx 0.03$  and  $\approx 0.09$  and does not fluctuate nearly so much with increasing temperature. At room temperature, the friction coefficient is about 0.1, but decreases sharply as the temperature is increased. At around  $160^\circ\text{C}$ , the friction coefficient is about 0.03. Upon further increasing the temperature to  $250^\circ\text{C}$ , the friction coefficient increases to about 0.1, but tends to decrease again with further increase in temperature. At  $380^\circ\text{C}$ , the friction coefficient is about 0.06.

In general, from the results of liquid-lubricated friction tests at 23, 100, 200, and  $300^\circ\text{C}$ , as well as the tests conducted as a function of temperature, it can be deduced that the friction coefficients of the  $\text{Si}_3\text{N}_4/\text{Si}_3\text{N}_4$  test pairs tend to increase with increasing temperature. Also evident is the fact that these pairs tend to exhibit an increasing degree of instability as temperature increases. We believe that the increase in friction coefficient with temperature is largely due to a steep decrease in oil viscosity (see Table 2). As is known, the lubricant film thickness is quite dependent on oil viscosity. For a given tribosystem, lower oil viscosity means thinner lubricant films and higher amounts of asperity-asperity contacts that can lead to higher friction and wear losses [27,28]. Nevertheless, the range of friction coefficients observed is typical of a boundary-lubricated sliding-contact regime [11,16]. Electron microscopic examination and a 3-D surface map in Fig. 7 provide further evidence that because of the extensive asperity-asperity contacts, pins slid against the uncoated  $\text{Si}_3\text{N}_4$  disks at  $300^\circ\text{C}$  suffered significant wear. At a higher magnification (see Fig. 8), it was deduced that the major cause of wear was microfracture.

Much of the instability in frictional traces in Figs. 2-5 and 6a can be attributed to the inability of X-1P to form an effective boundary film on sliding surfaces, because this oil did not contain any type of oil additives that could help produce a boundary film. Erratic frictional behavior at room temperature (see Fig. 2a) has a well-defined periodicity. Such a behavior could be due to a small-scale molecular interaction (e.g., adsorption and/or chemisorption) between lubricant and the sliding ceramic surface. Also, we believe that the drip-feeding method adopted during our sliding tests may have contributed to the degree of fluctuation, especially at elevated temperatures where X-1P became highly volatile. At 300°C, we noticed that the lubricant evaporated rather quickly, so that we had to increase the lubricant feed rate from 1 drop to 2 drops per 10 sec.

One of the unique characteristics of the X-1P lubricant was that after high-temperature tests, it left little or no deposit on the sliding surfaces. In the past, we evaluated a commercially available high-temperature fluid and found that it formed a significant amount of deposit on the sliding ceramic surfaces following wear tests at high temperatures and became essentially useless above 250°C [18,19].

As is clear from Figs. 2-5, the use of a silver film and the X-1P lubricant on sliding Si<sub>3</sub>N<sub>4</sub> surfaces produced a synergistic effect. Specifically, the steady-state friction coefficients of test pairs were substantially reduced when silver and X-1P were used simultaneously, especially at elevated test temperatures. These findings suggest that the silver film used in our study may be a good back-up lubricant.

Owing to its low shear strength, silver deforms easily to fill in the valleys between surface asperities of the Si<sub>3</sub>N<sub>4</sub> flat under the influence of normal and tangential forces during sliding. The shear deformation of silver becomes much easier at elevated test temperatures because of the thermal softening effect. As a result, the smoothness of the sliding contact surfaces of silver-coated Si<sub>3</sub>N<sub>4</sub> disks improves. This is evident from the low- and high-magnification SEM micrographs of a wear track formed at 100°C in Fig. 9. As described in more detail in the following paragraphs, as the contact surface becomes smoother, the composite roughness decreases, the  $\lambda$  ratio (the ratio of the lubricant film thickness ( $h$ ) to the composite surface roughness ( $\sigma$ ) of the contacting bodies,  $\lambda = h/\sigma$ ) increases, and the effectiveness of the liquid lubricant improves.

The regime of liquid lubrication and/or the effectiveness of a lubricant is determined by the  $\lambda$  (lambda) ratio. When  $\lambda$  is greater than 3, the sliding surfaces are sufficiently separated that essentially no asperity-asperity contacts are expected. This lubrication regime is referred to as hydrodynamic. For  $\lambda$  less than 1, the lubricant film thickness is smaller than the composite surface roughness and extensive asperity-asperity contacts through the lubricant film occur. This lubrication regime is known as boundary lubrication. Because of extensive asperity-asperity interactions across the lubricant film, both friction and wear may be high in the boundary-lubrication-regime. As a general rule,



the higher the  $\lambda$  ratio, the more effective the fluid-film lubrication.

Because of a much smoother surface finish achieved through the plastic deformation of silver films (see Fig. 9) the composite surface roughness of test pairs including a silver film was markedly reduced. Consequently, the  $\lambda$  ratio was increased. It is therefore reasonable to conclude that the observed decrease in the friction coefficient of all test pairs consisting of a Ag film was most likely due to the increased  $\lambda$  ratio which is analogous to more effective fluid-film lubrication. Furthermore, since the silver film effectively reduces the extent of asperity-asperity contacts across the sliding interfaces and carries a significant portion of the contact stresses during sliding contact, it can be regarded as an excellent boundary film.

After wear tests at 200°C and above, we noticed that some of the wear tracks on Ag-coated  $\text{Si}_3\text{N}_4$  flats contained large stains and/or islands of some kind of reaction film. Using energy-dispersive X-ray analysis in our SEM, we found that these islands were rich in phosphorus and fluorine in addition to silver. Both phosphorus and fluorine were constituents of the X-1P lubricant. It is possible that at such high temperatures, both of these constituents underwent chemical reaction with silver. Such a reaction film may have been acting as a boundary film and may have been responsible for the smoother friction traces and lower friction coefficients observed at elevated temperatures. However, in the long run, such chemical reactions with silver may have some adverse effects on the lifetime and overall performance of silver coatings. Long-term tests and more rigorous analyses are needed to shed light on the beneficial or detrimental effect of these reaction films on the durability and frictional performance of silver films.

## WEAR

Fig. 10 presents the average specific wear rates of the  $\text{Si}_3\text{N}_4$  pins during sliding against uncoated and silver-coated  $\text{Si}_3\text{N}_4$  disks at different temperatures. The values in parentheses indicate the final nominal contact pressures in MPa. The wear rates of pins are substantial during sliding against the uncoated flats and increase markedly with increasing test temperature. This is despite a significant reduction in final nominal contact pressure. On the other hand, the wear of pins slid against the silver-coated disks was difficult to measure. Using a scanning electron microscope and a surface profilometer at high magnifications, we found no evidence of a measurable wear scar on  $\text{Si}_3\text{N}_4$  pins after sliding against the silver-coated  $\text{Si}_3\text{N}_4$  disks at temperatures up to 300°C. However,  $\text{Si}_3\text{N}_4$  pins tested at 300°C appeared to have formed small wear scars after sliding tests against the Ag-coated  $\text{Si}_3\text{N}_4$  disks. We measured the diameters of these wear scars and converted them into the average wear rates shown in Fig. 10. For the pins rubbed at 200°C (see Fig. 11a) a circular contact area appeared where some silver had transferred from the disk side, but in the 3-D surface map (see Fig. 11b), it is not obvious whether the circular area had indeed suffered any measurable wear or not. Overall, these observations suggest that the silver film was quite effective in reducing direct

asperity/asperity interactions while sliding, whereas, as presented earlier, those pins used against the uncoated  $\text{Si}_3\text{N}_4$  disks underwent significant amounts of wear as shown in Fig. 10.

Wear of silver-coated disks was unmeasurable after tests with the liquid lubricant at all temperatures. Using a surface profilometer at vertical magnifications of 5,000 to 10,000X, we could not detect wear on the  $\text{Si}_3\text{N}_4$  ceramics. Essentially, the depth of wear tracks was very shallow and comparable to the thickness of silver films. Electron-microscopy inspection revealed that silver films remained intact on the wear tracks of  $\text{Si}_3\text{N}_4$  disks. When inspected at a fairly high magnification, only the tips of underlying asperities were visible and the wear appeared to be confined to these exposed asperity tips.

The results presented above demonstrate that the concurrent use of silver films and a synthetic lubricant at sliding interfaces can markedly lower the friction and wear of ceramic materials evaluated in this study. We propose the following explanation for the excellent friction- and wear-reducing capability of silver films when used in an oil-lubricated sliding contact. Because of its low shear strength, the silver film deforms readily to fill in the valleys between asperities under the influence of normal and tangential forces during sliding. As a result, the smoothness of the silver-coated-flats improves. Because of the increased  $\lambda$  ratio afforded by the increasingly smooth surface, the effectiveness of the lubricant oil improves. This is particularly true for those tests run at room temperature, where the oil has appreciable viscosity. At elevated temperatures, however, the oil may lose much of its viscosity, hence the load-bearing capacity. However, under such extreme conditions, the silver film acts as a boundary layer in supporting much of the load. Because of some thermal softening at elevated temperatures, the silver shears much more readily, hence affording lower friction.

In short, the test results have demonstrated that an effective means of achieving low friction and low wear on potential ceramic tribocomponents is feasible by the simultaneous use of liquid and solid-film lubricants. Most importantly, the results show that silver films and lubricant oils can have synergistic effects on the boundary-lubrication characteristics of ceramics due to easy shear of the silver.

## CONCLUSIONS

\* The results of this study demonstrate that a new means of achieving low friction and low wear on ceramics is feasible by the simultaneous use of both liquid and solid-film lubricants.

\* Specifically, the results show that when silver films are used at oil-lubricated sliding interfaces, the wear rates of both pins and flats were reduced to unmeasurable levels and the friction coefficients were reduced

by factors of 2 to .

\* 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 can act as a backup lubricant by undergoing easy shear during oil-lubricated sliding contacts. They can also impart an increasingly smooth surface finish to  $\text{Si}_3\text{N}_4$  flats thereby increasing the  $\lambda$  ratio, hence the effectiveness of the lubricant oil.

## ACKNOWLEDGMENT

This work was supported by the Tribology Program, Office of Transportation Materials, U.S. Department of Energy, under Contract W-31-109-Eng-38.

## REFERENCES

1. Dufrane, K. F., and Glaeser, W. A., "Wear of Ceramics in Advanced Heat Engine Applications," Int. Conf. on Wear of Materials, ASME, New York, ed. Ludema, K.C. (1987) pp. 285-291.
2. R. Kamo, M. Woods, and P. Sutor, "Development of Tribological System and Advanced High-Temperature Diesel Engines, in Proc. 3rd Advanced Heat Engine Workshop (1987), pp. 73-92.
3. K. F. Dufrane and W. A. Glaeser, "Performance of Ceramics in Ring-Cylinder applications," Int. Congr. and Exposition, Detroit, SAE preprint 870416 (Feb. 23, 1987).
4. M. L. Sheppard, "Automotive Performance Accelerates with Ceramics," Ceram. Bull., 69, pp. 1012-1021 (1990).
5. A. V. Levy and N. Jee, "Unlubricated Sliding Wear of Ceramic Materials," Wear, 121, pp. 363-380 (1988).
6. C. S. Yust and F. J. Cariggan, "Observations on the Sliding Wear of Ceramics," ASLE Trans., 28, pp. 245-252 (1984).
7. M. Woydt and K. -H. Habig, "High Temperature Tribology of Ceramics," Tribol. Int., 22, pp. 75-88 (1989).
8. G. W. Stachowiak and G. B. Stachowiak, "Unlubricated Friction and Wear Behavior of Toughened Zirconia Ceramics," Wear, 132, pp. 151-171 (1989).
9. V. Aronov and T. Mesyef, "Wear in Ceramic/Ceramic and Ceramic/Metal Reciprocating Sliding Contact. Part 1," J. Tribol., 108, pp. 16-21 (1986).
10. E. Breval, J. Breznak, and N. M. Macmillan, "Sliding Friction and Wear of Structural Ceramics," J. mat. Sci., 21, pp. 931-935 (1986).
11. J. J. Habeeb, A. G. Blahey, and W. N. Rogers, "Wear and Lubrication of Ceramics," Proc. Inst. Mech. Eng., Inst. Mech. Eng., London, UK, pp. 555-564 (1987).

12. W. Wei, J. Lankford J., and R. Kossowsky, "Friction and Wear of Ion-beam-Modified Ceramics for Use in High Temperature Adiabatic Engines," Mat. Sci. Eng., 90, pp. 307-315 (1987).
13. A. Erdemir, D. E. Busch, R. A. Erck, G. R. Fenske, and R. Lee, "Ion-Beam-Assisted Deposition of Silver on Zirconia Ceramics for Improved Tribological Behavior," Lubr. Eng. 47, 1991, pp. 863-872.
14. M. Kanakia, M. E. Owens, and F. F. Ling, "Solid Lubrication of Advanced Heat Engines", in Proceedings of Workshop on Fundamentals of High Temperature Friction and Wear with Emphasis on Solid Lubrication for Heat Engines, Industrial Tribology Institute, Troy, NY (1984), ed. Ling, F. F., pp. 19-38.
15. P. Pool, "Synthesizing Oils is a Slippery Job," Science, 246, pp. 444-446 (1989).
16. P. A. Gaydos and K. F. Dufrane, "Studies of Dynamic Contact of Ceramics and Alloys for Advanced Heat Engines," Final Report, Oak Ridge National Laboratory, ORNL/sub/84-00216/1, 1992.
17. P. Sutor and W. Bryzik, "Laboratory Development and Engine Performance of New-High-Temperature Diesel Engine lubricants," Int. Congr. Expo., Detroit, Mi, SAE preprint # 890145 (Feb. 27, 1989).
18. A. Erdemir, O. O. Ajayi, G. R. Fenske, R. A. Erck, and J. H. Hsieh, "The Synergistic Effects of Solid and Liquid Lubrication on the Tribological Behavior of Transformation-Toughened ZrO<sub>2</sub> Ceramics," Trib. Trans., 35, 1992, pp. 287-297.
19. O. O. Ajayi, A. Erdemir, J. H. Hsieh, R. A. Erck, and G. R. Fenske, "Combined Solid and Liquid Lubrication of Silicon Nitride under Boundary Conditions," Lubr. Eng., 48, 1992, pp. 584-591.
20. Erdemir, A., Fenske, G. R., Erck, R. A., and Cheng, C. C., "Ion-Assisted Deposition of Silver Films on Ceramics for Friction and Wear Control," Lubr. Eng., 46, pp. 23-30 (1990).
21. Erdemir, A., Fenske, G. R., Nichols, F. A., and Erck, R. A., "Solid Lubrication of Ceramic Surfaces by IAD-Silver Coatings for Heat Engine Applications," Tribol. Trans., 33, pp. 511-518 (1990).
22. O. O. Ajayi, A. Erdemir, R. A. Erck, G. R. Fenske, and F. A. Nichols, "The Role of Soft (Metallic) Film in Tribological Behavior of Ceramic Materials," Wear, 149, 1991, pp. 221-232.
23. Kyocera Co., "Mechanical and Industrial Ceramics," Sales Cat., 1987.
24. R. S. Gates, J. P. Yellets, D. E. Deckman, and S. M. Hsu, "Considerations in Ceramic Friction and Wear Measurements," Selection and Use of Wear Tests for Ceramics, ASTM, Philadelphia, STP 1010, Eds. Yust, C. S., and Bayer. R. G., pp. 1-23, (1988).
25. B. S. Nader, K. K. Kar, T. A. Morgan, C. E. Pawloski, and W. L. Dilling, "Development and Tribological Properties of New Cyclotriphosphazene High Temperature Lubricants for Aircraft Gas Turbine Engines," Trib. Trans., 35, 1992, pp. 37-44.
26. 1990 Annual Book of ASTM Standards, Vol. 3., Wear and Erosion; Metal Corrosion, G99-90, "Standard Test Method for Wear Testing with a Pin-On-Disk Apparatus," ASTM, Philadelphia, PA, 1990, pp. 391-395.
27. D. Dowson and G. R. Higginson, Elastohydrodynamic Lubrication, Pergamon Press, London (1977).
28. B. J. Hamrock and D. Dawson, Trans. ASME F, 99 (1977), 264.

## FIGURE CAPTIONS

Figure 1. A schematic depiction of the IBAU system used in this study.

Figure 2. Friction coefficient of  $\text{Si}_3\text{N}_4$  pin during sliding against uncoated and silver-coated  $\text{Si}_3\text{N}_4$  disk with X-1P liquid lubrication at room temperature.

Figure 3. Friction coefficient of  $\text{Si}_3\text{N}_4$  pin during sliding against uncoated and silver-coated  $\text{Si}_3\text{N}_4$  disk with X-1P liquid lubrication at 100°C.

Figure 4. Friction coefficient of  $\text{Si}_3\text{N}_4$  pin during sliding against uncoated and silver-coated  $\text{Si}_3\text{N}_4$  disk with X-1P liquid lubrication at 200°C.

Figure 5. Friction coefficient of  $\text{Si}_3\text{N}_4$  pin during sliding against uncoated and silver-coated  $\text{Si}_3\text{N}_4$  disk with X-1P liquid lubrication at 300°C.

Figure 6. Variation of friction coefficients of  $\text{Si}_3\text{N}_4$  pins during sliding against (a) uncoated and (b) silver-coated  $\text{Si}_3\text{N}_4$  disks as a function of increasing temperature

Figure 7. (a) Low-magnification scanning electron micrograph and (b) 3-D surface map of a wear scar formed on  $\text{Si}_3\text{N}_4$  pin during oil-lubricated sliding test with an uncoated  $\text{Si}_3\text{N}_4$  disk at 300°C (Magnification, 50X).

Figure 8. Scanning electron micrograph of wear scar formed on  $\text{Si}_3\text{N}_4$  pin during sliding test at 300°C. This figure shows the morphology of a typical microcrack formed on the contact surface.

Figure 9. (a) Low and (b) high-magnification scanning electron micrographs of a wear track formed on silver-coated  $\text{Si}_3\text{N}_4$  disk during oil-lubricated sliding test at 100°C.

Figure 10. Average wear rates of  $\text{Si}_3\text{N}_4$  pins during sliding against uncoated and silver-coated  $\text{Si}_3\text{N}_4$  disks at different test temperatures. Values in parentheses indicate the final nominal contact pressures (MPa). Initial mean Hertzian pressure was 166 MPa.

Figure 11. (a) Scanning electron micrograph and (b) 3-D surface map of wear scar formed on  $\text{Si}_3\text{N}_4$  pin during sliding against silver-coated  $\text{Si}_3\text{N}_4$  disk at 300°C.

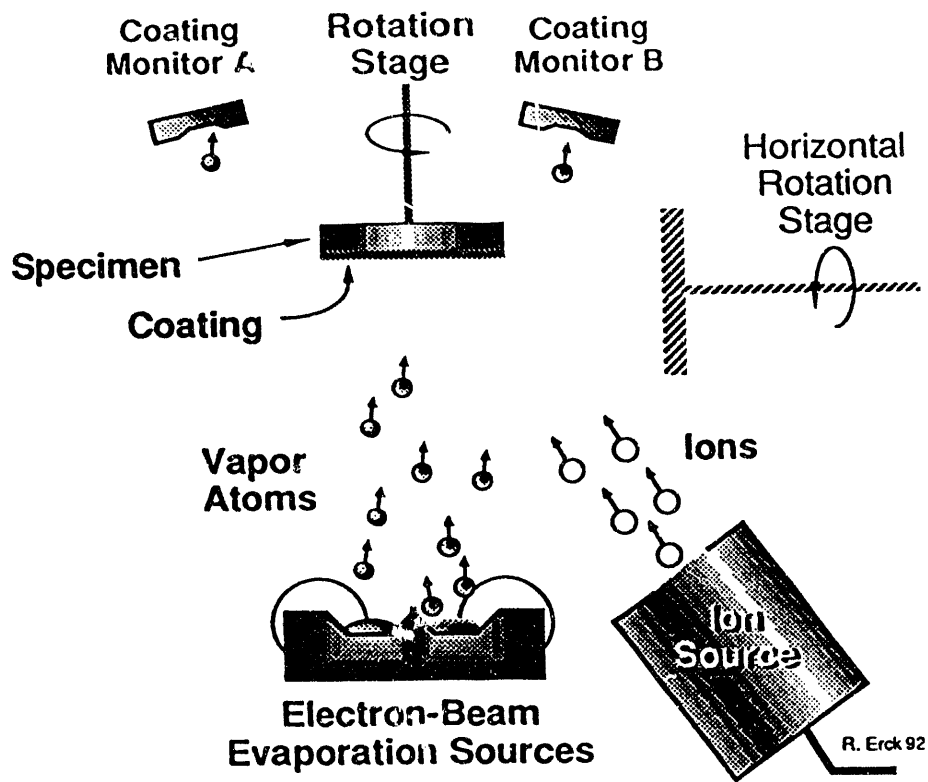


Figure 1. A schematic depiction of the IBAD system used in this study.

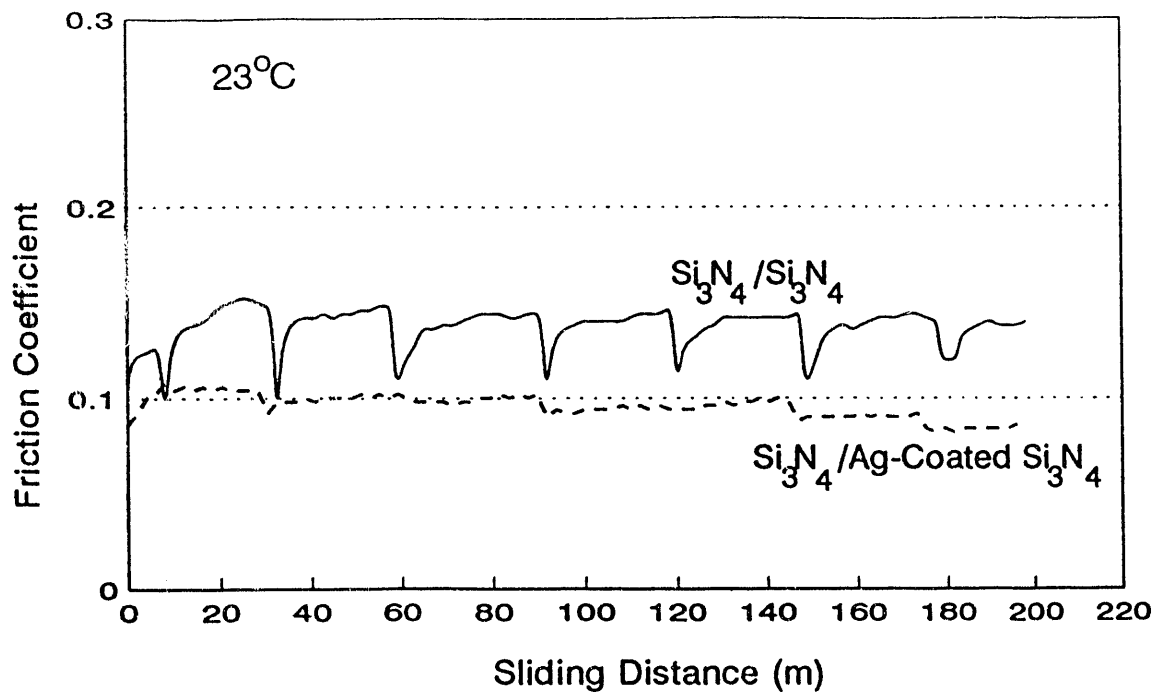


Figure 2. Friction coefficient of  $\text{Si}_3\text{N}_4$  pin during sliding against uncoated and silver-coated  $\text{Si}_3\text{N}_4$  disk with X-1P liquid lubrication at room temperature.

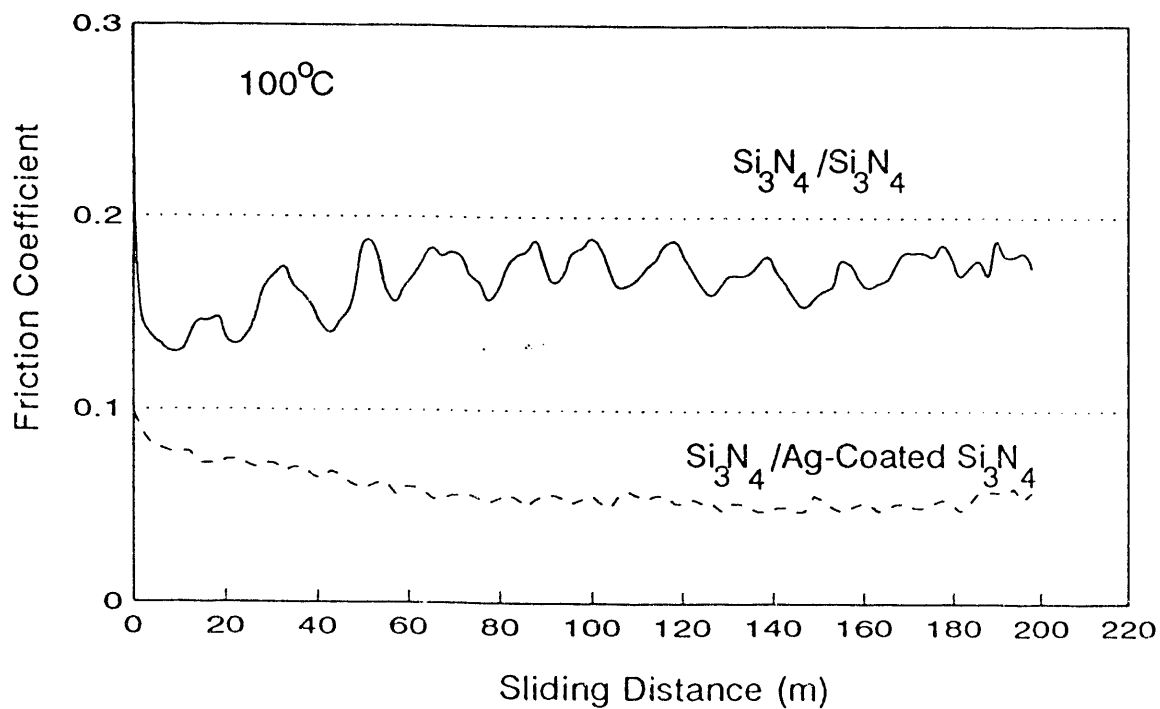


Figure 3. Friction coefficient of  $\text{Si}_3\text{N}_4$  pin during sliding against uncoated and silver-coated  $\text{Si}_3\text{N}_4$  disk with X-1P liquid lubrication at 100°C.

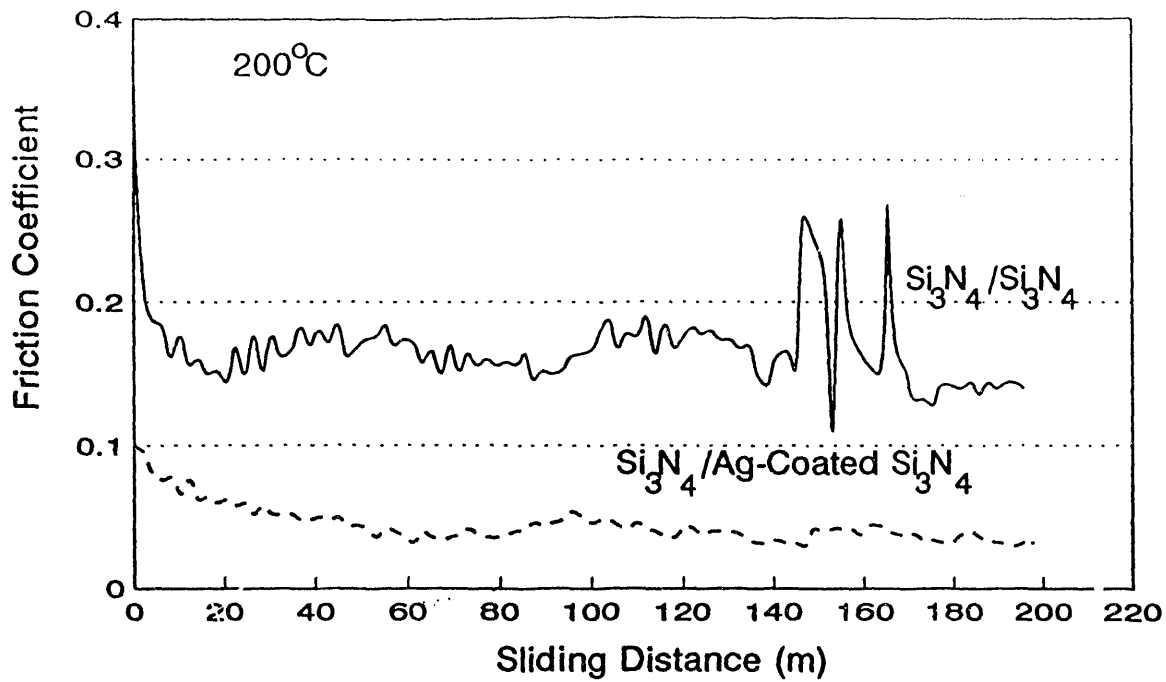


Figure 4. Friction coefficient of  $\text{Si}_3\text{N}_4$  pin during sliding against uncoated and silver-coated  $\text{Si}_3\text{N}_4$  disk with X-1P liquid lubrication at 200°C.

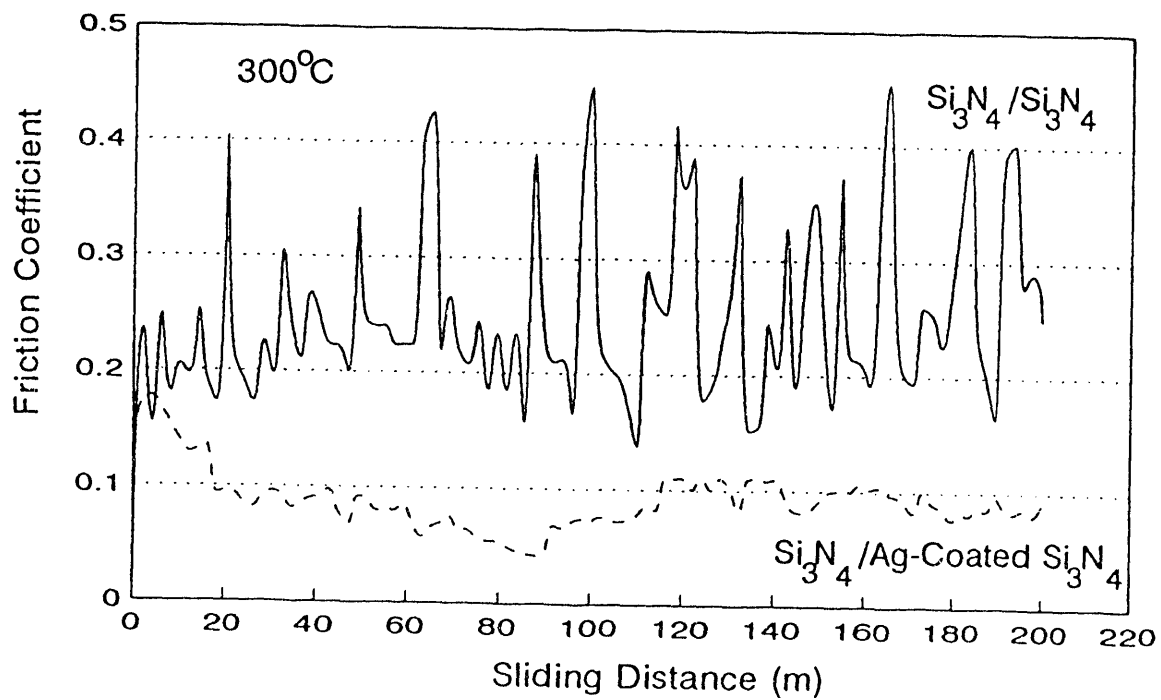
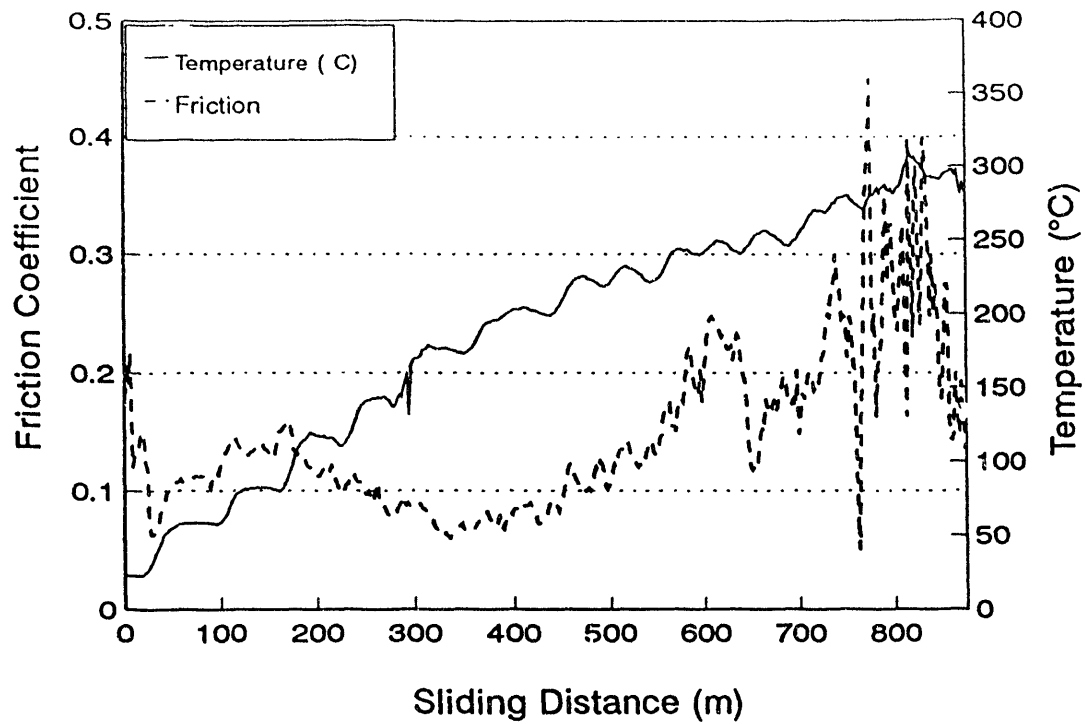
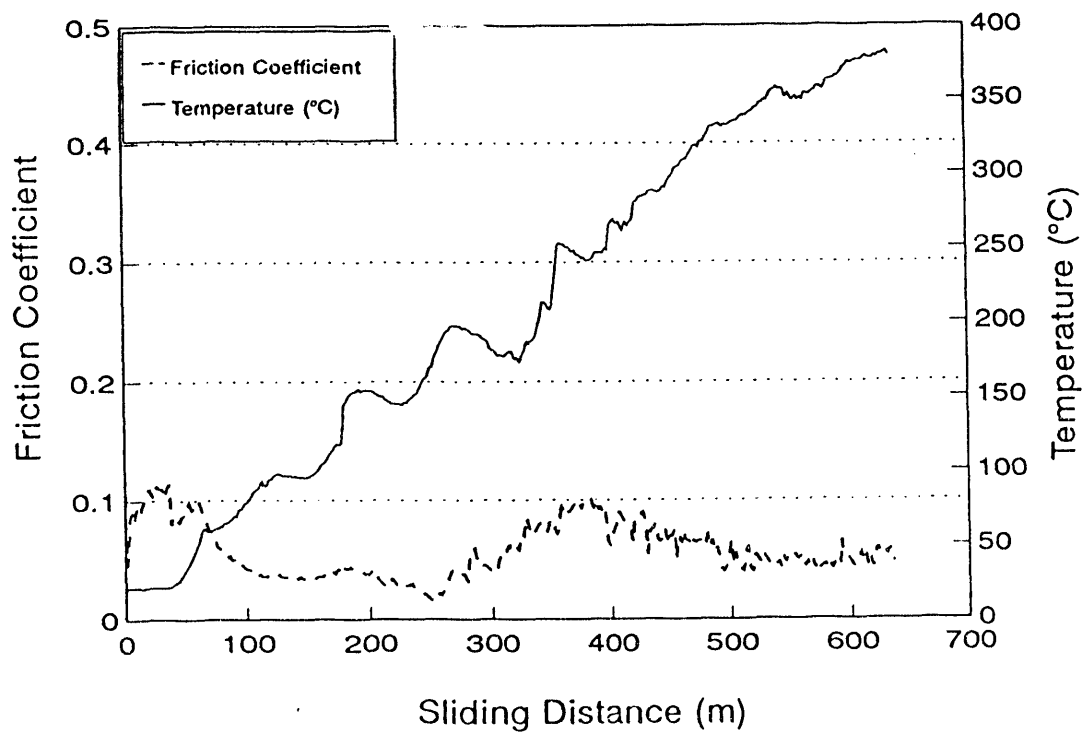


Figure 5. Friction coefficient of  $\text{Si}_3\text{N}_4$  pin during sliding against uncoated and silver-coated  $\text{Si}_3\text{N}_4$  disk with X-1P liquid lubrication at 300°C.



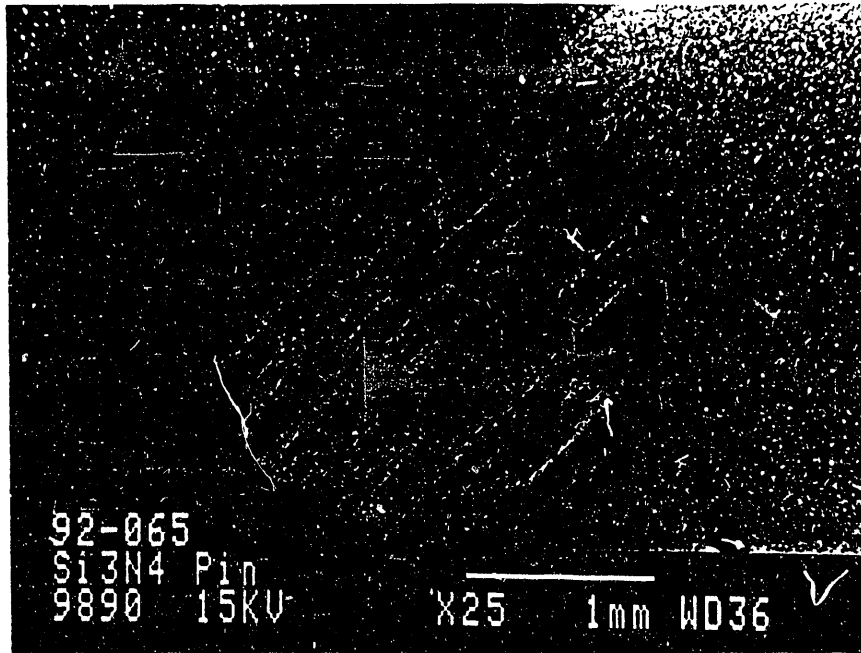


(a)

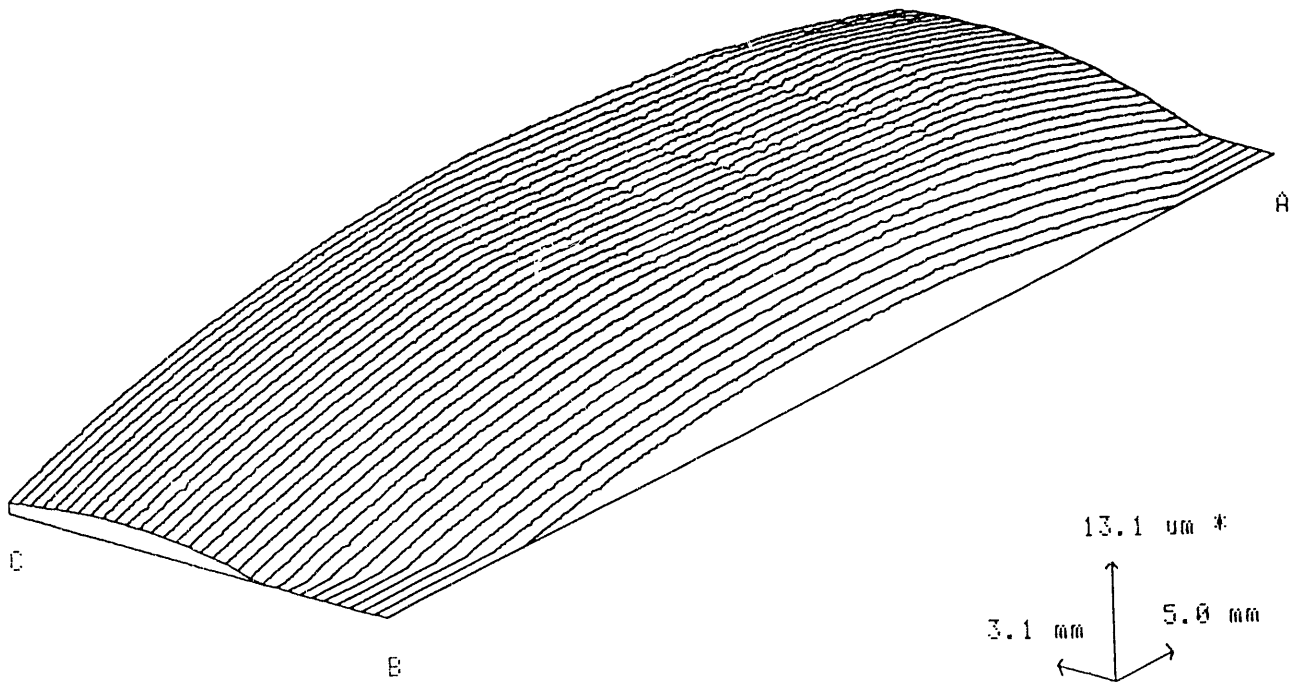


(b)

Figure 6. Variation of friction coefficients of  $\text{Si}_3\text{N}_4$  pins during sliding against (a) uncoated and (b) silver-coated  $\text{Si}_3\text{N}_4$  disks as a function of increasing temperature.



(a)



(b)

Figure 7. (a) Low-magnification scanning electron micrograph and (b) 3-D surface map of a wear scar formed on Si<sub>3</sub>N<sub>4</sub> pin during oil-lubricated sliding test at 300°C (Magnification, 50X).

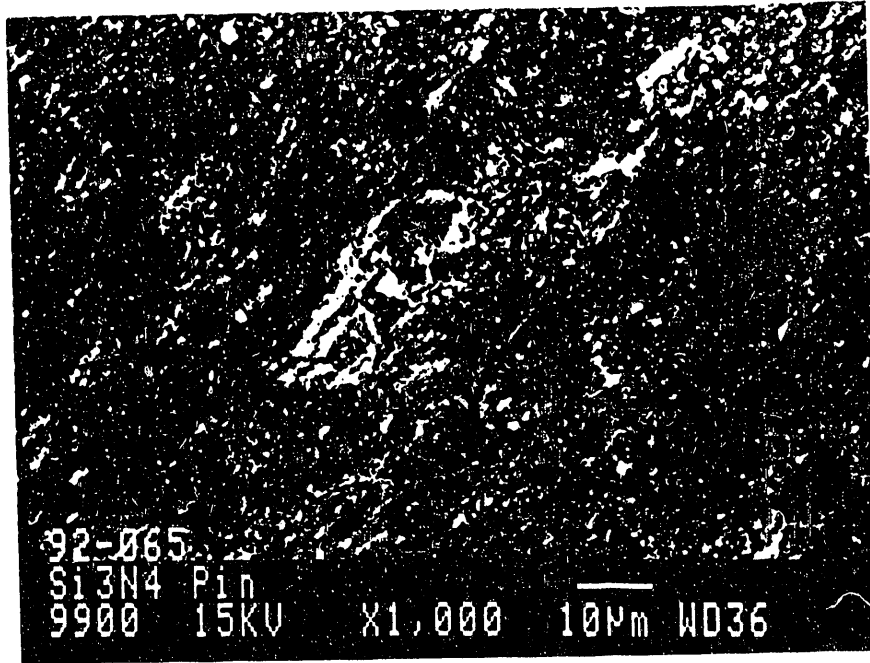
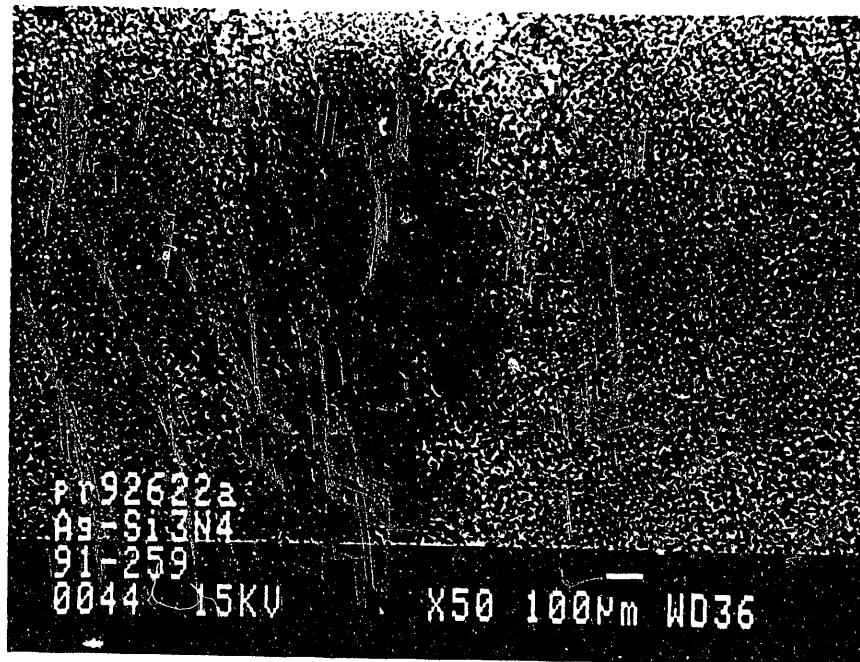
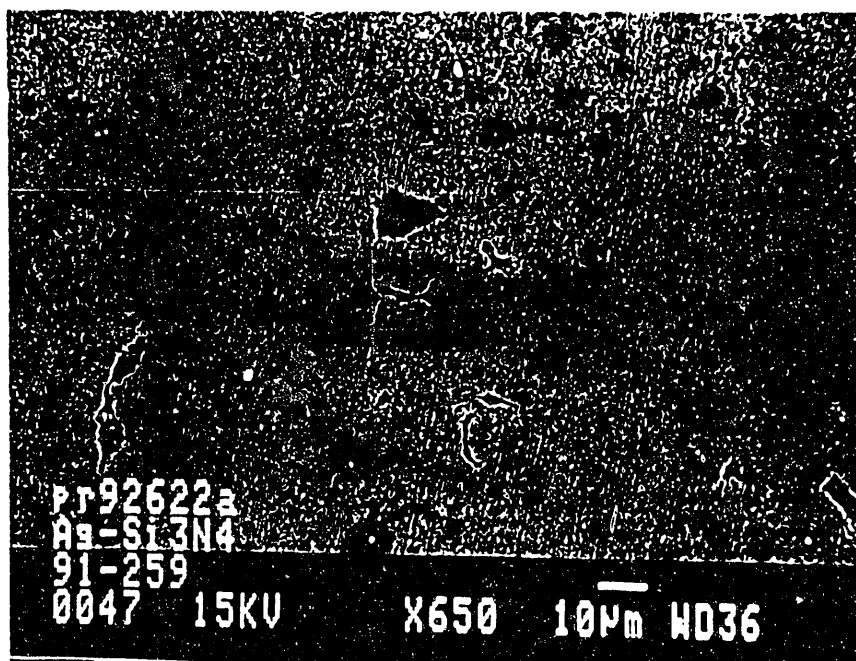


Figure 8. Scanning electron micrograph of wear scar formed on  $\text{Si}_3\text{N}_4$  pin during sliding test at  $300^\circ\text{C}$ . This figure shows the morphology of a typical microcrack formed on the contact surface.



(a)



(b)

Figure 9. (a) Low and (b) high-magnification scanning electron micrographs of a wear track formed on silver coated  $\text{Si}_3\text{N}_4$  disk during oil-lubricated sliding test at  $100^\circ$ .

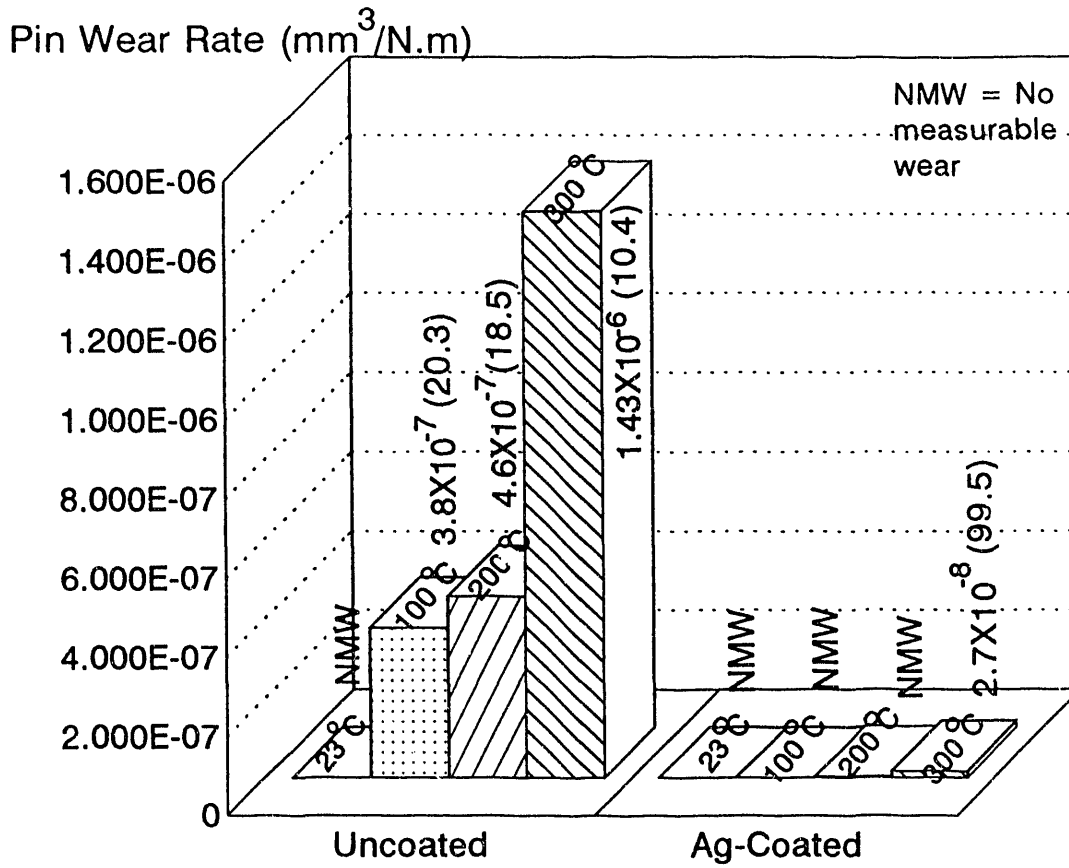
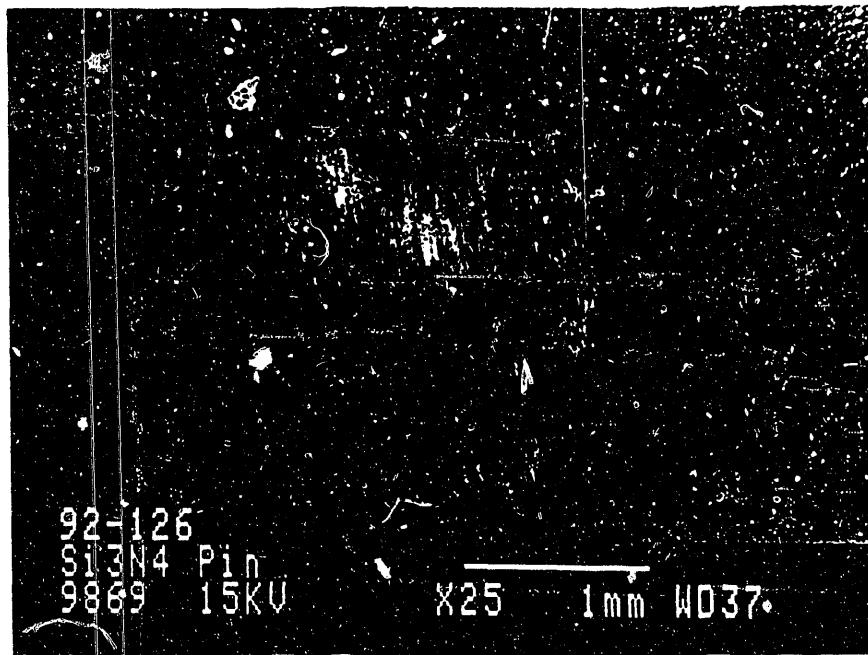
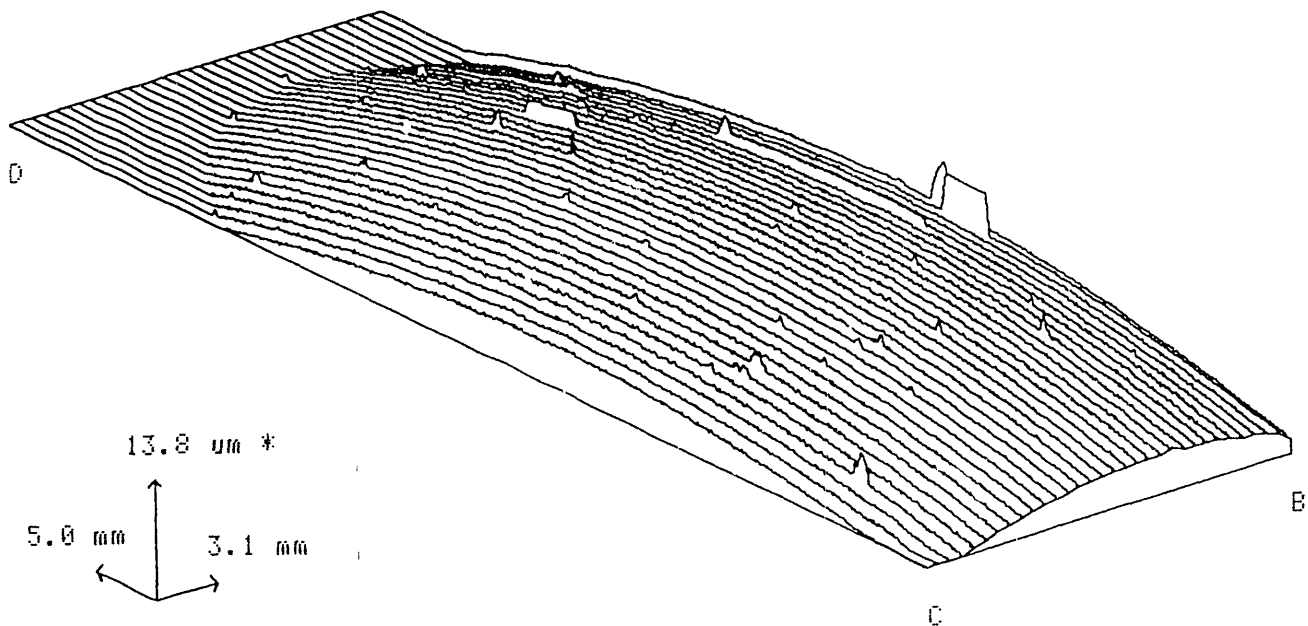


Figure 10. Average wear rates of  $\text{Si}_3\text{N}_4$  pins during sliding against uncoated and silver-coated  $\text{Si}_3\text{N}_4$  disks at different test temperatures. Values in parentheses indicate the final nominal contact pressures (MPa). Initial mean Hertzian pressure was 166 MPa.



(a)



(b)

Figure 11. (a) Scanning electron micrograph and (b) 3-D surface map of wear scar formed on Si<sub>3</sub>N<sub>4</sub> pin during sliding against silver-coated Si<sub>3</sub>N<sub>4</sub> disk at 300°C.

**END**

**DATE  
FILMED**

4 / 20 / 93

