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TRIBOLOGICAL PERFORMANCE OF DIAMOND AND DIAMONDLIKE CARBON FILMS AT ELEVATED TEMPERATURES*

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ABSTRACT

In this study, we investigated the tribological performance of diamond and diamondlike carbon (DLC) films as a function of ambient temperature. Both films were deposited on silicon carbide (SiC) by microwave plasma chemical vapor deposition and ion-beam deposition processes. Tribological tests were performed on a reciprocating wear machine in open air (20 to 30% relative humidity) and under a 10-N load using SiC pins. For the test conditions explored, the steady-state friction coefficients of test pairs without a diamond or DLC film were 0.7 to 0.9 and the average wear rates of pins were 10^{-5} to 10^{-7} mm³/N.m, depending on ambient temperature. DLC films reduced the steady-state friction coefficients of test pairs by factors of 3 to 5 and the wear rates of pins by two to three orders of magnitude. Low friction coefficients were also obtained with the diamond films, but wear rates of the counterface pins were high due to the very abrasive nature of these films. The wear of SiC disks coated with either diamond or DLC films was virtually unmeasurable while the wear of uncoated disks was substantial. Test results showed that the DLC films could afford low friction up to about 300°C. At higher temperatures, the DLC films became graphitized and were removed from the surface. The diamond films could withstand much higher temperatures, but their tribological behavior degraded. Raman spectroscopy and scanning electron microscopy were used to elucidate the friction and wear mechanisms of both films at high temperatures.

Key Words: Diamond and diamondlike carbon films, solid lubrication, high-temperature friction and wear.

INTRODUCTION

Extreme hardness, high chemical stability, and low friction character of diamond and diamond-like carbon (DLC) films make them good candidates for a wide range of tribological applications, including bearings, gears, seals, and metal-cutting and forming tools [1-3]. Unlike the very popular nitride and carbide coatings (e.g., TiN and TiC) which function more effectively under oil-lubricated sliding-contact conditions [4,5], smooth diamond and DLC coatings provide low friction and high wear resistance to most sliding surfaces even without lubrication [6-16]. Furthermore, these coatings are significantly harder and much stiffer than most carbides and nitrides.

Previous research has demonstrated that the friction and wear performance of both diamond and DLC coatings can be very sensitive to surface finish [17-19], environment [11,14,15], and ambient temperature [7,14,15,20,21]. According to Hayward and coworkers [17-19], the surface roughness of diamond coatings could have a substantial impact on friction and wear. In general, rougher diamond films give rise to higher friction and wear. The testing environment was also found to influence the tribological performance of these coatings [11,12,14,15]. For example, the friction coefficients of diamond and DLC films against various ceramic balls were reported to be about 0.04 and 0.02, respectively, in dry N₂, whereas in humid air these friction values increased by factors of 3 to 5 [12]. Tests by Dugger et al. [22] showed that the friction coefficients of diamond films could be very high in ultrahigh vacuum. They attributed high friction to the removal of surface adsorbates during sliding. Bleeding air into the test chamber reduced friction by more than one order of magnitude.

At elevated temperatures, the friction and wear performance of diamond and DLC films deteriorates substantially. At temperatures >300°C, the DLC films became more graphitelike [7,14,20] and delaminated from the sliding surfaces, thus losing their effectiveness. As for the diamond films, Kohzaki et al. [21] reported friction coefficients of 0.8 at 600°C for SiC balls sliding against the diamond films. In a series of recent papers, Gardos et al. [15,23,24] showed that the friction coefficients of polycrystalline diamond against itself are fairly low in mild vacuum

and at temperatures up to 500°C. At higher temperatures, Gardos et al. noted unexpected increases in friction and attributed such phenomena to the desorption of surface adsorbates that are known to passivate the dangling bonds of diamond surfaces.

In this paper, we present the tribological performance of diamond and DLC films on a comparative manner at temperatures up to 400°C. The DLC coatings performed reasonably well up to 300°C, but became graphitized at much higher temperatures. As for the diamond coatings, they could endure higher temperatures, but their tribological performance was not very good.

EXPERIMENTAL DETAILS

Test Materials

The pins and flats used in this study were sintered SiC, obtained from a commercial source [25]. The flat samples were in rectangular shape (38.1 x 50.8 x 4.2 mm) and had a surface roughness of about 0.03 μm center-line-average (CLA). SiC pins were 15 mm long and 8-mm in diameter. One end of each pin was rounded to a radius of curvature of 127 mm, and pin surface finish was $\approx 0.03 \mu\text{m}$ CLA. All specimens were ultrasonically cleaned sequentially in trichlorethylene, acetone, and methanol for 10 minutes each, then dried in an oven at 110°C for 20 min.

Deposition of Diamond and DLC Coatings

Thin DLC films ($\approx 1.5 \mu\text{m}$ thick) were ion-beam deposited on SiC at room temperature in a vacuum chamber equipped with a broad-beam ion source. A short description of the procedures used during the preparation of DLC coatings is given below, while more detailed description can be found in Refs. 7 and 10.

First, the SiC substrates were positioned directly below the ion source and sputter-cleaned with 1 keV, 2.5 mA/cm² Ar-ion beam for ≈ 3 -5 min. Argon gas used for sputter-cleaning was fed through

the cylindrical ion source of the broad-beam system and ionized by energetic electrons that were produced by a hot filament wire. At the conclusion of the sputter-cleaning step, the DLC deposition was begun by switching from argon to methane, the carbon source. The methane was fed through the same cylindrical ion source and was ionized by energetic electrons emitted by the hot filament wire. Operating conditions were adjusted to give an ion beam with an acceleration energy of 450 eV and current density of $\approx 2.5 \text{ mA/cm}^2$. Under these operating conditions, the deposition rate was $\approx 3 \text{ }\mu\text{m/hr}$. Deposition was continued until a 1.5- μm -thick DLC film was obtained. Because of the methane as carbon source, the DLC films produced here contained about 23 at.% hydrogen and had a Vickers hardness value of about 17 GPa.

The diamond films used in these study were prepared in a microwave-plasma-assisted chemical-vapor-deposition reactor. Prior to deposition, the optically polished SiC substrates were given a final dry vibratory polishing treatment with 0.1 μm diamond particles to enhance film nucleation and promote growth of a uniform, continuous diamond film. The films were deposited at substrate temperatures of approximately 900 to 950 °C. During the initial stages of deposition, the surfaces were cleaned in pure H_2 plasma at a flow rate of 100 sccm for approximately 15 min at 40 torr and a forward microwave power of 1250 W. After 15 min of surface cleaning, deposition was initiated by increasing the flow of methane to 2 sccm for the remainder of the run. The SiC samples were coated for 30 and 60 hours resulting in film thicknesses of ≈ 15 and $\approx 30 \text{ }\mu\text{m}$, respectively.

Tribological Tests

The tribological tests were conducted on a reciprocating wear test rig in which the pin specimen was firmly secured on a holder and drawn back and forth across the stationary flat. Frequency and stroke length were 1 Hz and 25 mm, respectively, giving an average sliding velocity of 0.05 m/s. Each test was run for 4000 cycles or 8000 passes, which amounts to sliding distance of $\approx 200 \text{ m}$. The dead weight on top of the pin specimen sliding against the flat was 10 N, which created a mean Hertzian pressure of $\approx 125 \text{ MPa}$. Room humidity ranged between 20 to 30% during the tests

at room temperature (e.g., 23°C). Further details of this test rig are given in Ref. 4.

For tests at high temperatures, electrical cartridge heaters were inserted into the upper and lower stainless steel plates that housed the pin and flat specimens. Friction force, which was monitored by strain-gauge transducer, was recorded continuously throughout the tests. The wear volume measurement of each pin specimen was based on the radius (R) of the hemispherical end of pins, and the microscopic determination of the wear scar radius (r), which were subsequently converted into wear volume (V) by the mathematical expression $\{V=(\pi r^4)/(4R)\}$ suggested in Ref. [26]. The wear of flat specimens was assessed from the three-dimensional traces of surface profiles across the wear tracks. For better accuracy and reproducibility, duplicate tests were run at each temperature, mean values of friction coefficients and wear rates are reported. The wear tracks and the scars, as well as the wear-debris particles, were examined by scanning electron microscopy (SEM) and Raman spectroscopy operating with a HeNe laser having a wavelength of 632.8 nm with an output power of 25 mW over a spot size of 2 μm .

RESULTS

Film Microstructure and Chemistry

The cross-sectional SEM images of the DLC and diamond films are shown in Fig. 1. As is clear from Fig. 1a, the structure of DLC film appears very dense and featureless, whereas the diamond film (Fig. 1b) is highly faceted and composed of coarse grains. Figure 2 reveals that the surface topography of this diamond film is very rough. Figure 3 shows the laser Raman spectra of both films. The spectrum of diamond film shows a sharp Raman band at 1332 cm^{-1} , which is characteristic of these films. It also features a broad peak at around 1520 cm^{-1} which may be due to non-diamond precursors [27,28].

The Raman spectrum of DLC is very different from that of diamond. It shows a very broad peak centered at 1520 cm^{-1} and a shouldered peak at around 1345 cm^{-1} . In general, both peaks are

typical of most carbon materials classified as DLC [28-30].

Friction and Wear

Figure 4 shows the friction coefficients of SiC pins during sliding against itself and against diamond and DLC coatings at different temperatures. As is evident, the friction coefficients of SiC against itself are quite high and vary significantly with temperature. Up to 200°C, their friction coefficients range from 0.8 to 0.9, although a slight decrease occurs during tests at 300 and 400°C. The friction coefficients of SiC pins against diamond film is about 0.4 at room temperature, but decreases to 0.3 - 0.25 at 300°C. At 400°C, the friction coefficient increases to 0.5. The friction coefficients of SiC pins against DLC-coated SiC flats fluctuate between 0.2 and 0.25 during tests at temperatures from 23° to 400°C. They appear to be relatively insensitive to increasing temperature. The asterisks along the friction coefficient at 400°C means that the 0.2 friction coefficient could be maintained for the first 1500 sliding cycles. After that, friction increased to 0.6, and microscopic examination of the sliding surface after the test revealed that the film was largely removed from the surface, as shown in Fig. 5.

Figure 6 shows the average specific wear rates of SiC pins sliding against itself and diamond and DLC-coated SiC flats at different temperatures. The wear rate of SiC pins against SiC is about $3.6 \times 10^{-7} \text{ mm}^3/\text{N.m}$ at room temperature, but increases markedly at higher temperatures. At room temperature, the wear rates of SiC pins sliding against the diamond-coated SiC flats were 6 times higher than those of the pins slid against the uncoated SiC flats. At elevated temperatures, pin wear rates increased substantially and in fact became comparable to those of the pins slid against the uncoated SiC flats. However, wear rates of SiC pins sliding against the DLC-coated SiC flats were very low, ranging from $1.2 \times 10^{-8} \text{ mm}^3/\text{N.m}$ at 200°C to about $2.2 \times 10^{-8} \text{ mm}^3/\text{N.m}$ at room temperature. These values are two to three orders of magnitude lower than the wear rates of balls slid against the uncoated disks. Photomicrographs in Fig. 7 compare the actual sizes of wear scars formed on SiC pins during tests at room temperature. As is clear, the largest wear scar was seen on a pin slid against the diamond-coated SiC flat (Fig. 7a), whereas the smallest scar was found

on a pin slid against the DLC-coated SiC flat (Fig. 7b). The wear scar formed on the pin slid against the bare SiC flat was much smaller than that formed against the diamond film, but significantly larger than that formed against DLC film (Fig. 7c).

SEM microscopy of the worn surfaces of SiC pins and uncoated SiC flats revealed that grain pullout and microfracture were the dominant wear modes for these samples (see Fig. 8). In the case of SiC pins slid against the diamond films, the wear was mostly dominated by microcutting (see Fig. 7a). The wear of pins sliding against the DLC-coated flats was in the form of fine polishing.

Three-dimensional surface maps in Fig. 9 compare the extent of wear damage occurred on bare, diamond, and DLC-coated SiC flats. As is clear, the extent of wear on the uncoated flat is rather significant. Wear of the DLC-coated SiC flats was hard to discern. Using a surface profilometer, we could not reliably detect measurable amounts of wear losses on the diamond and DLC-coated flats. Further SEM examinations revealed that wear was confined essentially to the sharp asperity tips of diamond film. Also loose debris particles filled in the valleys between sharp crystal facets of diamond films and resulted in a smooth wear track (see Fig. 10). The DLC films were still intact after sliding tests at temperatures to 300°C. However, as mentioned above, during tests at 400°C, the DLC film delaminated and the wear of SiC pin (see Fig. 6) became significant.

DISCUSSION: FRICTION

SiC/SiC Test Pairs

The high friction coefficients of the SiC/SiC test pairs (0.7-0.9) are typical for this ceramic under dry sliding conditions and comparable to values reported elsewhere [31]. The slight decrease in friction with increasing temperature may be due to the formation of a tribochemical film that provides marginal lubrication. Also, as reported by Buckley and Miyoshi limited amounts of

carbon are released from the SiC material during dynamic sliding at elevated temperatures, and this carbon may provide marginal lubrication [32]. Overall, the range of friction coefficients for the SiC/SiC pairs reported here is quite consistent with the previous data reported in Refs. 31 and 32.

SiC/DLC-SiC Test Pairs

The generally low friction coefficients of pairs with a DLC film are due largely to the very inert nature and easy shear character of DLC surfaces. Compared to the very low friction coefficients (0.005-0.05) in vacuum, dry N₂ or Ar [11,13,14], the coefficients reported in Fig. 4 are significantly higher. This is largely because of the fairly high humidity (e.g., 20-30%) during our friction tests. Overall, these friction coefficients are in keeping with those reported for DLC films in humid test environments [6,7,9,11,14]. One explanation given for the relatively higher friction coefficients in humid environments was that the real contact areas of sliding surfaces may become saturated with water molecules, and this exerts additional bonds across the contact interfaces, thus causing higher friction [14].

Figure 4 shows that the friction coefficients of SiC/DLC-coated SiC pairs did not significantly change during tests at temperatures up to 300°C. However, at 400°C, low friction could be maintained for the first 1500 cycles, after which the friction coefficient increased sharply to ≈0.6. This was due to the removal of DLC from the wear track (see Fig. 5). Laser Raman spectroscopy of this wear track (see Fig. 11) revealed that the remnants of the DLC film had essentially transformed to graphite. The peak positions of the graphitized DLC overlap with the D and G bands of the crystalline graphite. Therefore, the short lifetime of DLC at 400°C must have been due to graphitization. Graphite is soft and easy to remove from sliding surfaces. The Raman peaks at 794 and 970 are due to exposed SiC material. As is clear from Fig. 11, the Raman spectrum of DLC tested at 300°C is similar to that of virgin DLC, suggesting that this film is still stable at this temperature. Other investigators have also found that, at elevated temperatures, the DLC films lost their diamondlike character and become more graphitelike [7,14,20].

SiC/Diamond-coated SiC Pairs

As shown in Fig. 4, the friction coefficient of SiC pins against diamond films were significantly higher. We believe that these high friction coefficients are due primarily to the generally rough surface finish of these coatings (see Fig. 2). During successive sliding cycles, the gaps between asperities were partially filled with debris particles (see Fig. 10) and the summits of asperities were worn away, especially at elevated temperatures (see Fig. 12). As a result, a relatively smooth surface finish was eventually obtained. Therefore, the range of steady-state friction coefficients (0.25-0.5) shown in Fig. 4, is consistent with the increasingly smoother surface finish. Previous investigators have reported close correlation between surface roughness and friction coefficients of diamond films. In general, it was found that the higher the surface roughness the greater the friction coefficients. The friction coefficients reported in Fig. 4 are consistent with the results reported by these investigators [18,21-24,27].

As is clear from Fig. 11, the diamond films are not noticeably graphitized like the DLC films even after tests at 400°C. The Raman band at 1332 cm^{-1} (characteristic of diamond) is still very sharp. However, the shouldered peak at around 1580 cm^{-1} becomes more prominent, perhaps suggesting minor graphitization in this film as well.

DISCUSSION-WEAR

SiC/SiC Test Pairs

An overview of the wear mechanisms of SiC ceramics suggests that microfracture [31] and tribochemical [32] interactions have significant effects on the wear behavior of SiC. Electron microscopy of the wear scars and tracks of SiC pins and flats revealed large-scale brittle fracture, as well as some grain pullouts (Fig. 8). Therefore, we believe that intra- and/or inter-granular fracture was the dominant wear mechanism for SiC test pairs.

Wear due to tribochemical interactions, especially at higher temperatures, is very likely, but the physical evidence for this type of wear could not be verified in this study, presumably, the tribochemical films consisting of C and/or O were too thin to be detected with the Raman spectroscopy used in our study. The results of previous studies suggested the formation of such tribochemical films at elevated temperatures and showed correlation of the lower wear and friction coefficients to the presence of such films at sliding interfaces [32]. The friction coefficients in our case were high at all temperatures further suggesting that there was not significant film formation at the sliding interfaces of SiC/SiC test pairs.

SiC/DLC-SiC Test Pairs

The results from these pairs demonstrate that the DLC films can impart very low wear (1.2×10^{-8} mm³/N.m to 2.2×10^{-8} mm³/N.m) to sliding SiC surfaces. We believe that the high mechanical hardness (i.e., 17 GPa) and low-friction coefficients (i.e., 0.2) of these films were largely responsible for the low wear rates of pins and flats at temperatures up to 300°C. We attribute the significantly higher wear rates at 400°C to the removal of DLC films from the substrate surfaces (see Fig. 5). The Raman spectrum of film tested at 400°C revealed that the remaining film turned into graphite, suggesting that the practical uses of DLC films should be confined to temperatures of <300°C.

SiC/Diamond-Coated SiC

Electron microscopy examination of the wear tracks of diamond-coated flats indicates that these films remained intact on the sliding surfaces (see Figs. 9, 10) even at 400°C, although they underwent some polishing and/or smoothing during repeated sliding (see Fig. 12). Moreover, Fig. 10 reveals that some debris particles had transferred from the pin side and filled in the valleys between diamond asperities. Based on these observations, we can conclude that diamond films effectively protected underlying substrates against wear even at the highest test temperature of

400°C. However, because of their extreme hardness and rough surface finish, the diamond films caused substantial wear on the relatively softer counterface pins. This observation is consistent with the results of Haywood and of Kozakhi et al. who also reported high wear rates for rough diamond films at room and elevated temperatures.

CONCLUSIONS

Based on the results reported above, we can conclude the following:

- (1) Under the test conditions of this study, sliding of SiC on itself causes high friction and severe wear losses. Increasing the test temperature is detrimental to the wear behavior of SiC ceramics.
- (2) Based on electron microscopy evidence, microfracture appeared to be the dominant cause of wear for sliding SiC pins and flats.
- (3) Because of its smooth surface finish and low-friction character, DLC films reduced the wear rates of SiC pins by two to three orders of magnitude, depending on test temperature. At temperatures higher than $\approx 300^\circ\text{C}$, they were graphitized and removed effectively from the sliding surfaces.
- (4) Diamond films were able to endure much higher temperatures. However, because of a rough surface finish, friction and wear losses were substantial.
- (5) In selecting diamond or DLC films for tribological applications, it is important to remember that the limited-temperature capability of DLC and rough surface finish of diamond films can pose serious problems.

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FIGURE CAPTIONS:

Figure 1. Cross-sectional SEM photomicrographs of (a) DLC (b) diamond films used in this study.

Figure 2. Plan-view SEM photomicrograph of diamond film.

Figure 3. Laser Raman spectra of DLC and diamond films.

Figure 4. Friction coefficients of SiC pins against uncoated, DLC, and diamond-coated SiC flats at temperatures up to 400°C (test conditions: load, 10 N; speed, 0.05 m/s; sliding distance, 200 m; relative humidity, 30%).

Figure 5. SEM photomicrograph of wear track formed on DLC-coated SiC flat during test at 400°C. (test conditions: load, 10 N; speed, 0.05 m/s, relative humidity, 30%; sliding distance, 200 m).

Figure 6. Average wear rates of SiC pins during sliding against uncoated, DLC, and diamond-coated SiC flats at temperatures up to 400°C (test conditions: load, 10 N; speed, 0.05 m/s; sliding distance, 200 m; relative humidity, 30%).

Fig. 7. SEM photomicrographs of wear scars formed on SiC pins during sliding against (a) uncoated, (b) DLC-coated and (c) diamond-coated SiC flats (test conditions: load, 5 N; speed, 0.05 m/s, sliding distance, 200 m; relative humidity, 30%; temperature, 23°C).

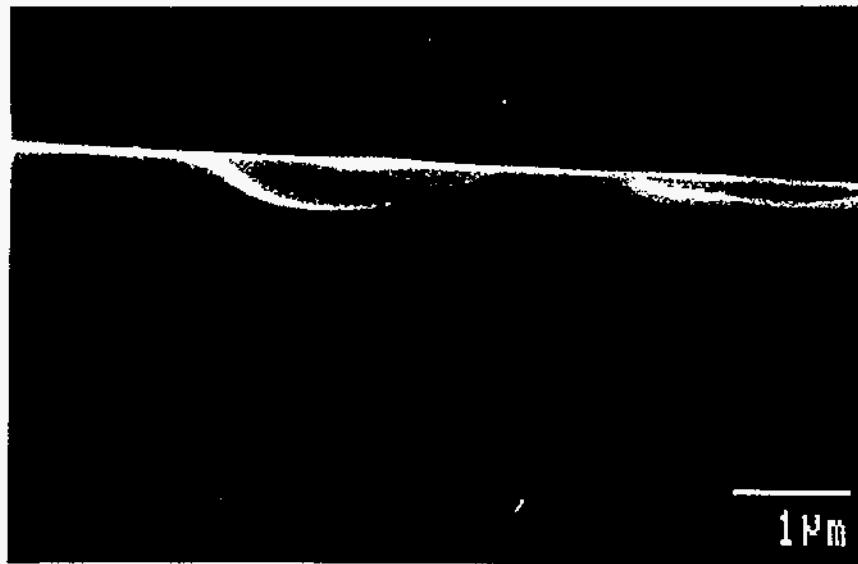
Fig. 8. SEM photomicrograph of worn surfaces of SiC flat formed during tests at 100°C.

Figure 9. 3-D surface maps of wear tracks formed on (a) uncoated, (b) DLC-coated, and (c) diamond-coated SiC flat (test conditions: load, 10 N; sliding velocity, 0.05 m/s; relative humidity, 20%; sliding distance, 200 m; temperature, 200°C).

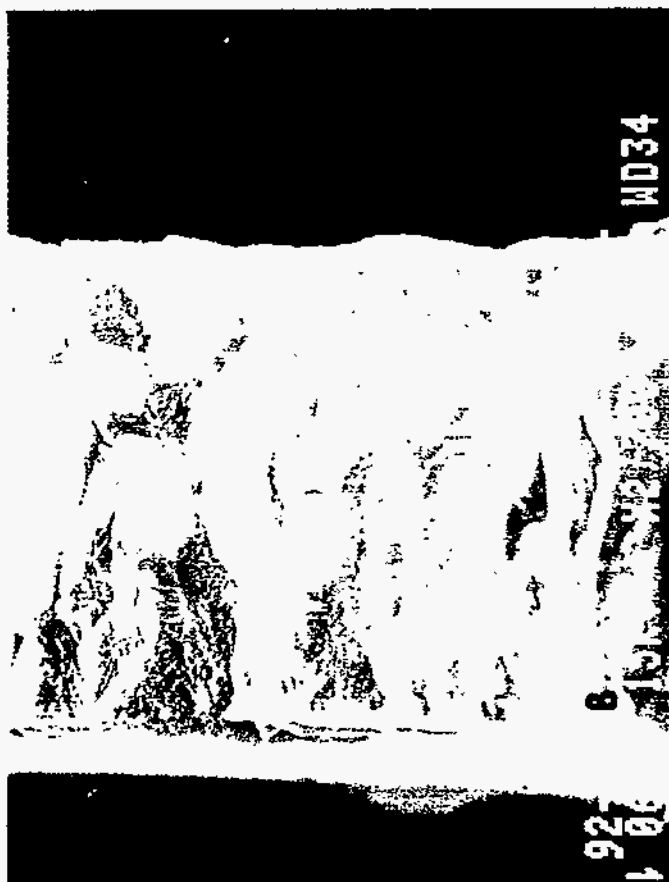
Fig. 10. SEM photomicrograph of worn surface of diamond-coated SiC flat formed during tests at 200°C.

Fig. 11. Raman spectra of graphite and diamond and DLC films before and after tests at various temperatures.

Fig. 12. Fig. 10. SEM photomicrograph of worn surface of diamond-coated SiC flat after test at 400°C.



(a)



(b)

Figure 1. Cross-sectional SEM photomicrographs of a. DLC b) diamond films used in this study.

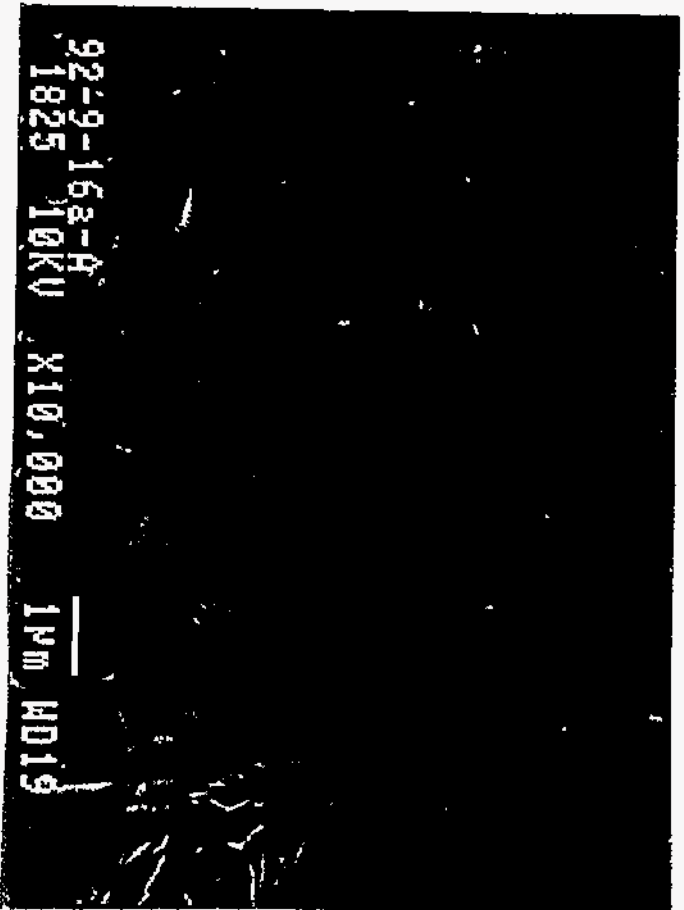


Figure 2. Plan-view SEM photomicrograph of diamond film.

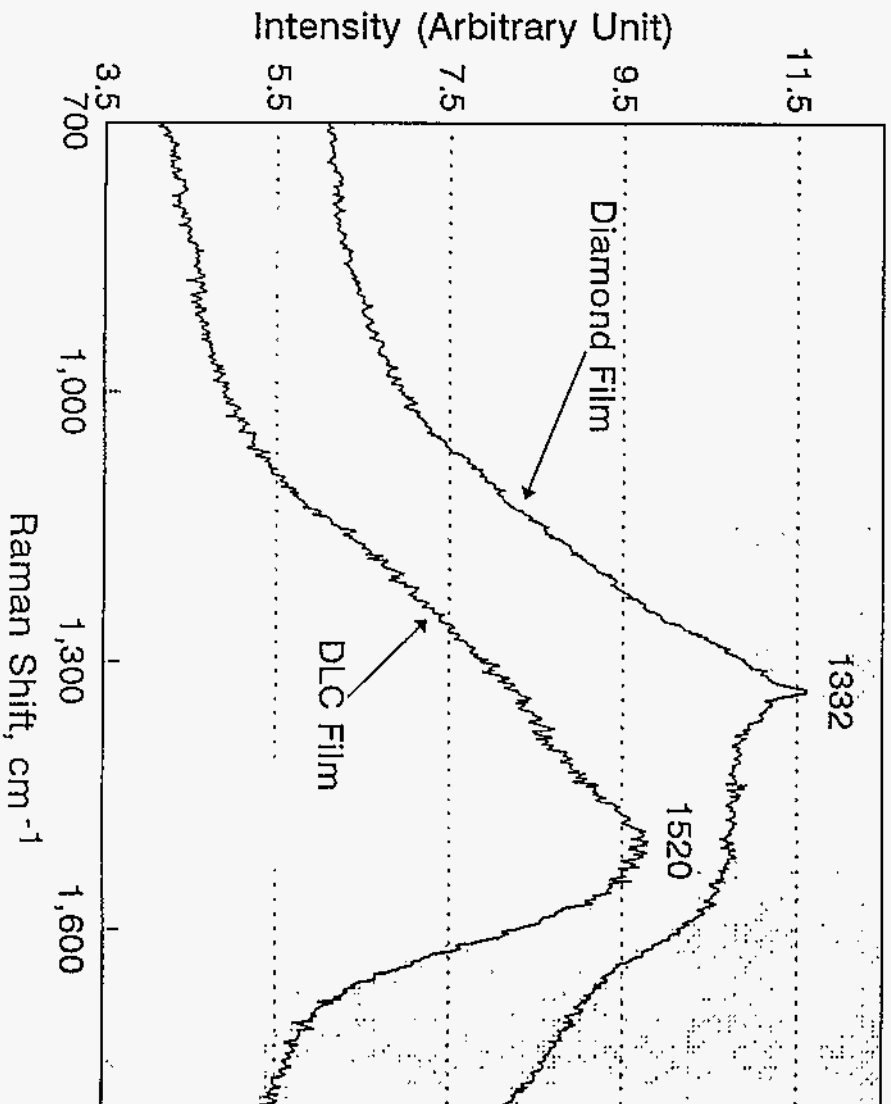


Figure 3. Laser Raman spectra of DLC and diamond films.

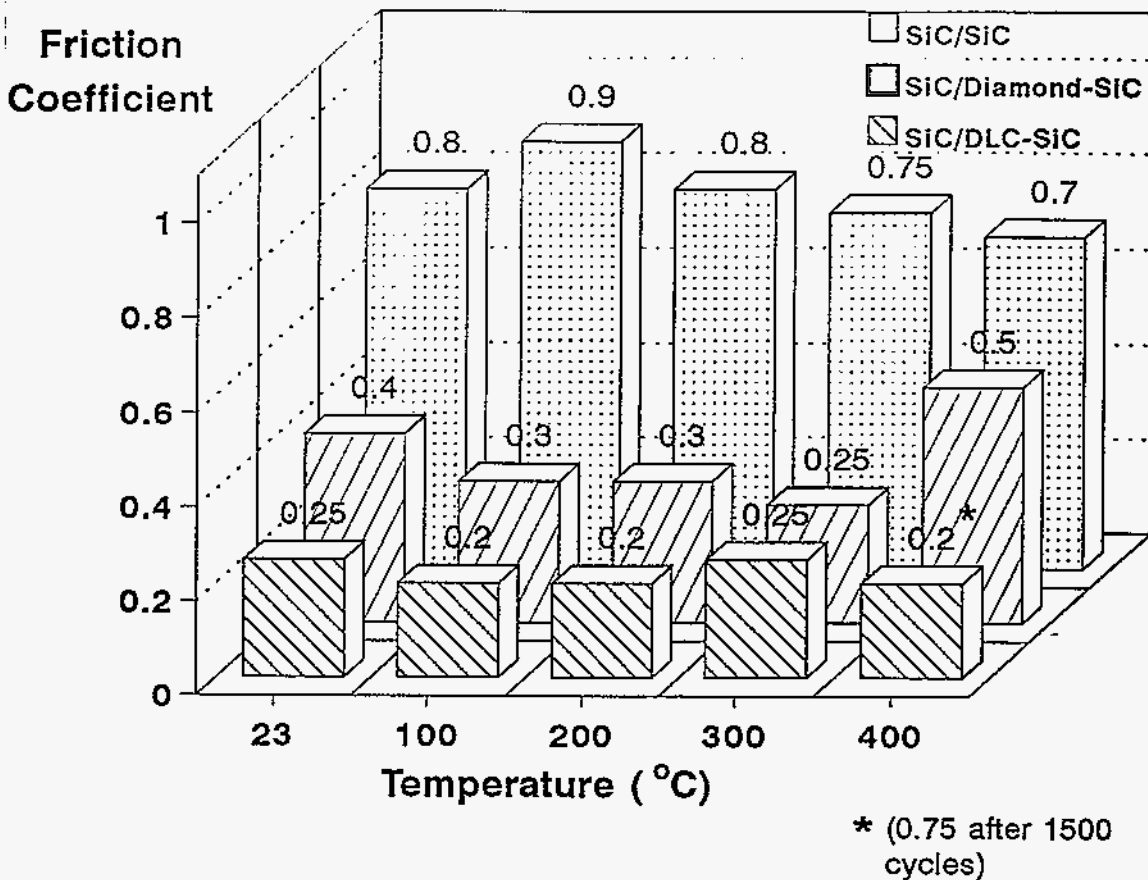


Figure 4. Friction coefficients of SiC pins against uncoated, DLC, and diamond-coated SiC flats at temperatures up to 400°C (test conditions: load, 10 N; speed, 0.05 m/s; sliding distance, 200 m; relative humidity, 30%).

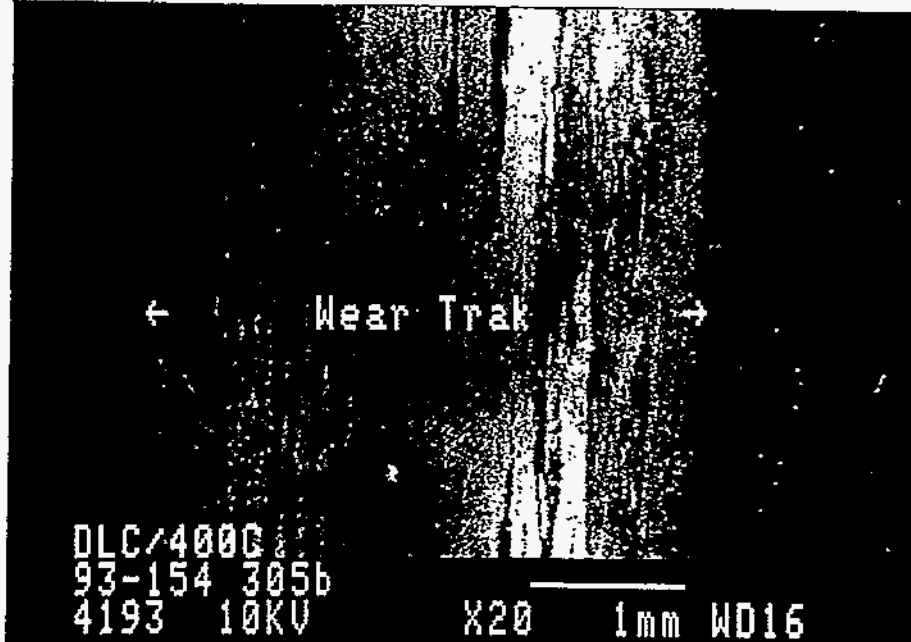


Figure 5. SEM photomicrograph of wear track formed on DLC-coated SiC during test at 400°C. (test conditions: load, 10 N; speed, 0.05 m/s; relative humidity, 30%; sliding distance, 200 m).

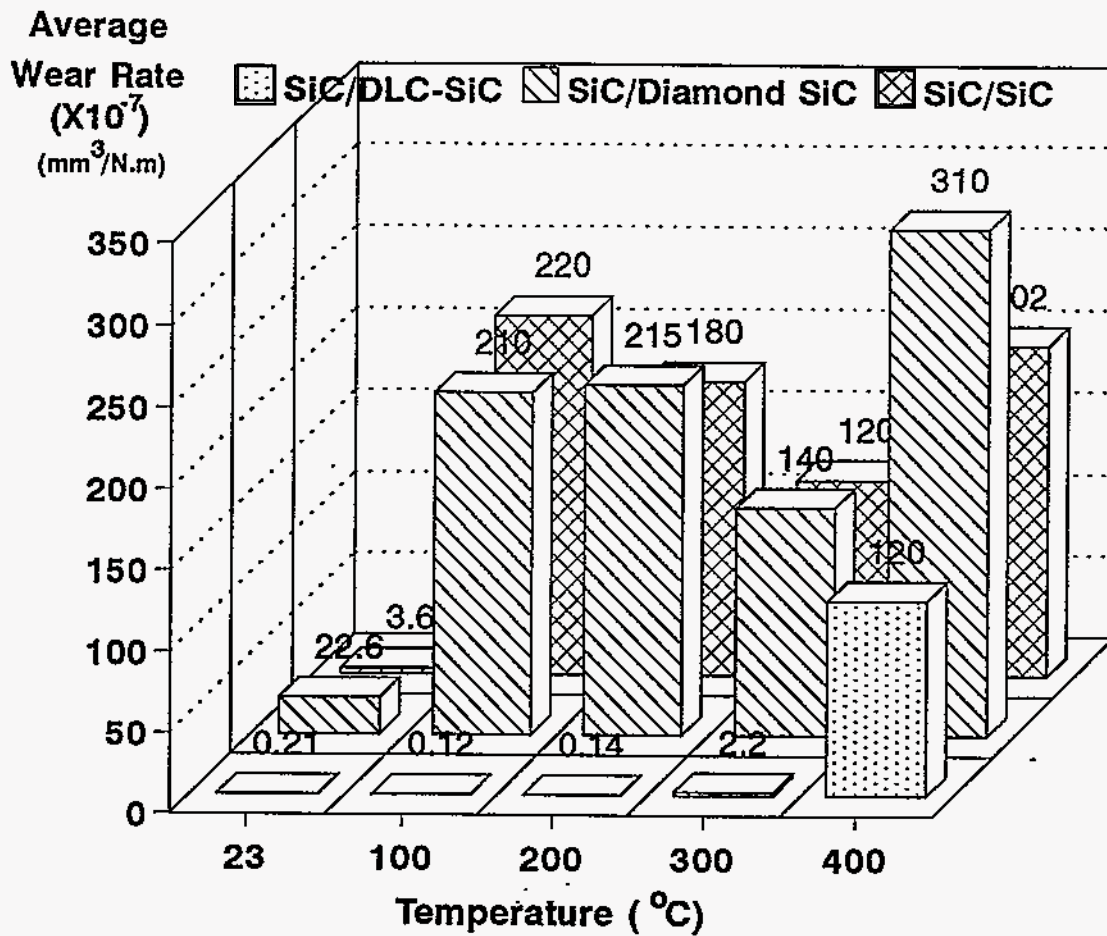
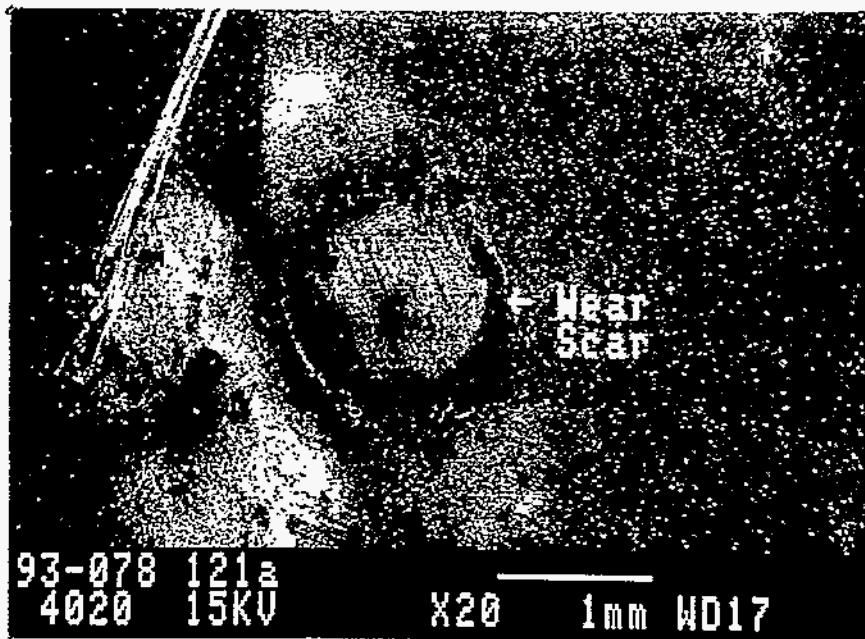
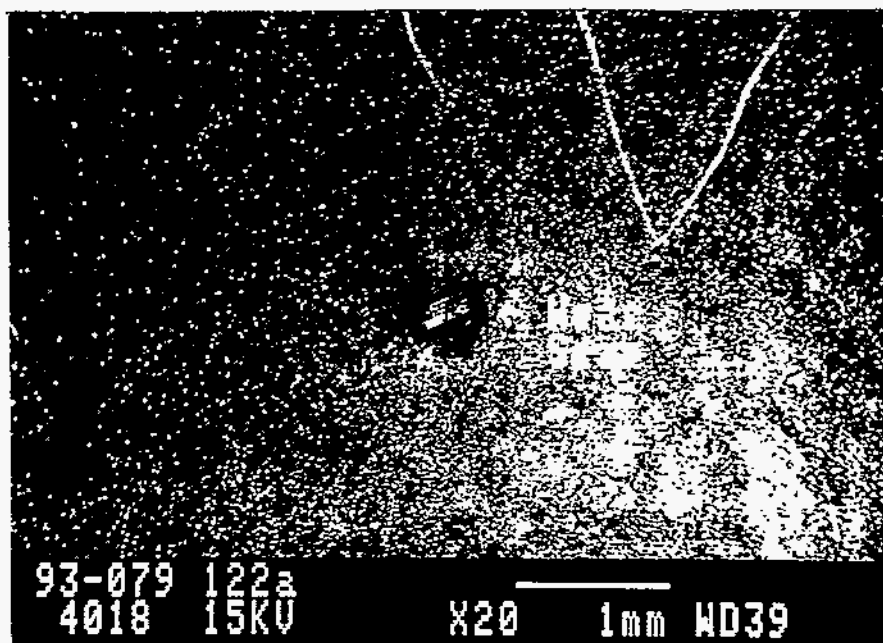
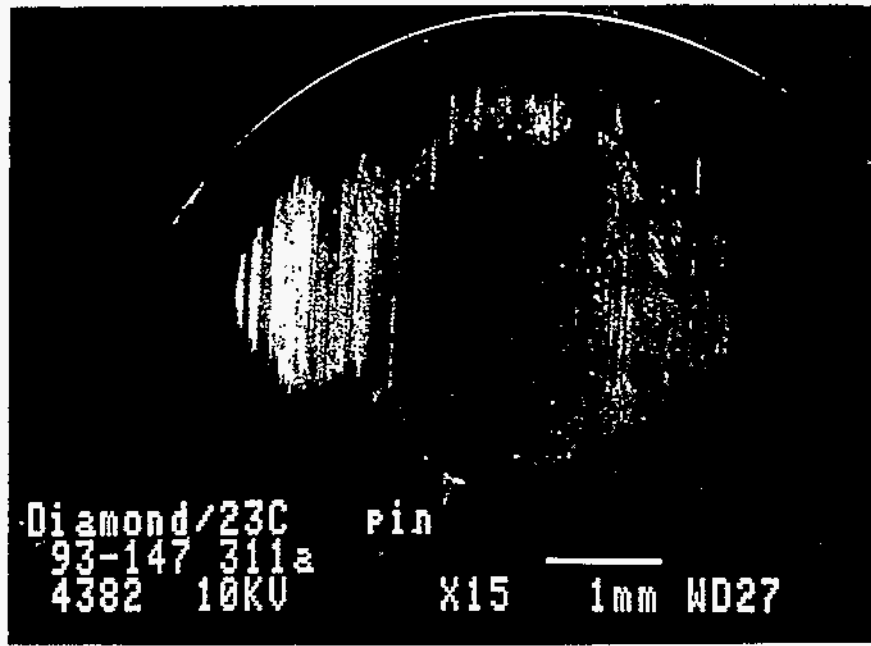


Figure 6. Average wear rates of SiC pins during sliding against uncoated, DLC, and diamond-coated SiC flats at temperatures up to 400°C (test conditions: load, 10 N; speed, 0.05 m/s; sliding distance, 200 m; relative humidity, 30%).



(a)





(c)

Fig. 7. SEM photomicrographs of wear scars formed on SiC pins during sliding against (a) uncoated, (b) DLC-coated and (c) diamond-coated SiC flats (test conditions: load, 5 N; speed, 0.05 m/s, sliding distance, 200 m; relative humidity, 30%; temperature, 23°C).

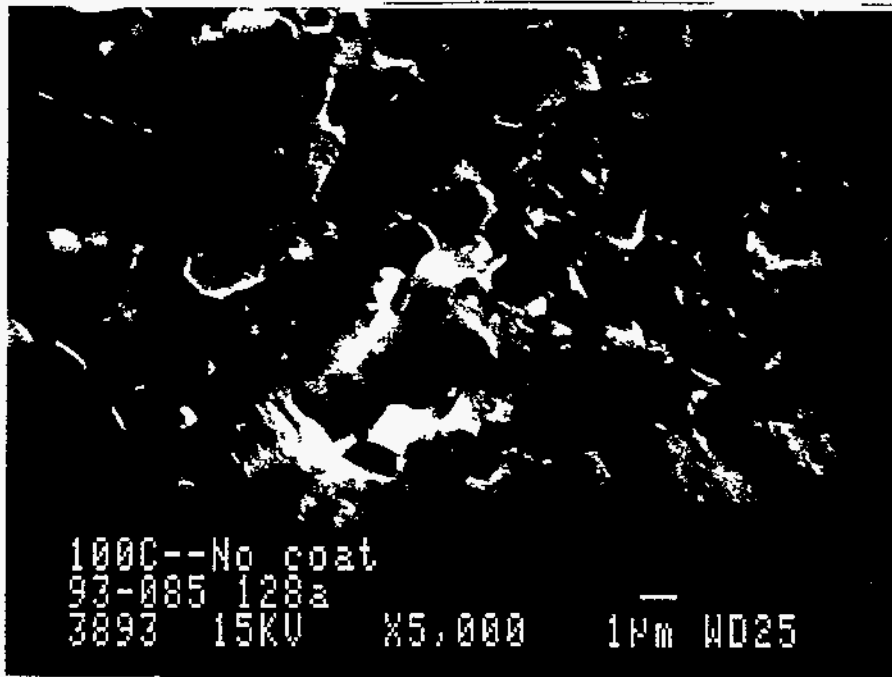
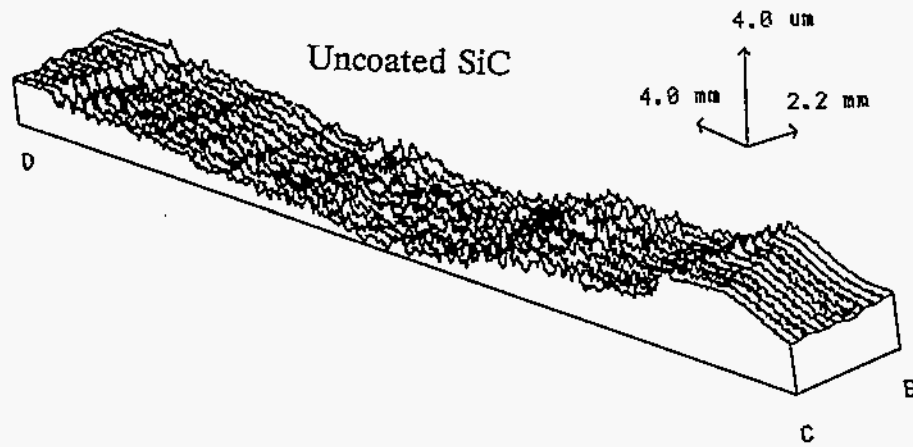
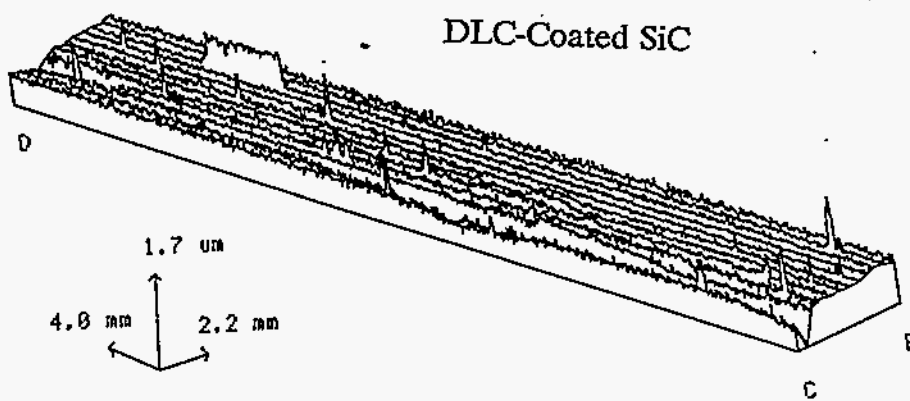


Fig. 8. SEM photomicrograph of wear surfaces of SiC flat formed during tests at 100°C.



(a)



(b)

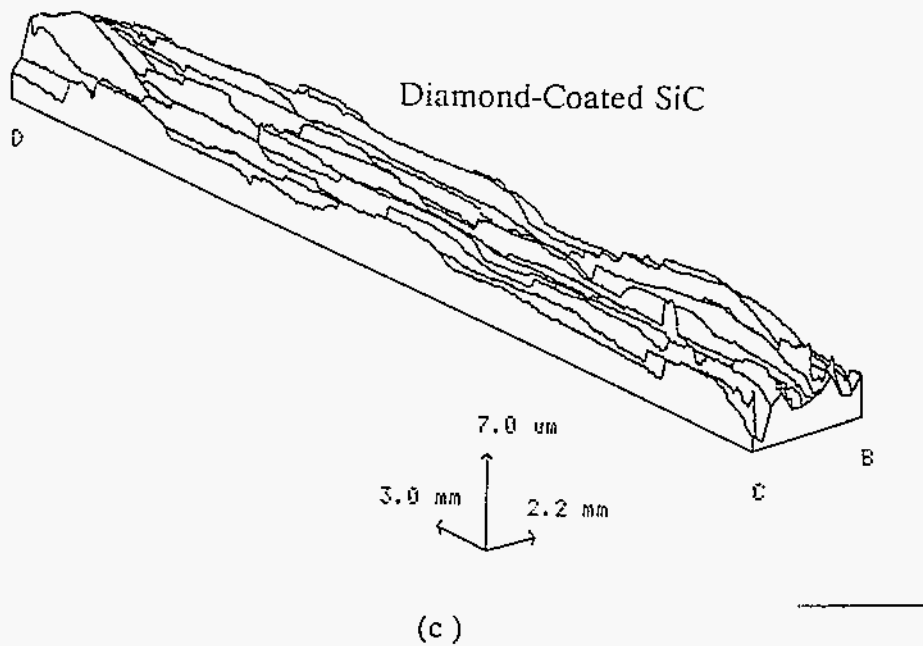


Figure 9. 3-D surface maps of wear tracks formed on (a) uncoated, (b) DLC-coated, and (c) diamond-coated SiC flat (test conditions: load, 10 N; sliding velocity, 0.05 m/s; relative humidity, 20%; sliding distance, 200 m; temperature, 200°C).

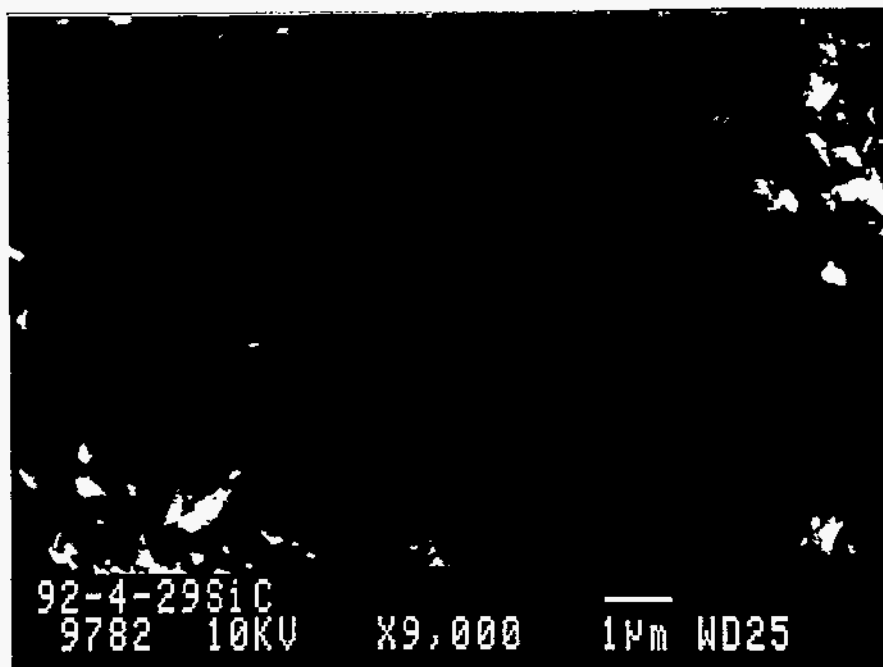


Fig. 10. SEM photomicrograph of worn surface of diamond-coated SiC flat formed during tests at 200°C.

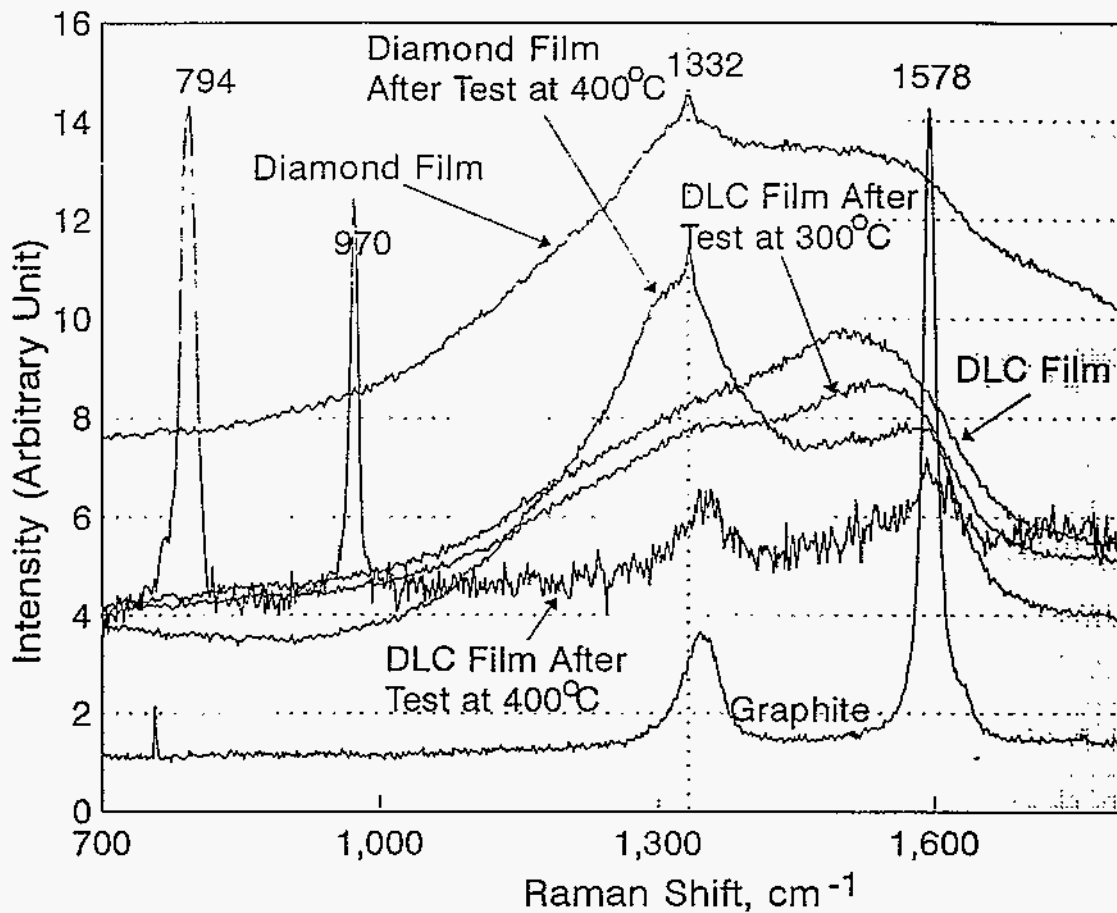


Fig. 11. Raman spectra of graphite and diamond and DLC films before and after tests at various temperatures.



Fig. 12. SEM photomicrograph of worn surface of diamond-coated SiC flat after test at 400°C.