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EFFECT OF METALLIC-COATING PROPERTIES ON THE TRIBOLOGY OF OIL-LUBRICATED, COATED CERAMICS*

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EFFECT OF METALLIC COATING PROPERTIES ON THE TRIBOLOGY OF OIL-LUBRICATED COATED-CERAMICS*

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Abstract

The friction and wear behavior of zirconia ceramics lubricated with solid coatings (Ag, Au, and Nb), deposited by ion-beam-assisted-deposition (IBAD) techniques, and a polyol-ester-based synthetic oil are presented. These results demonstrate that, although the simultaneous use of soft (e.g. Ag and Au) solid lubricants in conjunction with the synthetic lubricant significantly reduces the friction and wear under boundary lubrication at temperatures up to 250°C, the durability of the soft films was poor. In contrast, durability of Nb coating (in terms of chemical reactivity and adhesion during the tribo-tests) was better than that of the Ag or Au films. However, the friction and wear behavior of the Nb-coated films was poorer than that of the ceramics coated with Ag or Au.

INTRODUCTION

Ceramics are being considered for use in high-temperature applications where their excellent strength and chemical stability are needed [1-4]. In some applications, such as low-heat-rejection engines, some ceramic components will be in sliding contact with other components and it is therefore important to consider their tribological properties. Unfortunately, the tribological properties of many ceramics are quite poor when compared to the behavior of materials commonly in use at lower temperatures [5,6]. Whereas advanced liquid lubricants are under development, it may prove necessary (particularly under boundary-lubrication regimes) to utilize solid lubricants in conjunction with liquid lubrication.

Recent results [7,8] indicate that soft metallic films deposited on ceramic substrates by ion-beam-assisted-deposition (IBAD) techniques can significantly reduce friction and wear at elevated temperatures under dry sliding conditions. Good results were also obtained during the initial stages of liquid-lubricated wear tests performed on IBAD Ag-coated ceramics; however, reactions with sulfur-bearing oil additives eventually lead to delamination of the Ag films [9].

Consequently, a series of tests, reported here, were performed to determine if coatings of a highly reactive element (Nb) or a noble element (Au) would perform comparably to Ag, yet remain intact for longer periods of time.

EXPERIMENTAL DETAILS

Friction and wear tests were done with pin-on-flat contact geometry in reciprocating sliding. The pins (8 mm diameter and 15 mm long) were rounded at one end to a 127 mm radius of curvature. Details of the test device have been described elsewhere [10]; tests were done at a normal load of 50 N, reciprocating frequency of 1 Hz, and a stroke length of 25 mm; creating an average sliding speed of $0.05 \mathrm{ms}^{-1}$ for a total of 2000 cycles. Three temperatures : 23°C (room temperature), 150°C, and 250°C were used. The 50 N normal load produce an initial mean Hertzian pressure of about 208 MPa for ZrO_2 on ZrO_2 contact. The nominal bearing pressure is known to decrease very rapidly once wear begins due to the change in contact area resulting from wear [8]. A set of tests were conducted under dry conditions and without any metal coatings at the three temperatures to serve as reference to which the effectiveness of the lubricants can be compared. For the high-temperature dry tests, both the flat and pin specimens were heated to the required temperature. Oil-lubricated tests were done using uncoated, Ag-coated, Au-coated and Nb-coated flats. A 100% polyol-ester-based synthetic oil with proprietary blend of additives

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including ZDDP was used. The lubricant was applied by immersing the flat specimen in a bath of the oil during the test, creating a fully flooded contact. The high-temperature tests were done by heating the oil bath and the pin specimens to the desired temperature.

The friction coefficient was continuously monitored in all tests by a strain-gauge device. The amount of pin wear was calculated from the dimensions of the wear scar measured by an optical microscope. The wear volume in the pin is estimated by the equation $V_p = (\pi r^4)/4R$, where r is the wear-scar radius and R is the radius of curvature [11]. The tests were stopped after 500, 1000, and 2000 cycles and the pin wear-scar dimensions were measured. From these measurements, specific wear rate in the pin can be determined as a function of the number of cycles. The "instantaneous" specific wear rate was calculated from the average rate for each range of test cycles as $\Delta V/(F_n \Delta S)$, where ΔV is the change in the wear volume in that range, F_n is the applied normal force, and ΔS is the incremental sliding distance.

The worn surfaces of the pin and the flat were examined by optical and scanning electron microscopes (SEM) equipped with an X-ray energy-dispersive spectrometer (EDS) and a wavelength-dispersive spectrometer (WDS). The pin and uncoated flat specimens were sputter-coated with a thin layer of carbon before examination to prevent charging during SEM analysis.

RESULTS AND DISCUSSION

The frictional behavior under various test conditions is shown in Figures 1a - c for tests at room temperature, 150°C and 250°C respectively. For ZrO_2 sliding pairs without either metallic coating (solid lubricant) or oil lubricant, sliding started in all cases with a relatively low friction coefficient of 0.15, but increased gradually to a higher steady value. The rate at which the steady friction was attained and the value of the friction coefficient depended on the ambient test temperature. At room temperature, the steady friction coefficient of about 0.65 was attained after about 1000 cycles of sliding. At 150°C, the steady friction was lower at a value of about 0.58, and was attained after 700 cycles of sliding. A steady friction coefficient value of about 0.55 was attained in about 100 cycles for the test done at 250°C. The initial low friction coefficient may be due to the contamination and/or adsorption of the various species from the test environment onto the surface. The "contaminant" is gradually removed by the sliding contact. The removal is accelerated by thermal desorption as the temperature is increased, and thus the friction coefficient attained the steady value faster at higher temperatures.

With oil lubrication without any metallic coating, the friction coefficient was nearly constant at about 0.16 throughout the test duration at the three test temperatures. This is due to the fact that all test conditions were under the boundary-lubrication regime as shown by the film thickness calculations [9]. The friction coefficient of 0.12 - 0.2 is typical for the boundary-lubrication regime.

When the oil lubricant was applied to the metal-coated ceramics, the frictional behavior was strongly dependent on the mechanical properties of the coating material. In all coated, oil-lubricated cases at room temperature (Figure 1a), sliding started with a friction coefficient of 0.14. However, with soft metals (Au and Ag), the friction decreased gradually during the first 500 cycles to a steady value of 0.05, while with Nb, which is harder, the friction coefficient remained unchanged at about 0.14 throughout the duration of the tests. This value was only slightly less than that of the uncoated, oil-lubricated test with a value of 0.16. Scanning electron microscope (SEM) micrographs elucidated the reasons for the differences in the metallic coating behavior. A very smooth contact area, with little or no evidence of wear, was generated on the surface of the soft coating (Figure 2). This has the effect of increasing the λ ratio, defined as the ratio of lubricant film thickness (h) to the composite surface roughness (σ) of the two contacting surfaces [12]. The result is the creation and maintenance of a fluid film between the two sliding surfaces and thus the test that started under a boundary regime could with time end up with a continuous fluid film at the interface. The decrease in the friction to a steady value is thus associated with the smoothing of the soft film. In the case of the harder Nb coating, no significant smoothing of the contact area was observed (Figure 3). Instead, wearing of the coating occurred with debris which consists primarily of Nb (according to EDX analysis) on the surface (Figure 3a). In all the tests, the Nb coating was not worn through as evident by Nb map (figure 3b). The lack of a decrease in surface roughness of the Nb coating ensures that the test was under boundary conditions at all times and an appreciable direct contact between the pin and the coating occurred during the entire duration of the test resulting in the coating's wear. The fact that the friction coefficient of Nb-coated and uncoated, oil-lubricated tests remained unchanged during the test duration results from both being under boundary conditions. At higher temperatures of 150° and 250°C, the Nb coating was observed to be more effective in reducing friction than it was at room temperature (Figures 1b and 1c). In fact at 250°C, the steady-state friction coefficient of Nb-coated and oil-lubricated tests was about 0.06, which is substantially lower than the room temperature value. With soft films, only a slight decrease in the friction was observed with increasing temperature.

Upon prolonged exposure to the oil, a failure of Ag was observed (figure 4), details of which have been described [9]. Although the detailed mechanism of the film delamination is not understood yet, it appears to involve the diffusion of S additive from oil lubricant to the coating/substrate interface thereby weakening it. Poor adhesion of Au to the ZrO_2 substrate also led to poor durability. The coating failure occurred more quickly at higher temperatures. The increase in the friction coefficient of the Ag-coated test at 250°C (Figure 1c) to the same level as that of the uncoated flat was due to the failure of the coating. Nb coatings, on the other hand, remained well-bonded to the ZrO_2 substrate during the duration of all the tests, even though coating removal by a continuous wear process was occurring. These differences in the adherence of various coatings result from differences in the reactivity of the Ag, Au and Nb coating materials. The less reactive Ag and Au metals could not benefit from the possible enhancement of the adhesion through chemical interaction at the coating/substrate interface. The more reactive Nb appeared to have benefited from such interaction, resulting in its superior adhesion and durability. Furthermore, chemical interactions between Ag and S additives in the synthetic oil were detrimental to the durability of the silver coatings.

In all oil-lubricated tests, there was no measurable amount of wear in the ZrO_2 flats. With Nb coatings, however, wear occurred in the coating layer but the coating was not worn through as previously discussed. With Ag and Au coatings, there was only plastic flow in the coating layer and no material removal. In some cases, however, these soft coatings did fail by delamination at the coating/substrate interface as mentioned above. The variation of the "instantaneous" specific wear rates in the mating ZrO_2 pins is shown in Figure 5. At room temperature (Figure 5a), the pin specific wear rate under dry conditions sliding against an uncoated flat averaged about 10^{-5} $mm^3N^{-1}m^{-1}$ and increased continuously with the number of sliding cycles (from 2×10^{-6} to 3×10^{-5} $mm^3N^{-1}m^{-1}$). With oil lubrication, without metallic coating, the pin specific wear rate was reduced by about three orders of magnitude to about 10^{-8} $mm^3N^{-1}m^{-1}$ and showed a slight decrease with the number of cycles. For oil-lubricated, metal-coated flats, the pin wear behavior was strongly influenced by the mechanical properties of the coating material just as was the friction. With the relatively soft coatings (Ag and Au), there was no measurable wear in the pin for the entire duration of the tests. For Nb coatings, there was a significant amount of wear in the pin ($\approx 10^{-7}$ $mm^3N^{-1}m^{-1}$). The difference resulted from the fact that the soft metal coatings were deformed, thereby increasing the λ ratio and preventing direct contact between the pin and the flat while the λ ratio remained unchanged for Nb and thus there was continuous interaction between the flat and the pin surfaces leading to wear, as previously discussed.

At higher temperatures of 150° and 250°C (Figures 5b and 5c), the pin wear rate reflected the changes in the oil lubricant and the metallic coating properties. At 150° and 250°C, there was a substantial increase in the wear rate of the pin sliding with the uncoated flat when compared with the room-temperature value. This is due primarily to the decrease in the viscosity of the oil, which led to more intimate contact between the sliding surfaces. With Nb coatings, a decrease in the hardness with increasing temperature resulted in more plastic deformation of the coating and less wear in the mating ZrO_2 pins. This effect was more pronounced at 250°C. With the softer coatings, the accelerated failure of the film at higher temperatures, particularly with Ag coatings, resulted in some measurable wear. This was more pronounced at 250°C when the " instantaneous" wear rate at the end of the test was comparable to that of the uncoated flat. This was due to the early failure of the film. Recall that a similar behavior was observed for the friction coefficient.

The results presented above indicate that although Ag and Au films had low wear rates and friction, their use was limited by chemical interaction with oil additives that eventually led to coating delamination. Conversely, Nb coatings remained bonded to the substrate but suffered from higher friction and wear rates. A plausible way by which the problems encountered with different metallic coatings (i.e. poor adhesion of soft Ag and Au films and lack of adequate deformation of the reactive Nb film) could be solved is by combining the benefits of each kind through multilayer coating. Conceivably, Nb could be deposited first onto ZrO_2 and then Ag or Au on top of that. Such a multilayer coating would possibly have the good adhesion of Nb to ZrO_2 and the plasticity of the soft top layer. Study of this multilayer-coating approach is underway.

CONCLUSIONS

Results of the present study show that a simultaneous application of solid (in the form of thin metallic film) and liquid (synthetic oil) lubricants provides effective lubrication for ZrO_2 under boundary-contact conditions. Both the friction coefficients and wear rates were substantially reduced by this method. Soft metals (Ag and Au) were found to be particularly effective because they can easily deform and thus modify the lubrication regime. However, their poor reactivity did not help their adhesion to the ZrO_2 substrate materials. On the other hand, relatively hard Nb coatings were not as effective in reducing friction and wear as the soft film particularly at room temperature. However, higher chemical reactivity of Nb resulted in its being well-bonded to the substrate in all cases while the less reactive Ag and Au coatings are prone to coating/substrate interface delamination. At higher temperatures due to a decrease in hardness, the coatings performed better, although the durability of the soft coatings was reduced further due to poor adhesion.

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CAPTION FOR FIGURES

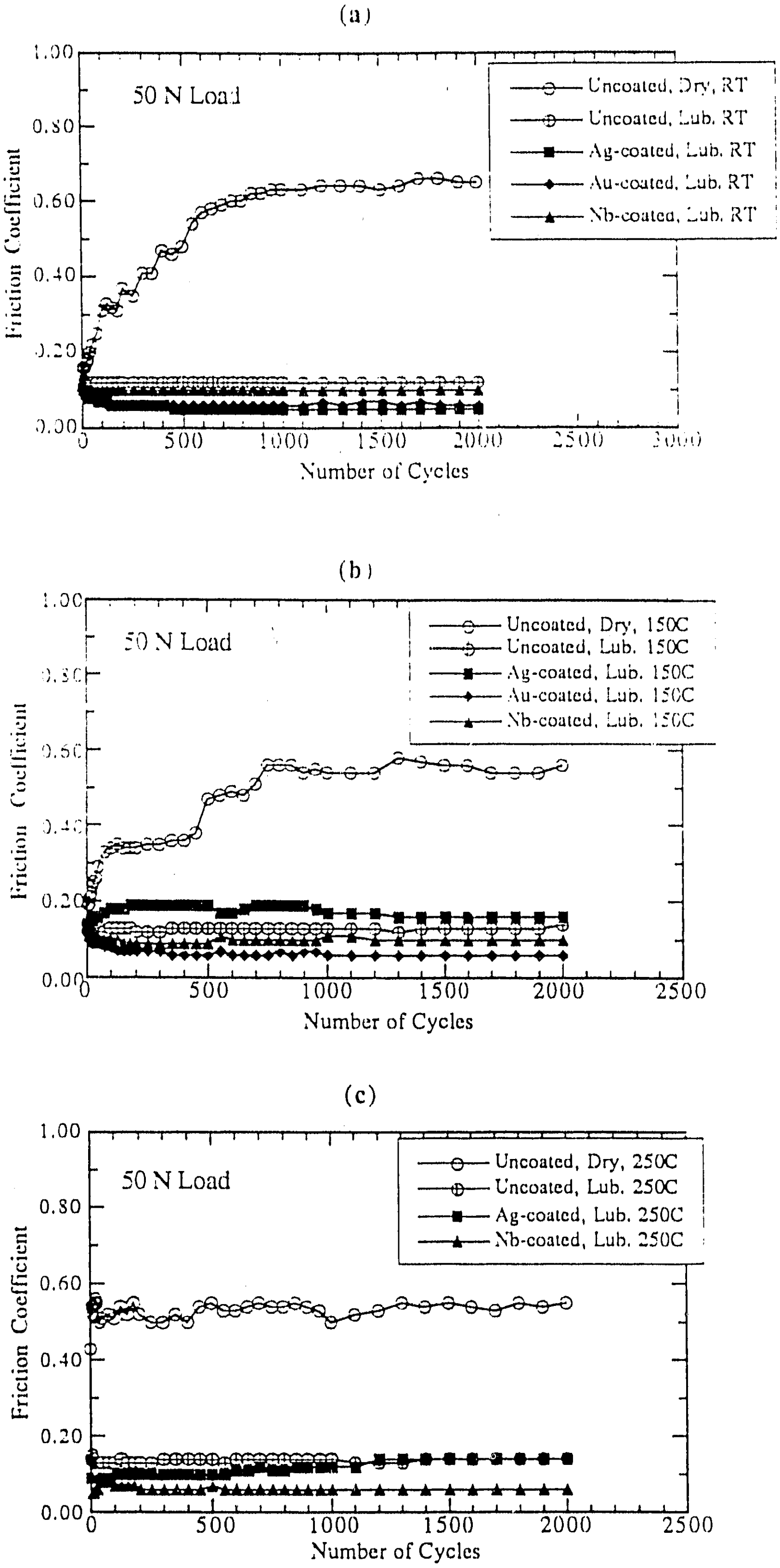
Figure 1: Variation of Friction Coefficient with the number of cycles for ZrO_2 sliding on ZrO_2 under various test conditions at (a) room temperature (b) 150°C and (c) 250°C.

Figure 2: SEM micrograph of wear track on Ag-coated ZrO_2 flat after oil-lubricated test showing the smoothing of the contact area.

Figure 3: SEM micrograph of wear track in oil-lubricated Nb-coated ZrO_2 flats showing (a) wear of Nb coatings (b) EDS map of Nb indicating the film has not been worn through.

Figure 4: SEM micrograph of failed Ag coating after prolonged exposure to the oil lubricant.

Figure 5: Variation of "instantaneous" specific wear rate of ZrO_2 pins with the number of cycles after tests under the specified conditions at (a) room temperature (b) 150°C and (c) 250°C.



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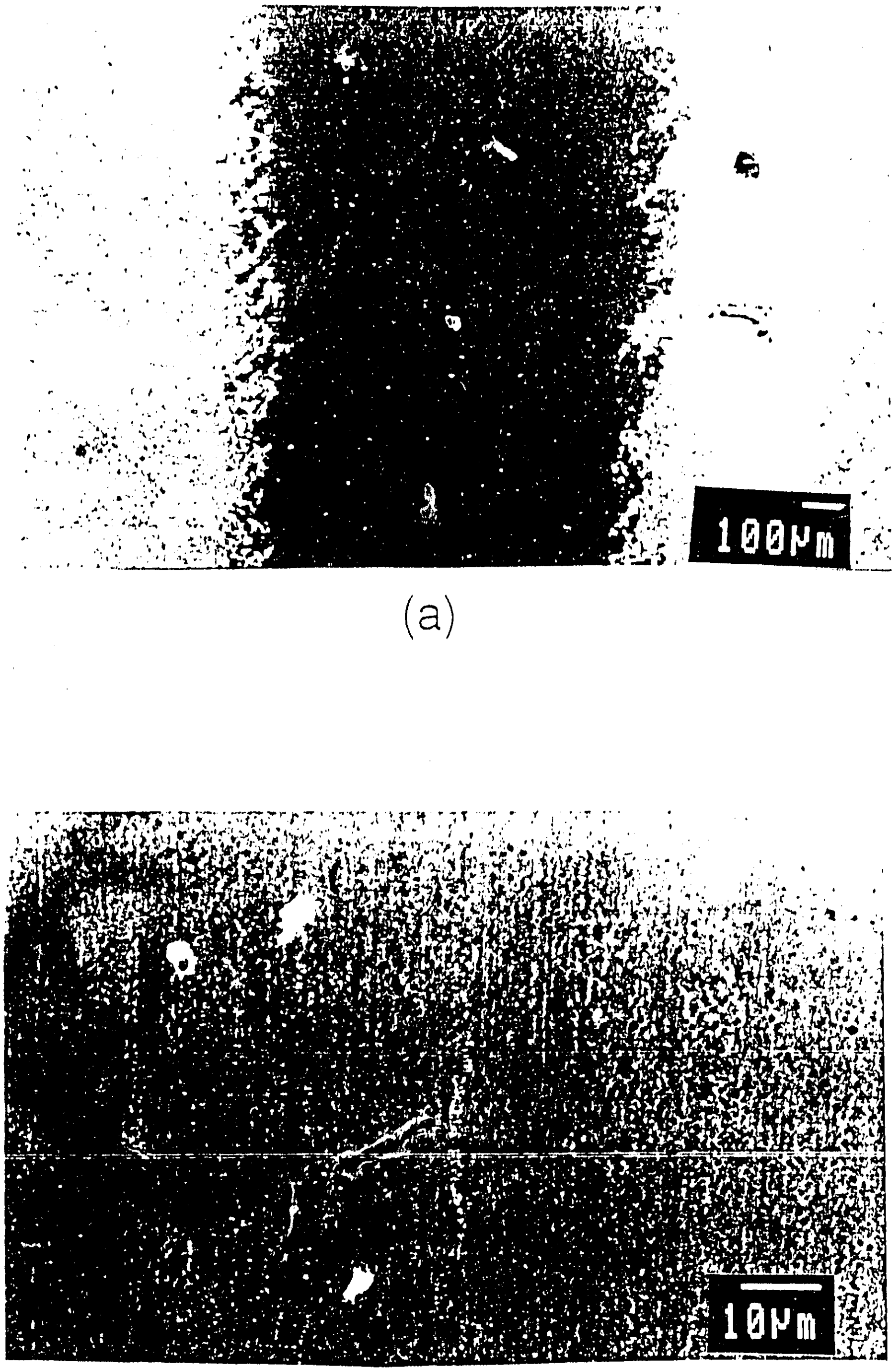
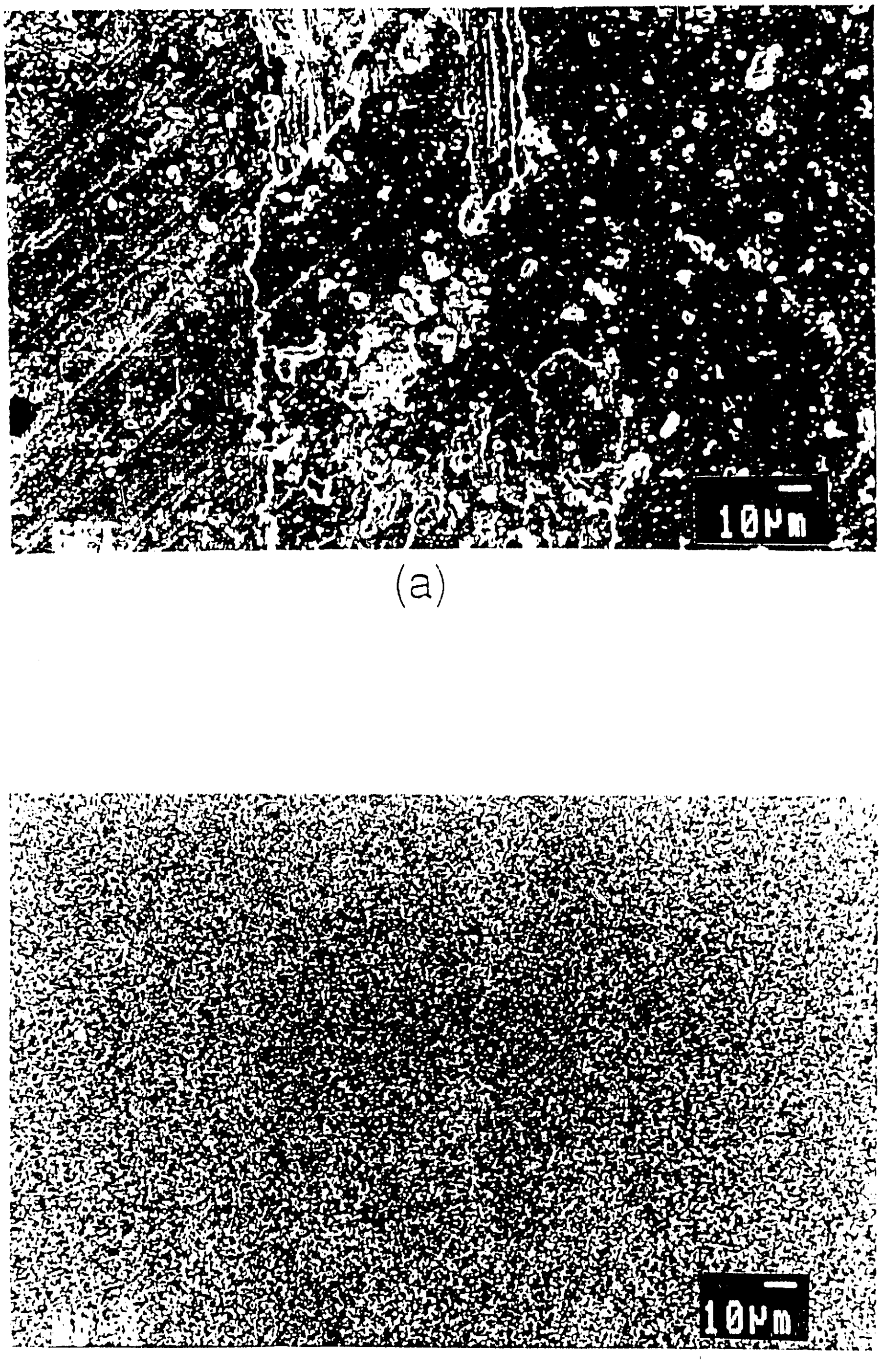


(a)

(b)

Figure 2: SEM micrograph of wear track on Ag-coated ZrO_2 flat after oil-lubricated test showing the smoothening of the contact area.



(a)

(b)

Figure 3: SEM micrograph of wear track in oil-lubricated Nb-coated ZrO_2 flats showing (a) wear of Nb coatings (b) EDS map of Nb indicating the coating has not been worn through.

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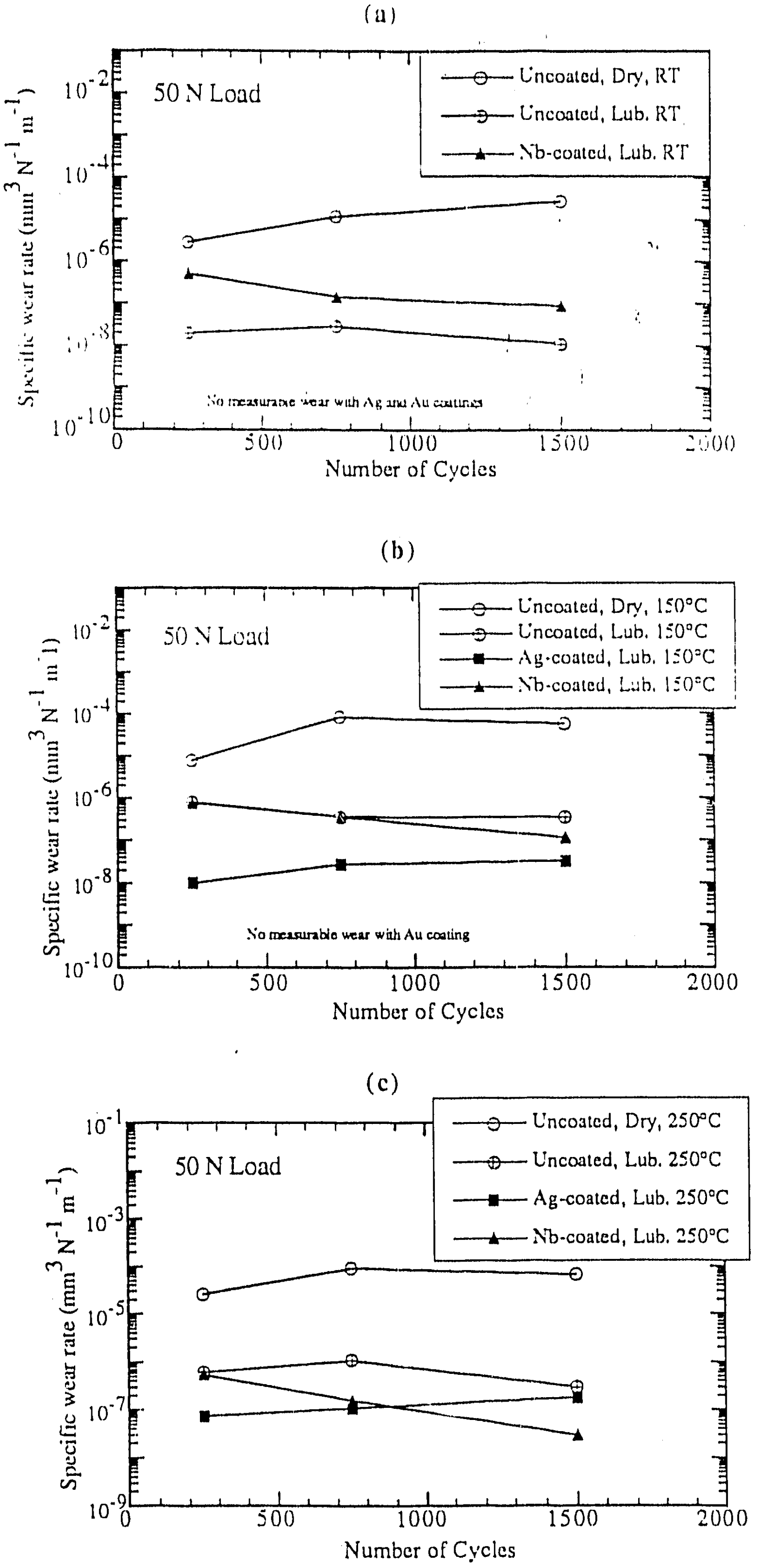

Figure 5: Variation of "Instantaneous" specific wear rate of ZrO_2 pins with the number of cycles after tests under the specified conditions at (a) room temperature (b) 150°C and (c) 250°C.

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