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Tribological Properties of Ag/Ti Films on Al_2O_3 Ceramic Substrates

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Christopher DellaCorte, Stephen V. Pepper,
and Frank S. Honecy
Lewis Research Center
Cleveland, Ohio

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TRIBOLOGICAL PROPERTIES OF Ag/Ti FILMS ON Al_2O_3 CERAMIC SUBSTRATES

by

C. DellaCorte, S.V. Pepper, and F.S. Honecy
National Aeronautics and Space Administration
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135

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ABSTRACT

Ag solid lubricant films, with a thin Ti interlayer for enhanced adhesion, were sputter-deposited on Al_2O_3 substrate disks to reduce friction and wear. The dual Ag/Ti films were tested at room temperature in a pin-on-disk tribometer sliding against bare, uncoated Al_2O_3 pins under a 4.9 N load at a sliding velocity of 1 m/sec.

The Ag/Ti films reduced the friction coefficient by 50 percent to about 0.41 compared to unlubricated baseline specimens. Pin wear was reduced by a factor of 140 and disk wear was reduced by a factor of 2.5 compared to the baseline. These films retain their good tribological properties including adhesion after heat treatments at 850 °C and thus may be able to lubricate over a wide temperature range. This lubrication technique is applicable to space lubrication, advanced heat engines, and advanced transportation systems.

INTRODUCTION

Current research efforts related to hypersonics, advanced heat engines and space power generation have emphasized the need for lightweight high temperature materials. Ceramics show promise for fulfilling many of these needs due to their low density, high hardness and relatively high degree of thermal and chemical stability (Ref. 1). However, many anticipated applications for ceramics include rubbing or sliding contact. Since recent research indicates that unlubricated ceramic friction and wear performance is poor, the lubrication of ceramics is critical to their successful implementation (Ref. 2).

There are several different approaches to lubricate ceramic surfaces. These include conventional liquid lubrication, solid film lubrication and surface modification techniques. Conventional liquid lubrication is limited to relatively low operating temperatures (<350 °C) due to the inherent thermal instability of available oils (Ref. 3). Surface modification usually involves some sort of implantation or diffusion of ions or atoms into the surface to improve toughness and hence wear properties, or to change the surface chemistry to improve friction and wear properties (Refs. 4 to 6). But surface modification techniques can be costly and have limited lifetimes for many applications.

Solid film lubrication, therefore, is a common approach to lubricate ceramics. Traditional solid lubricants include burnished or sputtered graphite or MoS_2 and soft metallic

films. These lubricants function by providing a low shear strength layer between the sliding surfaces to reduce friction and wear.

In general, solid lubricants do a good job of protecting rubbing surfaces. However, poor adhesion of the lubricant coating to the substrate can limit coating life by causing coating delamination and failure. This is especially true when very stable lubricants such as PTFE or metallic gold films are applied to ceramics such as SiO_2, Al_2O_3, etc.

One way to improve adhesion and wear life of solid lubricants to ceramics is to combine Physical Vapor Deposition (PVD) techniques with an ion beam as in ion beam assisted deposition (IBAD). Erdemir et al. (Ref. 7) have pioneered the use of IBAD silver deposited films onto Al_2O_3 to improve sliding friction and wear up to 400 °C. The IBAD process provides a dense film which is in intimate contact with the substrate. Improved adhesion seems to be due to a continuous lubricant/substrate interface largely free of voids or contaminants. Although these films produced by the IBAD technique are tribologically successful, the deposition system is fairly complicated and expensive to operate. Also, at temperatures above 500 °C, the silver films have a tendency, through surface diffusion, to dewet the ceramic and "ball-up" compromising their lubricating ability.

One way to reduce silver's tendency to dewet at elevated temperatures and also to produce coatings in a simpler and

less costly manner than IBAD is to use sputter deposition techniques and enhance adhesion by introducing an intermediate bond layer or coating between the substrate and the lubricant coating. Ideally, this layer should bond well to both the ceramic and the silver lubricating film without adversely reacting with or dewetting the lubricant over a wide temperature range.

Active transition metals, such as titanium, chromium, and copper have been used in the past to join or braze metals to oxide ceramics like Al_2O_3 (Refs. 8 to 11). These metals function by reacting with the oxide substrate to form their own oxides and thus are well adherent yet they also retain their metallic nature at the free surface or interface to bond well to the overlay coatings such as lubricant films of silver.

The method used in our study to lubricate ceramics over a wide temperature range, therefore, is to use a thin (≈250Å) sputtered Ti bond layer between an Al_2O_3 substrate and a thick (1.5 μm) silver lubricant layer. The titanium acts as a bond layer to improve adhesion, and the silver, due to its low shear strength, provides lubrication for the ceramic to reduce friction and wear. Also in this case, at elevated temperatures, silver wets titanium and hence the films are stable and will not dewet the Al_2O_3 substrate.

To test this lubrication method, wear disks of Al_2O_3 were coated with the Ag/Ti films and slid against uncoated Al_2O_3 pins in a pin-on-disk tester at a temperature of 25 °C. Some

samples were heat treated in Argon at 850 °C to determine the effects of high temperature on the tribological performance and wetting characteristics of the Ag/Ti films.

MATERIALS

The tests conducted in this study were pin-on-disk tests. Both the pins and the disks were made from Al_2O_3 (99.4 percent pure). The properties of the Al_2O_3 and composition are given in Table 1. The commercially obtained specimens were fabricated using cold pressing and sintering followed by finish grinding and polishing. The wear surfaces were finish polished to approximately 0.1 μm rms. The pins were hemispherically tipped with a 2.54 cm radius of curvature and were tested bare, with no coatings. The disk wear surfaces were flat. Some were sputter coated with titanium and silver as adhesion enhancing and lubricating films respectively. As a control sample, some silver films were deposited without a titanium bond layer.

The films were sputter deposited as follows: first the disks were sputter cleaned using argon ions at 0.500 kW at 20 mtorr for 5 min; titanium was then deposited at 0.500 kW, 1.0 Pa to a thickness of 250±20Å (as determined from a quartz standard), finally a 1.5 μm thick silver film was sputtered over the titanium layer at 0.500 kW, 1.0 Pa. The base pressure of the chamber was $\leq 7 \times 10^{-5}$ Pa. For comparison, some samples were made without the Ti films (only the Ag film) or were bare, sputter cleaned Al_2O_3 samples. Figure 1 shows an optical micrograph of a typical Ag/Ti film. The films contain

some porosity which is predominantly due to the surface pits and roughness of the alumina substrate.

PROCEDURES

Prior to testing, the pin specimens were cleaned with ethyl alcohol, scrubbed with levigated alumina and water to remove any surface contaminants, rinsed with deionized water and dried. Some of the coated disks were heat treated in an argon atmosphere at 850 °C for 1 hr to promote diffusion/ reactions between the Al_2O_3 substrate and the titanium film in an effort to enhance adhesion. An inert atmosphere was chosen to prevent either gross oxidation of the titanium film or reduction or hydriding as might occur if heated in a hydrogen atmosphere. This heat treatment also serves as a test of the film wettablity at an elevated temperature.

The pin-on-disk test rig used in this study, including its operation and its data acquisition system are described in detail in Ref. 12. The following is a brief description of the rig and the test procedures.

To test the specimens, the disk was first mounted onto the test rig spindle. Total indicated run-out (T.I.R.) for the face was measured and kept to less than 0.025 mm to reduce dynamic loading forces during the test. The pin specimen was then mounted into its holder and aligned. The tests were run in ambient air at 1 m/sec sliding velocity (370 rpm) under a 4.9-N load. Testing for each specimen typically lasted 30 min although some specimens were run for up to 90 min of sliding.

During the test, friction force, load force, specimen temperature and test humidity were recorded.

After testing, the specimens were removed and wear measurements were made using optical microscopy (to measure pin wear scar diameters) and stylus surface profilometry (to measure disk wear). To gain a better understanding of the wear process SEM/EDS X-ray analysis was also performed. Auger depth profiles were done on coated samples before and after heat treatments to help ascertain the effects of the heat treatment on the films.

RESULTS

Tribotests

The friction and wear results, averaged over the 30 min tests, are shown in Table II and plotted in Fig. 2. For the following discussion, baseline data refers to tests of unlubricated alumina disks in sliding against alumina pins. Control data refers to silver coated disks, without the titanium underlayer, sliding against alumina pins. Ag/Ti films refers to disk specimens coated with both the titanium underlayer and the silver lubricating layer.

The friction for the Ag/Ti films is 40 to 60 percent below the friction of the baseline unlubricated specimens. Pin wear for the Ag/Ti film coated Al_2O_3 is very low, $1.1 \pm 4 \times 10^{-8}$ $mm^3/N\text{-}m$ and is about 140 times lower than the unlubricated baseline case.

The silver films tested without the titanium bond layers, the control specimens, initially showed low friction (≈ 0.4)

but by the end of 1800 m of sliding (30 min) the friction increased dramatically (to about 0.7) indicating that the coating was failing. Pin wear was an order of magnitude higher than the Ag/Ti films. Therefore, without the Ti bond layer the silver film life is not adequate.

Disk wear for the Ag/Ti coated Al_2O_3 disks is about 2.5 times lower than the baseline. Surface profilometry and microscopic analyses indicated that very little Al_2O_3 is worn from the disk surface and most of the wear is made up of the soft silver coating. Therefore, the Ag/Ti films provide protection from wear to the Al_2O_3 ceramic surfaces during sliding.

Also shown in Table II is the friction and wear data for Ag/Ti samples which had been heat treated in argon for 1 hr at 850 °C. Although the friction and wear are slightly higher than the unheated specimens, the films are not visibly changed by the heat treatment. This indicates that the Ag/Ti films will probably be durable and functional during and after elevated temperature use. It is important to note that the films did not dewet or "ball up" during the heat treatment as shown in Fig. 3 for Ag films without the Ti adhesion layer. This is a further indication of the potential for high temperature use of the films.

Auger depth profiles for the Ag/Ti films prior to and after heat treatment show that there is some intermixing of the films and between the Ti film and the Al_2O_3 substrate. However, the films remain essentially intact and, as evidenced

from the tribodata, still provide a significant lubrication effect even after the 850 °C heat treatment.

Figures 4 and 5 show SEM micrographs of worn specimens. When sliding against the Ag/Ti films, silver transfers to the pin surface forming a lubricating film, Figure 6. This helps reduce the friction and wear. Figures 7(a) to (d) shows X-ray dot maps of Ag, Ti and Al on the disk wear surface. Even after 1800 m of sliding, the predominant surface film is Ag. Holes in the film appear as bright areas of Al signal. Ti can be seen at the edges of the "holes." These edge outlines in the titanium dot map (Fig. 7(c)) were confirmed as titanium rather than an analysis artifact by zooming in on the edges at higher magnification and obtaining the complete spectrum showing the Ti signal. From the micrographs it can be inferred that the layered film structure remains in the wear area with the Ag film providing lubrication.

DISCUSSION

Silver has been used as a solid lubricant for many years (Ref. 13). It is a soft metal which provides lubrication in sliding contacts by plastically deforming or shearing. Yet, silver retains enough compressive strength to support loads and reduce wear. Silver is also relatively stable both chemically and thermally at low and high temperatures. For these reasons, silver is a good solid lubricant.

In order to be used as a solid film lubricant, the film must adhere to the surface to be lubricated. Since the adhesion of relatively inactive metals like silver or gold to

oxide ceramics such as Al_2O_3 is poor, techniques to improve adhesion must be employed. The technique employed in this study is the use of a titanium "bond coat" layer between the silver film and the Al_2O_3 substrate. Titanium is a very active metal which bonds readily to Al_2O_3, possibly forming some type of TiO_2-Al_2O_3 compound. By depositing more than a few mono-layers of titanium, even though the interface between the Ti and Al_2O_3 becomes a complex oxide, the outer free surface of the titanium film is thought to remain metallic in nature allowing an adherent metallic bond with the Ag film.

Another benefit of using an intermediate bond layer such as titanium is high temperature wettability. Silver does not wet Al_2O_3. At temperatures above 400 °C, a continuous silver film in contact with Al_2O_3 will diffuse into "islands" and will eventually dewet or "ball up" on the surface, no longer acting as a lubricant film. Other researchers have observed the dewetting phenomena even with Ag films energetically deposited on Al_2O_3 using the IBAD process (Ref. 7). By employing an intermediate layer of Ti which wets both Ag and Al_2O_3, the Ag film does not dewet even after 1 hr heat treatments at 850 °C. See Fig. 3. Therefore, the use of Ag/Ti films may be extended to temperatures above 400 °C.

As a qualitative measure or test of the adherence of the Ag/Ti films, the wear mechanism can be examined. SEM analyses indicate that the predominant substrate wear mechanism occurring is grain pull out of the alumina substrate. See Figs. 4 and 5. This observation shows that the bond between the

silver and titanium and between the titanium and the alumina is good.

The secondary wear mode, due to plastic deformation of the silver film, is a gradual film thinning which results in a slow increase in the friction coefficient. During sliding, the friction coefficient is about 0.4. After long duration test (≈5 to 10 km of sliding), the friction coefficient slowly increases to about 0.55. Subsequent analyses of the wear track on the disk show that the films have worn thin and high spots of the alumina substrate are present on the surface increasing friction and wear. The gradual thinning of the silver lubricating film (as determined with EDS analyses adjacent to the alumina "high spots") combined with the excellent adhesion enhancement of the titanium film contribute to the graceful wearing out of the system. This slow graceful failure is a desirable engineering feature as it can be easily monitored compared to catastrophic type failure such as coating delamination.

Also, an examination of the worn pin specimens indicates that Ag is transferred to the pin wear scar also providing lubrication. This transferred silver is probably mechanically bonded to the wear scar surface and would not necessarily adhere during high temperature testing. Nonetheless, its presence is indicative of a lubricating layer helping to protect the ceramic from wear.

At first glance it is difficult to understand how a soft film of silver could cause the wear of the Al_2O_3 counterface

pin. However, the pin wear is probably due to the abrasivity of the pulled out Al_2O_3 wear particles as well as any Al_2O_3 high points or asperities in the wear track. Even though the pin does sustain a finite amount of wear it is over two orders of magnitude lower than the unlubricated baseline case.

The tribodata indicates that the disk wear is not reduced by use of the silver/Ti films nearly as much as the pin wear. In fact, the disk wear is only reduced by a factor of about 2.5. This is partly due to the fact that the disk wear volume includes the soft Ag/Ti film wear and can not distinguish the amount of actual substrate wear from the film wear. Therefore, when judging the effectiveness of the lubricity of the Ag/Ti films, it is more appropriate to consider the pin wear.

It has been discussed that the titanium adhesion layer prevents dewetting of the Ag film at elevated temperatures. This was evidenced by the results of static heat treatments in argon at 850 °C. Auger analyses before and after heating indicate that some interdiffusion between the Ag and Ti does also occur during the heat treatment. The changes, however, are small. The triboperformance for the heat treated films, although not as good as for films which were not heated, indicates that the heat treated films are still tribologically useful. Friction is reduced by 35 to 40 percent, pin wear by a factor of about 20 and disk wear by a factor of about 2 compared to the unlubricated baseline case. These results indicate that the use of the Ag/Ti films may be possible to temperatures as high as 850 °C and will be

reported in the future.

At this point, some mention should be made regarding the friction coefficient measured for the Ag films and how they compare to theoretically expected values. When a thin lubricating layer is deposited onto a hard substrate, the friction coefficient can be approximated as the ratio of the bulk shear strength of the lubricating layer to the contact pressure or:

$$\mu = S/P_H$$

S shear strength of lubricating film in GPa

P_H contacting pressure (average Hertzian pressure) in GPa

This equation assumes that the ploughing component of the friction is small and that the ceramic substrate and pin deform elastically. The derivation of this equation can be found in Refs. 14 and 15.

To arrive at a value for the friction coefficient, one must know the contact pressure during the sliding tests as well as the bulk shear strength of the film. The contact pressure can be approximated from the Hertz equations (Refs. 16 and 17). Initially the contact pressures will be nearly Hertzian. For our tests, the initial contact pressure, P_H, is about 0.38 GPa. As wear occurs on the hemispherical pin, the apparent contact area will increase, dramatically lowering the contact stress below the Hertzian value.

The shear strength of silver is a function of pressure and can be obtained from bulk shear measurements made by P.W. Bridgeman during the 1940's and 1950's (Ref. 18). Using this data, the shear strength, S, is about 0.09 GPa. There-

fore, the friction coefficient is predicted to be about 0.24 which is very close to our measurements. We typically measure about 0.25 during initial sliding (Fig. 2) when the contact is approximately Hertzian and the friction coefficient increases to about 0.4 as pin wear occurs which reduces the contact stress.

Although this analysis is simplistic, it serves as a good yardstick by which to gauge our experimental results. Based upon these analyses, we can infer that our films are providing a lubricating effect through the shearing of the metallic film thereby reducing friction and wear.

CONCLUSIONS

1. Sputtered Ag/Ti films reduce the sliding friction and wear compared to unlubricated ceramics and provide lubrication by separating the ceramic surfaces allowing shear to occur at the sliding interface.

2. The titanium interlayer enhances adhesion of the silver layer as evidenced by the tribological test results which indicate that failure (wear by grain pullout) occurs within the substrate and not at the film/substrate interface.

3. The use of an active metal interlayer prevents the dewetting of the Ag lubricant film which would otherwise occur at high temperature.

4. Based upon the test results and analyses, these films may be suitable to provide lubrication for advanced applications such as engine seals, bearings and other sliding components.

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TABLE I. - PROPERTIES OF Al_2O_3 MATERIAL TESTED

Property	Value
Composition (wt %)	99.9 A_2lO_3 and trace Fe, Si
Density	3.9 g/cc
Young's modulus	386 GPa
Hv	2000 kg/mm^2
Toughness	4.2 MPa$\sqrt{m}$
Thermal expansion coefficient	8.0×10^{-6}/°C
4-Point bend strength	344 MPa
Poisson's ratio	0.23
Thermal conductivity	22 w/m K

TABLE II. - SUMMARY OF TRIBODATA

Disk specimens	Average friction coefficient	Pin wear factor	Disk wear factor
Unlubricated Al_2O_3	0.87±0.03	$150\pm30\times10^{-6}$ $mm^3/N\text{-}m$	$4.4\pm0.4\times10^{-5}$ $mm^3/N\text{-}m$
$Ag/Ti/Al_2O_3$ without heat treatment	0.41±0.04	$1.1\pm0.4\times10^{-8}$ $mm^3/N\text{-}m$	$1.7\pm0.4\times10^{-5}$ $mm^3/N\text{-}m$
$Ag/Ti/A_2lO_3$ with heat treatment	0.54±0.08	$7.8\pm2.6\times10^{-8}$ $mm^3/N\text{-}m$	$2.1\pm0.6\times10^{-5}$ $mm^3/N\text{-}m$
Ag/Al_2O_3	0.30 initially inclusive to ≈0.7	$120\pm30\times10^{-8}$ $mm^3/N\text{-}m$	Erratic (not able to measure)

Test conditions: Room temperature air, R.H. 40 to 60 percent, 1 m/sec sliding velocity, 4.9 N load. Wear factors measured after 30 min of sliding. Uncertainties represent one standard deviation for the Ag/Ti tests (without heat treatment) and data scatter for other cases.

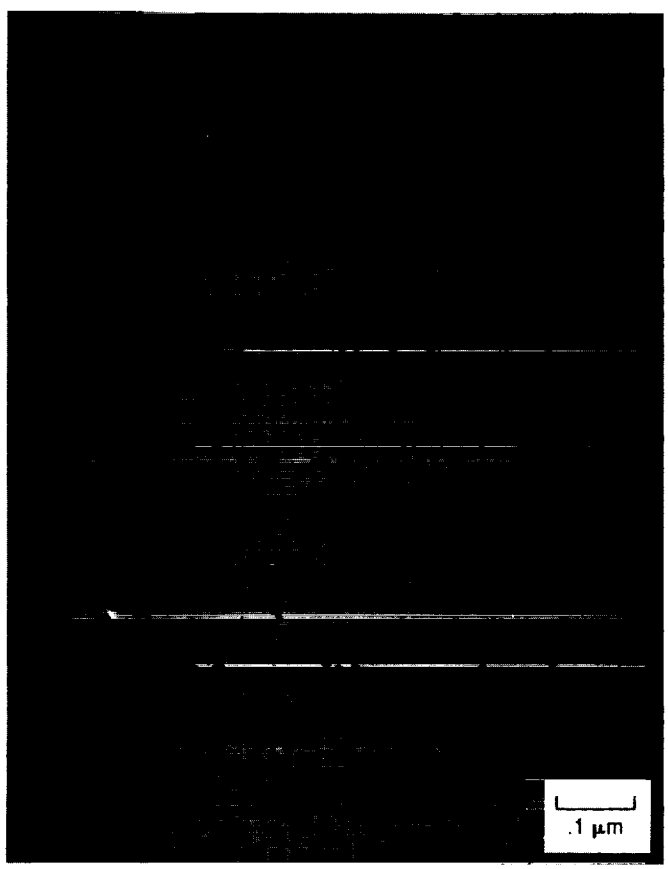

Figure 1.—Optical photomicrograph of Ag/Ti film on polished (≈ 0.1 μm rms surface finish) Al_2O_3 disk specimen. Magnification is 100x. Black spots are pores due to pits and nonuniformities of Al_2O_3 substrate surface.

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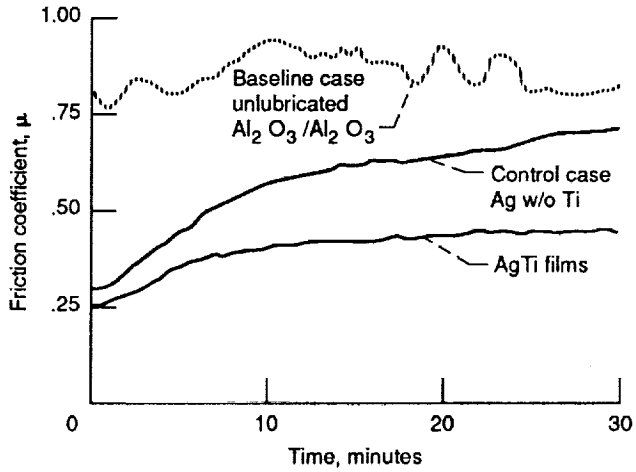

Figure 2.—Friction coefficient vs. sliding time for unlubricated, silver and silver/titanium lubricated specimens. Test velocity, 1m/s; load, 4.9N, atmosphere, ambient air, 25 °C.

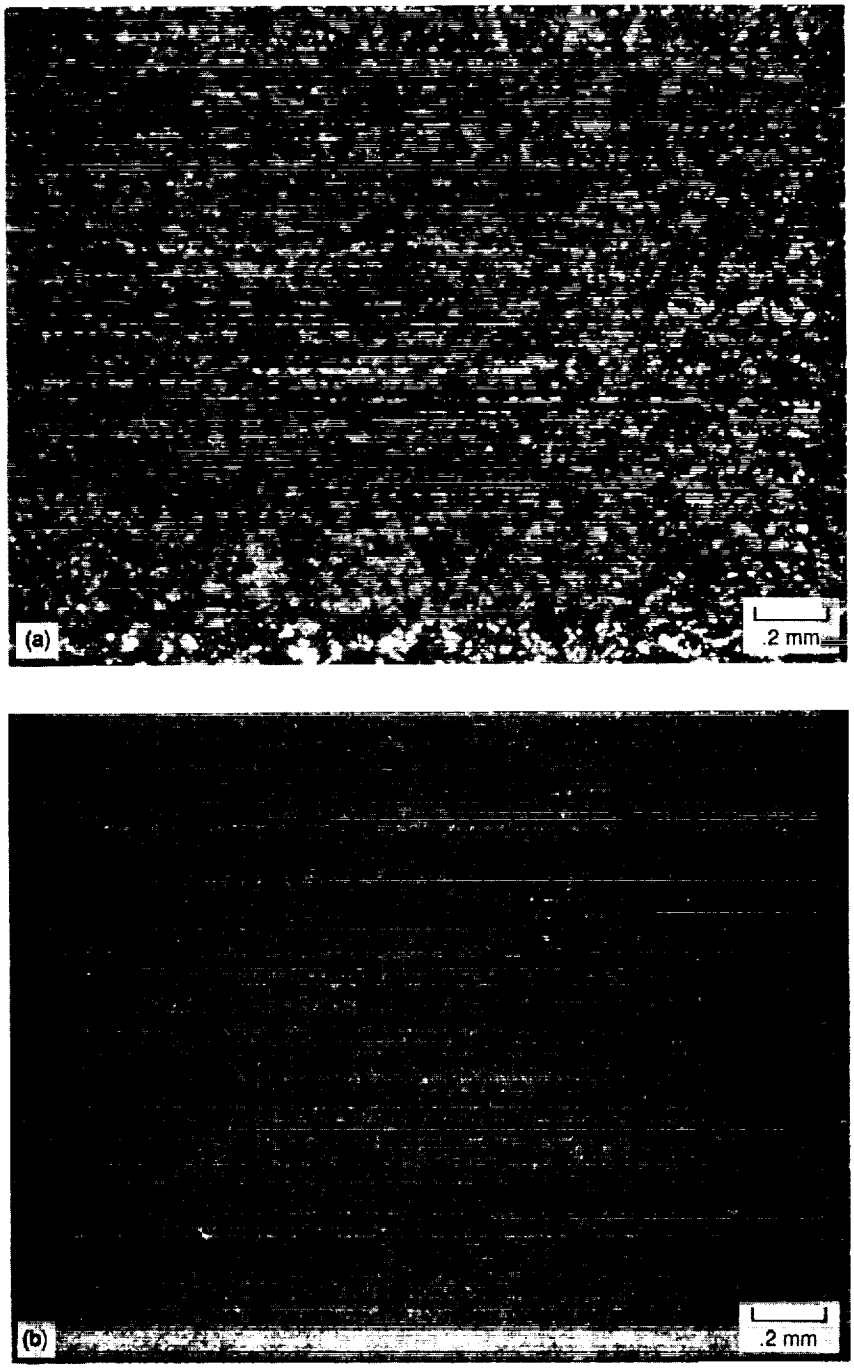

Figure 3.—Optical photomicrograph of silver coatings on Al_2O_3 substrates after heat treatment at 850 °C. In (a), control case w/o Ti interlayer show silver dewetting ("bright" spots are dewetted silver "ball"). In (b) Ag/Ti films do not dewet.

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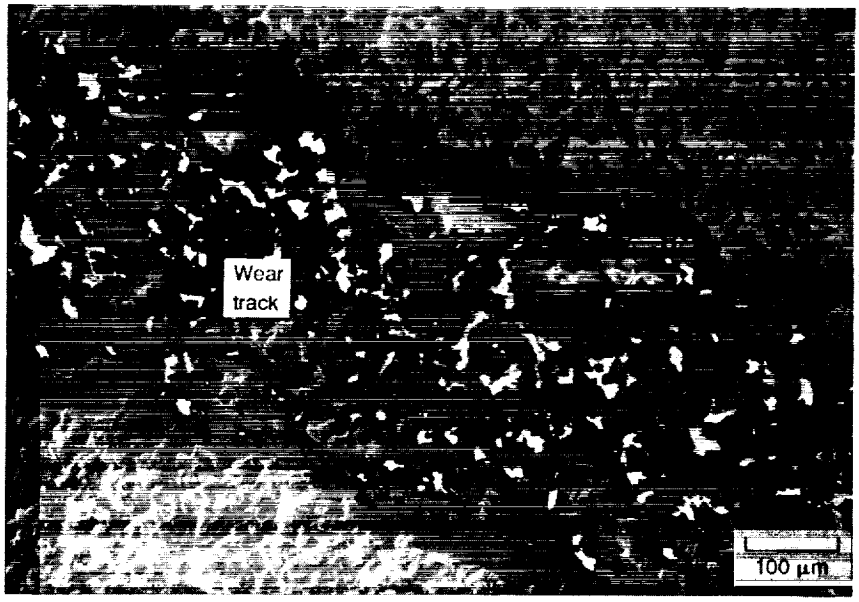

Figure 4.—SEM micrograph of worn Ag/Ti film on disk surface. Bright areas are the charging of the Al_2O_3 substrate showing through in "holes" due to surface porosity of original Al_2O_3 surface and due to grain pullout.

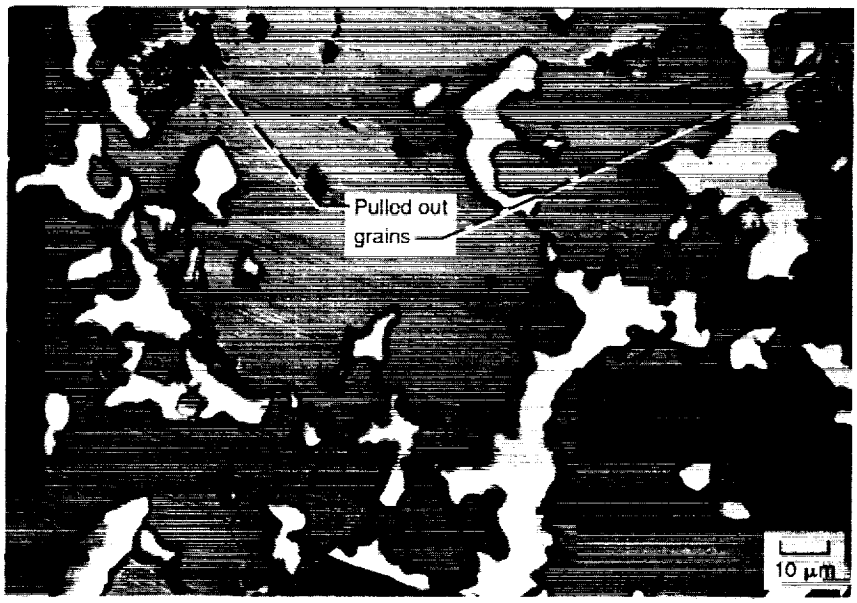

Figure 5.—Close up view of worn Ag/Ti film on disk surface. Smooth grey areas are silver lubricating film. Bright areas are Al_2O_3 "holes".

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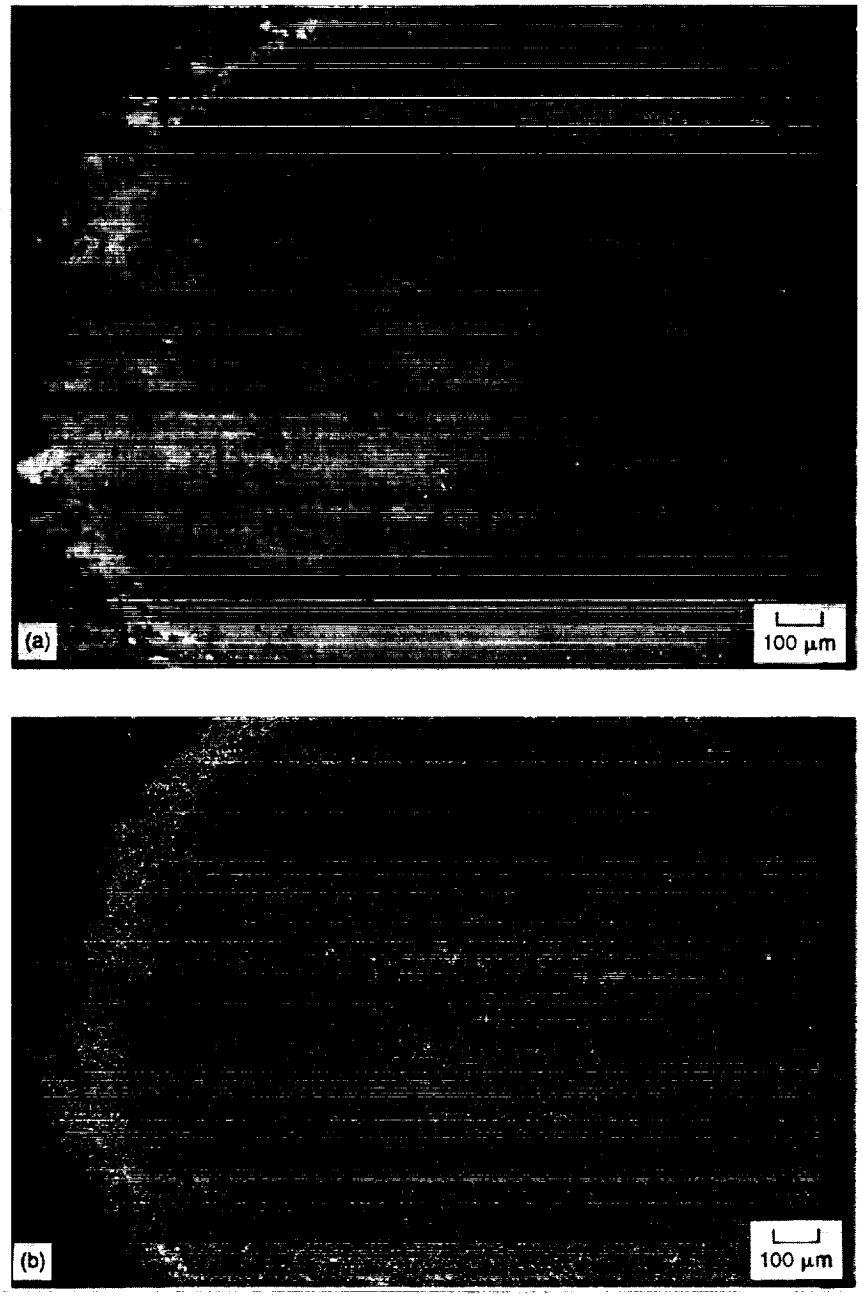

Figure 6.—Optical micrograph (a) and corresponding silver x-ray dot map (b) of pin wear scar after sliding against Ag/Ti film.

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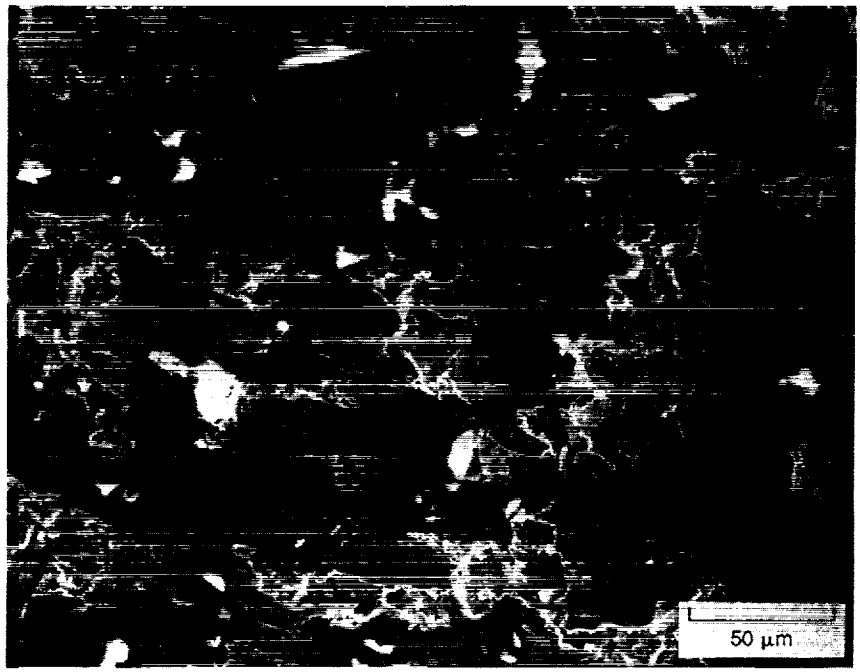
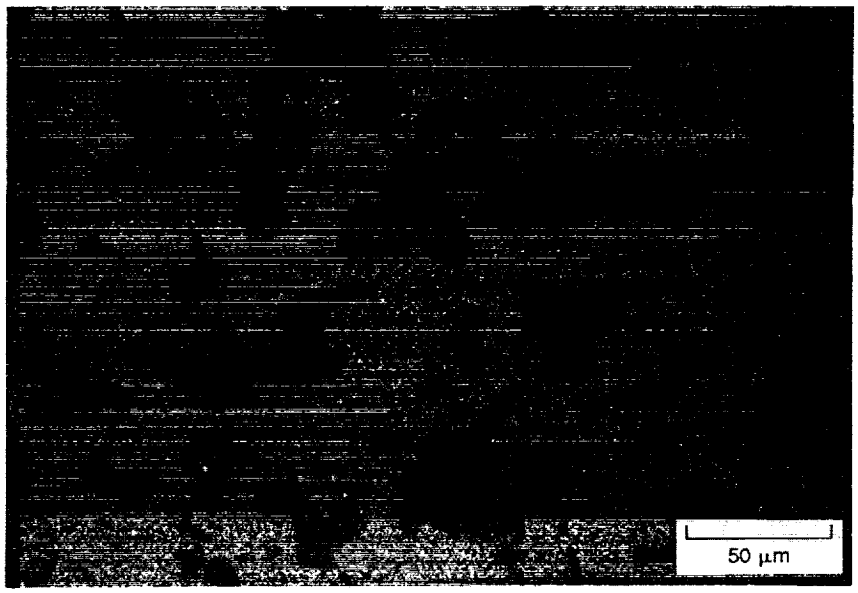


(a) Secondary electron image.



(b) Silver x-ray dot map.

Figure 7.—SEM micrographs of worn disk surface after 1800 meters of sliding. X-ray dot maps (b, c, and d) show that predominant surface film is silver

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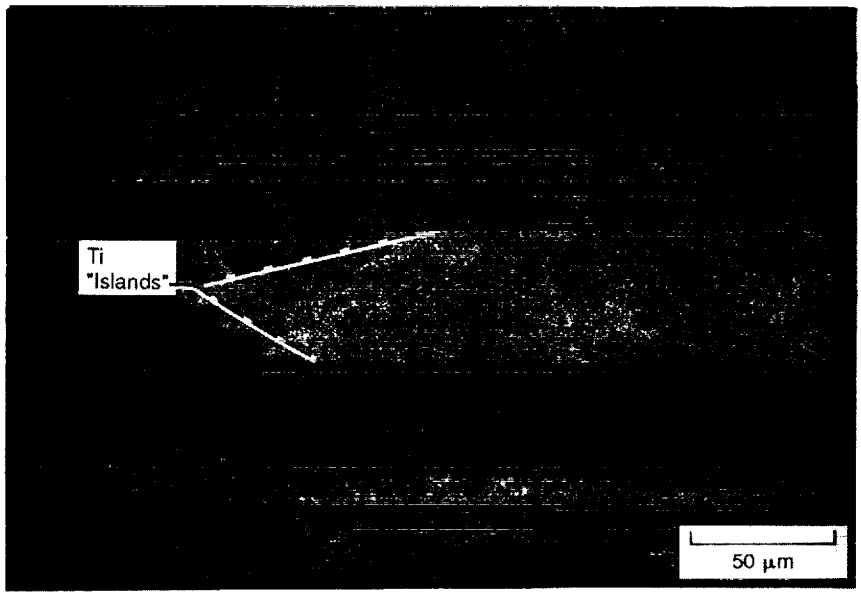
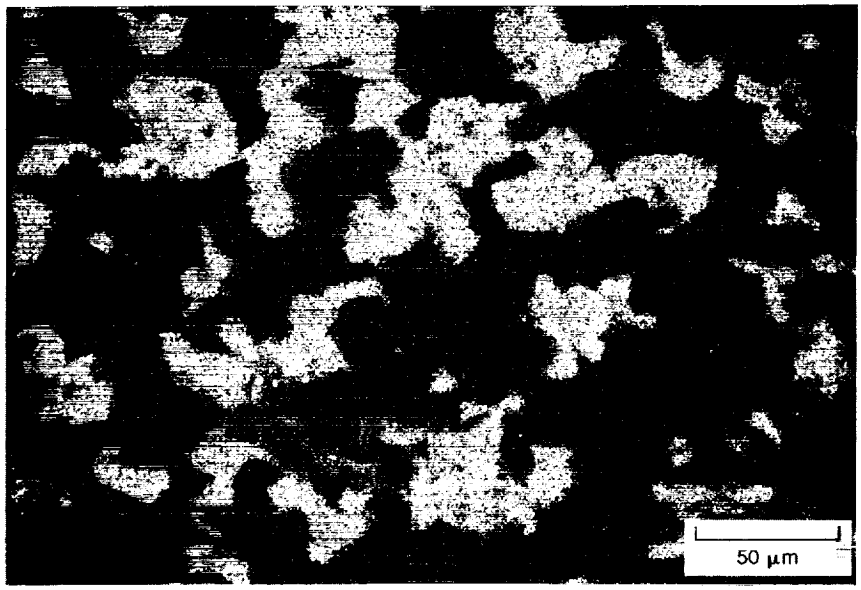


(c) Titanium x-ray dot map. Note "island rings" of titanium around boundaries of aluminum areas.



(d) Aluminum dot map.

Figure 7.—Continued.

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Report Documentation Page

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15. Supplementary Notes

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16. Abstract

Ag solid lubricant films, with a thin Ti interlayer for enhanced adhesion, were sputter-deposited on Al_2O_3 substrate disks to reduce friction and wear. The dual Ag/Ti films were tested at room temperature in a pin-on-disk tribometer sliding against bare, uncoated Al_2O_3 pins under a 4.9 N load at a sliding velocity of 1 m/s. The Ag/Ti films reduced the friction coefficient by 50 percent to about 0.41 compared to unlubricated baseline specimens. Pin wear was reduced by a factor of 140 and disk wear was reduced by a factor of 2.5 compared to the baseline. These films retain their good tribological properties including adhesion after heat treatments at 850 °C and thus may be able to lubricate over a wide temperature range. This lubrication technique is applicable to space lubrication, advanced heat engines, and advanced transportation systems.

17. Key Words (Suggested by Author(s)) Solid lubrication; Ceramics; High temperature; lubrication; Friction; Wear		18. Distribution Statement Unclassified–Unlimited Subject Category 23	
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