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**FRICION AND WEAR PERFORMANCE OF ION-BEAM DEPOSITED DIAMONDLIKE CARBON FILMS ON STEEL SUBSTRATES\***

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# FRICITION AND WEAR PERFORMANCE OF ION-BEAM DEPOSITED DIAMONDLIKE CARBON FILMS ON STEEL SUBSTRATES\*

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## ABSTRACT

In this study, we investigated the friction and wear performance of ion-beam-deposited diamondlike-carbon (DLC) films (1.5  $\mu\text{m}$  thick) on AISI 440C steel substrates. Furthermore, we ran a series of long-duration wear tests under 5, 10, and 20 N load to assess the load-bearing capacity and durability limits of these films under each load. Tests were performed on a ball-on-disk machine in open air at room temperature  $\approx 22 \pm 1^\circ\text{C}$ , and humidity,  $\approx 30 \pm 5\%$ . For the test conditions explored, we found that (1) the steady-state friction coefficients of pairs without a DLC film were in the range of 0.7 to 0.9 and the average wear rates of 440C balls (9.55 mm diameter) sliding against uncoated 440C disks were on the order of  $10^{-5} \text{ mm}^3/\text{N.m}$ , depending on contact load; (2) DLC films reduced the steady-state friction coefficients of test pairs by factors of 6 to 8, and the wear rates of pins by factors of 500 to 2000; (3) The wear of disks coated with a DLC film was virtually unmeasurable while the wear of uncoated disks was quite substantial, (4) these DLC films were able to endure the range of loads, 5 to 20 N, without any delamination and to last over a million cycles before wearing out. During long-duration wear tests, the friction coefficients were initially on the order of 0.15, but decreased to some low values of 0.05 to 0.07 after sliding for 15 to 25 km, depending on the load, and remained low until wearing out. This low-friction regime was correlated with the formation of a carbon-rich transfer film on the wear scar of 440C balls. Micro-laser-Raman spectroscopy and scanning-electron microscopy were used to examine the structure and chemistry of worn surfaces and to elucidate the wear- and friction-reducing mechanisms of the DLC film.

## INTRODUCTION

Because of their attractive properties, diamond and diamondlike-carbon (DLC) films have gained considerable interest in high-technology tribological industries in recent years. Extreme hardness, high elastic modulus, excellent wear and corrosion resistance, high thermal and chemical stability, and the low-friction nature of these films make them good prospects for a wide range of tribological applications, such as magnetic hard disks, ball and rolling bearings, high-precision gears, cutting tools, mechanical seals, etc. [1-6].

The DLC films can be deposited at low substrate temperatures and at fairly high deposition rates by such methods as ion-beam deposition, DC and RF sputtering, ion plating, cathodic-arc, and plasma-enhanced chemical vapor deposition [1,5]. However, the synthesis of high-quality diamond films is currently feasible only at high deposition temperatures ( $>500^{\circ}\text{C}$ ) and at low growth rates. The individual grains of diamond films tend to grow preferentially in certain crystallographic orientations and the surface finish of these films is highly-faceted and generally rough. Current emphasis on diamond-film technology is directed toward the development of new methods and/or procedures that can allow the synthesis of diamond films at lower deposition temperatures and faster rates. A smoother surface finish is also much to be desired.

Apparently, DLC films lack the high thermal conductivity of diamond, but their electronic, mechanical, and optical properties are said to be comparable to those of the diamond films. Like diamond, they are mechanically rigid, chemically inert, optically transparent, and electronically insulating [1,2,7]. The reported hardness values of DLC films vary between  $\approx 2000$  and  $\approx 9000$   $\text{kg}/\text{mm}^2$  compared with diamond-film values of  $8000$ - $10,000$   $\text{kg}/\text{mm}^2$  [1,2,8,9]. Unlike diamond,

DLC films are amorphous and lack preferred growth orientation, hence their surface finish is highly smooth. Within the amorphous structure of DLC films, there exist very-short-range-ordered diamond (characterized by  $sp^3$ -type tetrahedral bonds) and graphite (characterized by  $sp^2$ -type trigonal bonds) precursors [1,9]. Accordingly, they are characterized as degenerate or imperfect forms of diamond and/or graphite, with properties ranging from diamondlike to graphitelike [1]. Depending on the carbon source and deposition technique, large amounts of hydrogen can be incorporated in the amorphous structure of DLC films [1,7,9,10]. They are sometimes referred to as hydrogenated amorphous-carbon films. Apparently, hydrogen plays a critical role in the stabilization of the  $sp^3$ -type tetrahedral bonds, and has profound effects on optical, electronic, and mechanical properties of these films [1,9].

The friction coefficient of DLC films is influenced markedly by counterface material, growth processes and conditions, and test environments [1,2,10-17]. Among others, test environment and relative humidity have the greatest effect on the frictional behavior of DLC films. For instance, in dry air,  $N_2$  and/or inert test environments, the friction coefficients of DLC against both metallic and ceramic materials can vary between 0.01 and 0.05. In vacuum, the friction coefficients are even lower. Enke reported a friction coefficient of 0.005 for a DLC film sliding against a steel ball [12]. However, in humid air, friction coefficients can reach values as high as 0.3. Also, at elevated temperatures ( $>400^\circ\text{C}$ ), the DLC films were found to lose their diamondlike character and become increasingly graphitelike with friction coefficients typical of graphite precursor [10,15,18].

The purpose of this study is to investigate the friction and wear performance of DLC films produced on 440C bearing steels by ion-beam deposition. Specifically, the effects of load on friction and wear performance of DLC films were evaluated and a series of long-duration wear

tests were run under 5, 10, and 20 N to assess the load-bearing capacity and lifetime of these films. Laser-Raman spectroscopy and scanning-electron microscopy were used to examine the structural and chemical nature of DLC films and other test materials.

## **EXPERIMENTAL PROCEDURES**

### **Test Materials**

The ball and disk specimens used in this study were fabricated from AISI 440C bearing steel. The disk specimens, 50 mm in diameter by 5 mm thick, were ground to a surface finish of  $0.1\pm 0.02$   $\mu\text{m}$  center-line average (CLA). The counterface 440C balls were 9.5-mm in diameter with a surface finish of better than 0.05  $\mu\text{m}$  CLA. Balls and disks had a Vickers hardness value of  $8.0\pm 0.2$  GPa. Prior to friction testing and/or DLC coating, disks 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.

### **Ion-Beam Deposition of Diamondlike Carbon**

Thin DLC films  $\approx 1.5$   $\mu\text{m}$  thick were ion-beam deposited on 440C disks at room temperature in a vacuum chamber equipped with a broad-beam ion source. A schematic illustration of this deposition system is shown in Fig. 1. In this deposition system, methane - the carbon source - was fed through a cylindrical ion source (see Fig. 1) and ionized by energetic electrons produced by a hot filament wire.

DLC films were applied on 440C substrates in a three-step process. Specifically, the samples were positioned directly under the ion source and sputter-cleaned with a 1 keV, 2.5 mA/cm<sup>2</sup> Ar-ion beam for about 3 to 5 min. Subsequently, an intermediate hydrogenated-silicon carbide (SiC:H) bondlayer was sputter-deposited on these substrates by rotating the samples out of the beam and into the sputter-coating position as illustrated in Fig. 1. Simply, methane is bled into the vacuum chamber and the 1 keV argon beam is allowed to sputter the silicon target shown in the figure. The combination of the Ar-ion-sputtered Si and the C and H derived from the methane results in a SiC:H bond layer of 10- to 20-nm thickness. Our experience has shown that this thickness range is sufficient for attaining good adhesion between the substrate steel and DLC films. Without the SiC:H layer, we could not obtain good bonding between DLC films and steel substrates.

At the conclusion of the SiC:H step, the source was switched back from Ar to methane operation. Specifically, the disk samples were rotated to a position directly beneath the beam, and the deposition of DLC was commenced. Operating conditions were adjusted to give an ion beam with an acceleration energy of 450 eV and a current density of  $\approx 2.5$  mA/cm<sup>2</sup>. At this operating condition, the deposition rate was  $\approx 3$   $\mu$ m/hr. Deposition was continued until a 1.5  $\mu$ m-thick DLC film was obtained. Substrates were water-cooled via their mounting plates to keep their temperatures significantly below the tempering temperature ( $\approx 175^\circ\text{C}$ ) of 440C.

### **Friction and Wear Tests**

Friction and wear tests were performed with pairs of 440C balls and DLC-coated and uncoated 440C disks on a ball-on-disk tribometer in open air at room temperature,  $23 \pm 1^\circ\text{C}$ , and a humidity of  $30 \pm 5\%$ . The sliding velocity and distance were 0.5 m/s and 1 km, respectively. Tests with

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uncoated disks were also performed and their friction and wear performance was compared to that of the 440C disks with a DLC film. The dead weight applied to the ball ranged from 1 to 20 N, which created initial mean Hertzian contact pressures of approximately 0.32 to 0.87 GPa. Frictional force was monitored with the aid of a linear variable-displacement transducer and was recorded on chart papers throughout the tests. A series of long-duration wear tests were run under 5, 10, and 20 N load and at 0.5 m/s to primarily assess the load-bearing capacity, endurance life, and long-term friction and wear performance of the DLC films under each load. The diameter of the wear track varied between 34 and 41 mm during these tests, producing steadily decreasing nominal pressures far below the initial mean Hertzian pressures.

Wear-volume calculations on the balls were based on microscopic determination of the diameter of the circular wear scars combined with the assumption that the wear scar is flat. The wear of disk specimens was estimated from the traces of surface profiles across the wear tracks. Two to three tests were run under conditions described above to check the reproducibility of the friction and wear data. The results were quite reproducible, with deviations of  $\approx \pm 6$  to  $\pm 15\%$ . Wear scars and tracks were examined with a scanning electron microscope and surveyed in 3-D with a surface profilometer.

## **RESULTS AND DISCUSSION**

### **DLC Film Microstructure and Chemistry**

Figure 2 shows the microstructure of a typical DLC film produced in this study. This film was deposited on a thin ( $\approx 1$  mm-thick) SiC substrate under conditions similar to those employed during the coating of 440C substrates. The highly brittle nature of the SiC substrate made it possible to

fracture this DLC film in cross section and to examine its microstructure at high magnifications. As is evident, this DLC film has a very dense morphology and appears to be free of volume defects. The columnar morphology, that is typical of most metallic and ceramic coatings produced by physical vapor deposition (e.g., reactive evaporation, sputtering, ion plating, etc.), is not evident in this DLC film. Also there is no evidence of a gap or discontinuity at the DLC/SiC interface suggesting that the DLC film bonds intimately to the SiC substrate.

Micro-laser Raman spectra of the DLC films used in this study revealed a peak at  $\approx 1356 \text{ cm}^{-1}$  (appearing as a shoulder in Fig. 3) and another broad peak centered at  $\approx 1552 \text{ cm}^{-1}$ . The shoulder peak at  $\approx 1356$  may have been due to a highly disordered graphite precursor, while the broad peak at  $\approx 1552 \text{ cm}^{-1}$  may have been due to amorphous carbon. In general, these Raman peaks are typical of the DLC films and consistent with the Raman data presented elsewhere [1,19].

Figure 4a shows the effect of increasing load on the steady-state friction coefficients of 440C balls during sliding against uncoated and DLC-coated 440C disks. As is clear, for the DLC-coated disks, the friction coefficient decreased steadily with increasing load. The steady-state friction coefficient was  $\approx 0.16$  under 1 N, but decreased to 0.12 as the load was increased to 10 N.

Compared to those very low friction coefficients (e.g., 0.005-0.05) in vacuum, dry air and/or inert environments [10-16], the friction coefficients of our DLC films (see Fig. 4a) are significantly higher. This is presumably because of the fairly humid test environment (e.g.,  $30 \pm 5\%$ ) that we had during our friction tests. The friction coefficients reported in this study are consistent with the previous friction results obtained in moderately to highly humid test environments [13,14,16,20,21]. One explanation given for the relatively higher friction coefficients in humid environments was that



the real contact areas of sliding surfaces became saturated with water molecules, and this created extra bonds across the contact interfaces thus causing higher friction [10]. For the much lower friction coefficients in vacuum, dry, and inert test environments, Memming et al. [13] and Kim et al. [16] proposed the formation of a transfer layer on the rubbing surfaces of counterface balls. The transfer films that they found on ball counterfaces were rich in carbon and had a structural chemistry similar to that of DLC film.

The steady-state friction coefficients of 440C/440C test pairs vary between 0.7 and 0.9, depending on normal load (see Fig. 4a). This range of friction coefficient is common for the dry sliding pairs of 440C steels.

Figure 4b presents the average specific wear rates of 440C balls during sliding against the uncoated and DLC-coated 440C disks under loads of 1 to 10 N. As is clear, the specific wear rates (normalized with respect to load) of those balls sliding against the DLC-coated 440C decreased with increasing load indicating the lack of validity of Archard's Law for these tests, which in turn indicate an average wear rate increasing less than linearly with increasing load. As for the DLC-coated 440C disks, their wear rates were very difficult to assess. Using a surface profilometer at vertical magnifications up to 50,000X, we could not detect any significant wear on substrate steels.

As shown in Fig. 4b, the balls slid against the uncoated disks experienced very high wear rates. Compared to those balls slid against the DLC-coated disks, these balls wore at average rates of nearly 500 times higher under a 1 N load and more than 2000 times higher under 10 N. Furthermore, as is clear from a 3-D surface map in Fig. 5, the uncoated disks sliding against these balls experienced severe alterations in surface topography, but wear volumes were very difficult

to estimate in any quantitative way. As mentioned above, with increasing wear scar diameters the nominal bearing pressure steadily decreases. The numbers near the data points in Fig. 4b show the values at the end of the tests. These are to be compared with the initial mean Hertzian pressures of 320 MPa for 1 N to 670 MPa for 10 N.

Figure 6 shows the friction and wear performance of DLC coatings during lifetime tests under 5, 10, and 20 N loads. As is clear, both the lifetime and the friction coefficient of DLC films are affected by the contact load. Initially, the friction coefficients of pairs were lower under heavier contact loads. This observation is qualitatively consistent with what was presented in Fig. 4a, and the absolute values here are only slightly higher for 5 and 10 N. However, after about 50,000 revolutions, a reverse trend became increasingly noticeable. Specifically, the friction coefficients became higher under heavier loads. As shown in Fig. 6, beyond  $\approx 200,000$  revolutions, the friction coefficient is  $\approx 0.05$  under 5 N, as opposed to  $\approx 0.07$  under a 20 N load. Also, under higher loads, the lifetime of DLC films were relatively shorter. At a 5 N load, the DLC film had a lifetime of about 1,100,000 revolutions or 131 km sliding distance, whereas at a 20 N load, the lifetime was about 850,000 revolutions or 91 km sliding distance. A 3-D surface map in Fig. 7 shows the physical condition of a wear track formed under a 20 N load on a DLC-coated 440C disk. The average wear rates (wear volume ( $\text{mm}^3$ ) divided by contact load (N) and total sliding distance (m)) of 440C balls slid against the DLC-coated 440C disks under 5, 10, and 20 N loads during lifetime tests were  $3 \times 10^{-9}$ ,  $2 \times 10^{-9}$ , and  $10^{-9}$   $\text{mm}^3/\text{N.m}$ , respectively. The first two rates (average over the entire tests), though possibly somewhat elevated due to the inclusion of the rising friction coefficient portion of the lifetime curves, are approximately half those calculated above for the cases of 5 and 10 N for 1 km sliding distance, which indicates the usual situation, i.e., that the instantaneous wear rates are decreasing as the sliding distance increases.

Overall, the wear-test results presented above demonstrate that ion-beam-deposited DLC films are quite resistant to wear and impart low friction to sliding steel surfaces. We believe that high mechanical hardness and the low-friction character of these carbon films were largely responsible for the low wear rates of both balls and disks. Also, it is clear from the lifetime tests that these films have excellent endurance lives and load-bearing capacity.

In an attempt to understand the mechanism of low friction and long life during lifetime tests, we conducted a series of special tests under 10N. Specifically, we allowed the sliding test to continue until reaching a friction coefficient of about 0.06. At that point, e.g.,  $\approx 250,000$  revolutions ( $\approx 29$  km sliding distance), we stopped the tests, recovered the balls, and inspected the wear scars under a scanning electron microscope. As shown in Fig. 8a, there were large colonies of transfer layers on the wear scars with an orientation parallel to the sliding direction. The corresponding wavelength-dispersive C X-ray map in Fig. 8b revealed that these colonies were rich in carbon. However, similar inspection on the wear scars of balls used in short-distance (e.g., 1 km as denoted in Fig. 6) tests of Fig. 4 did not reveal any type of transfer layer. It is possible that a very thin transfer layer may have formed on the wear scars of these balls too, but it may have been too thin or beyond the detection limit of our wavelength-dispersive X-ray probe. In short, we believe that the much lower steady-state friction coefficients observed at large sliding distances during lifetime tests are related to the formation of a carbon-rich transfer layer on the sliding ball.

The availability of wear-scar diameters after three sliding distances of 1, 29, and 141 km, at 10 N gives us the capability of estimating the "instantaneous" wear rate at this constant load as a function of sliding distance. Using the change in wear volume and the change in elapsed sliding distance, these calculations yield  $3.6 \times 10^{-9}$ ,  $1.4 \times 10^{-9}$ , and  $1.6 \times 10^{-9}$  mm<sup>3</sup>/N.m after the three

respective sliding distances. Although subject to the uncertainties inherent in utilizing three different tests for these estimates, these numbers indicate an initially much higher wear rate which steadily decreases with sliding distance (hence with decreasing nominal pressure). The slightly larger value calculated at the longest distance may arise from an enhanced wear during the final rising-friction-coefficient portion of the lifetime test.

As mentioned earlier, Memming et al. [13] and Kim et al. [16] have also reported low friction coefficients for their tribosystems in which steel and  $\text{Si}_3\text{N}_4$  balls were rubbed against DLC films. They found that the wear scars of balls were covered with a carbon-rich transfer layer to which they attributed the low friction coefficients. In another study, Hirvonen et al. reported the formation of a carbon-rich transfer layer on steel balls after a long period of sliding and have argued that relatively lower steady-state friction coefficients were due to the carbon-rich transfer layers [21]. In their friction tests, the friction coefficients were initially 0.17 but decreased to  $\approx 0.12$  after the formation of a carbon-rich transfer film on the wear scars.

## CONCLUSIONS

1. In general, DLC films prepared by the ion-beam-deposition method are very wear-resistant, long-lasting, and capable of imparting low friction to sliding steel surfaces.
2. The steady-state friction coefficients of these films are affected by the contact loads and the formation of a transfer film on the counterface balls.

3. For those tests (i.e., short sliding distance of 1 km) where little or no transfer film was detected, the friction coefficients decreased with increasing load, whereas, in the lifetime tests where a carbon-rich transfer film was present on the ball (after sliding distances of 20 to 25 km) the friction coefficients were in the range of 0.05 to 0.07 and they tended to increase with increasing load.

4. The lifetime of DLC films is dependent on contact load. Generally, the higher the contact load, the shorter the lifetime.

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**FIGURE CAPTIONS**

Fig. 1. Schematic depiction of the ion-beam-deposition system.

Fig. 2. Cross-sectional scanning electron micrograph of a DLC film used in this study.

Fig. 3. Micro-laser-Raman Spectrum of a DLC film used in this study

Fig. 4. Variation of (a) friction coefficient and (b) average wear rate of 440C balls during sliding against DLC-coated and uncoated 440C disk with increasing load (The numbers near the data points show the final nominal bearing pressures in MPa). The sliding velocity was 0.5 m/s.

Fig. 5. 3-D surface map of wear tracks formed on uncoated 440C during sliding against 440C ball under 10-N load at sliding velocity of 0.5 m/s.

Fig. 6. Variation of friction coefficient of 440C balls during sliding against DLC-coated 440C disk in lifetime tests under 5, 10, and 20N loads with wear-track diameters of 36, 41, and 34 mm, respectively. The sliding velocity was 0.5 m/s.

Fig. 7. 3-D surface map of wear track formed on DLC-coated 440C disk during lifetime test under 20N at sliding velocity of 0.5 m/s.

Fig. 8. (a) Scanning electron micrograph and (b) corresponding carbon X-ray map revealing the formation of a C-rich transfer film on a wear scar formed during an interrupted lifetime test under 10 N load at 0.5 m/s sliding velocity.



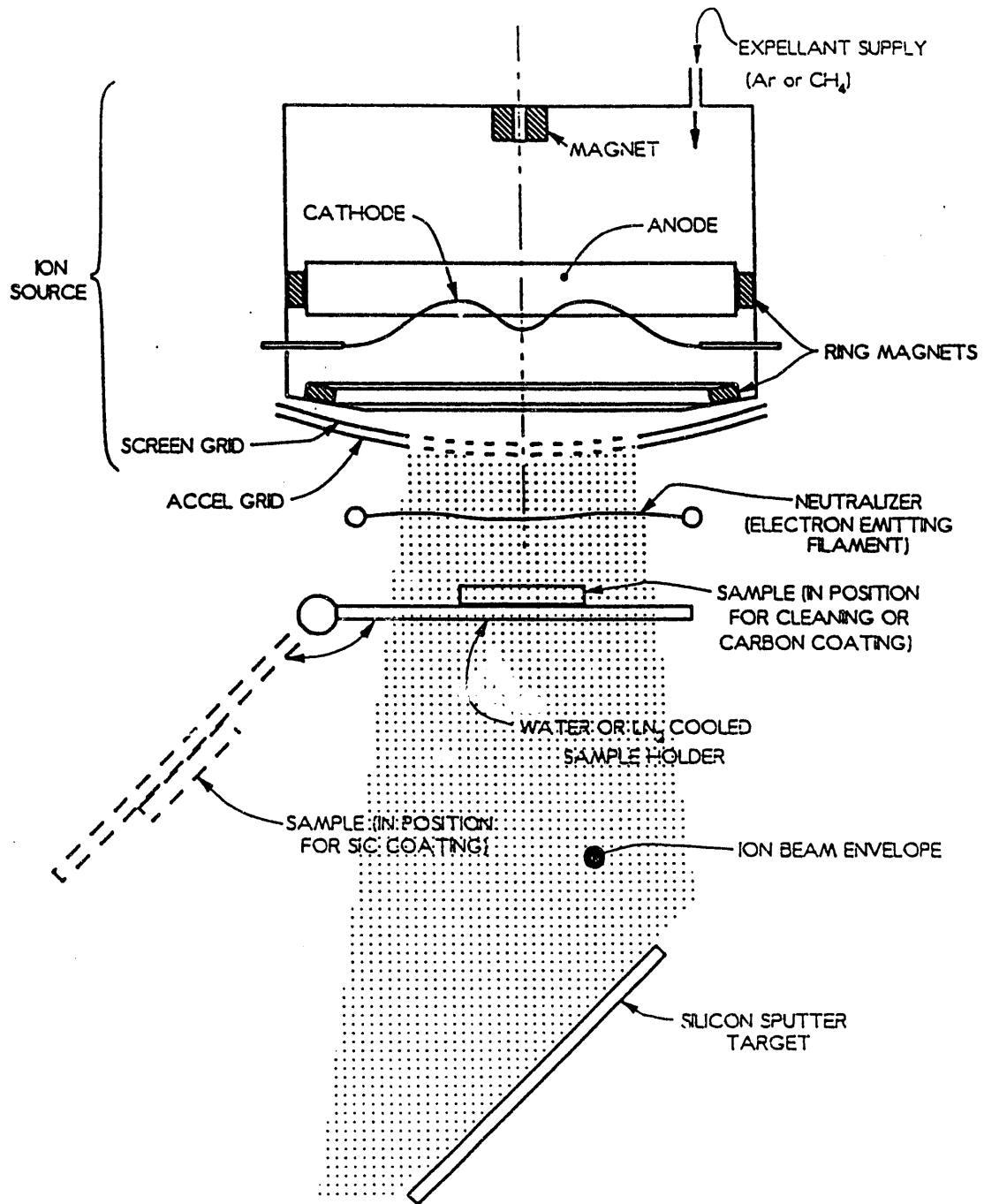


Fig. 1. Schematic depiction of the ion-beam-deposition system.

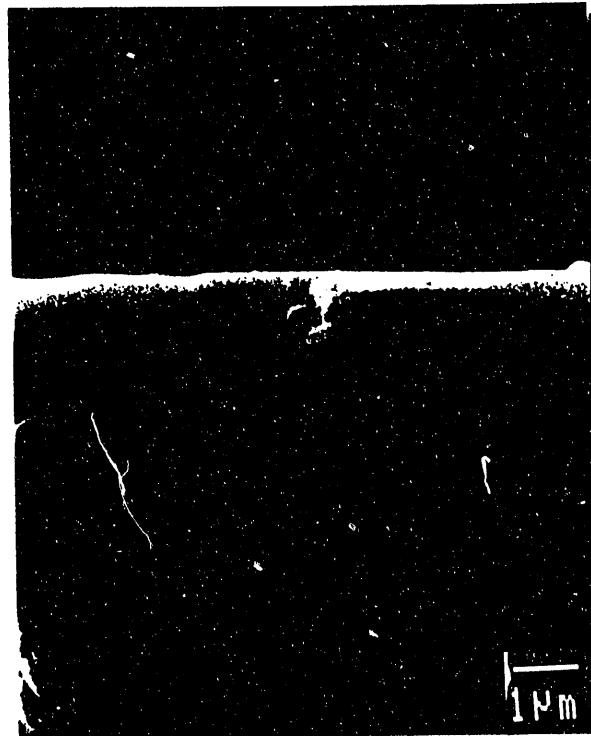


Fig. 2. Cross-sectional scanning electron micrograph of a DLC film used in this study.

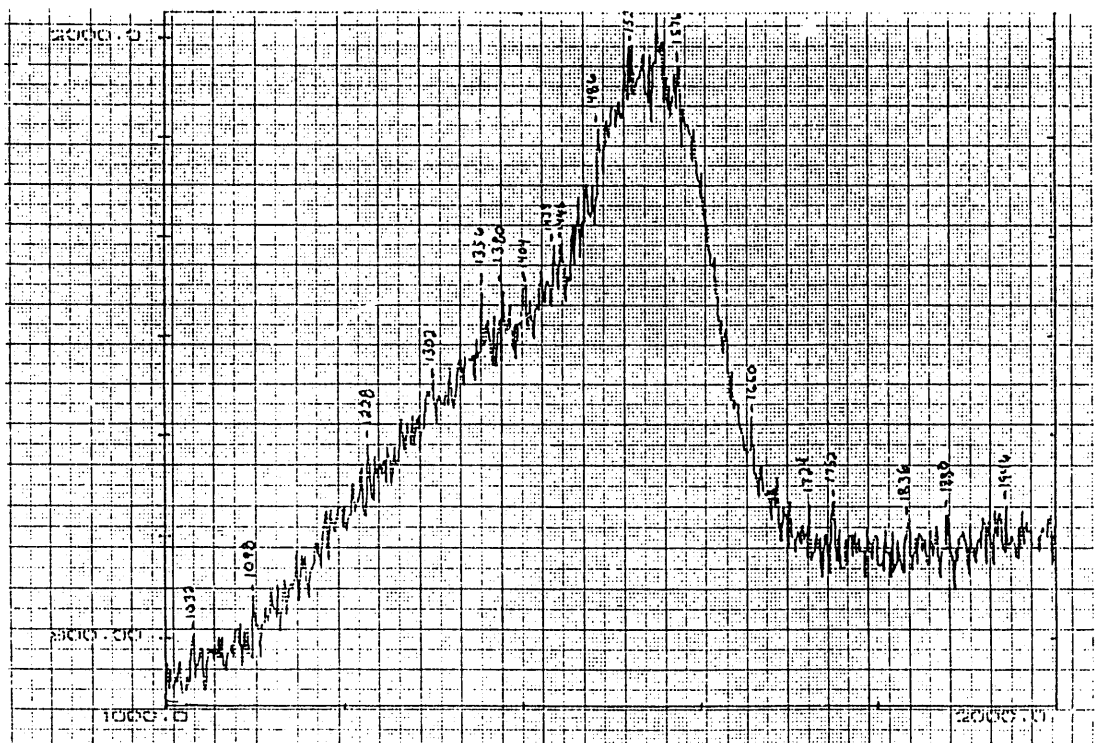
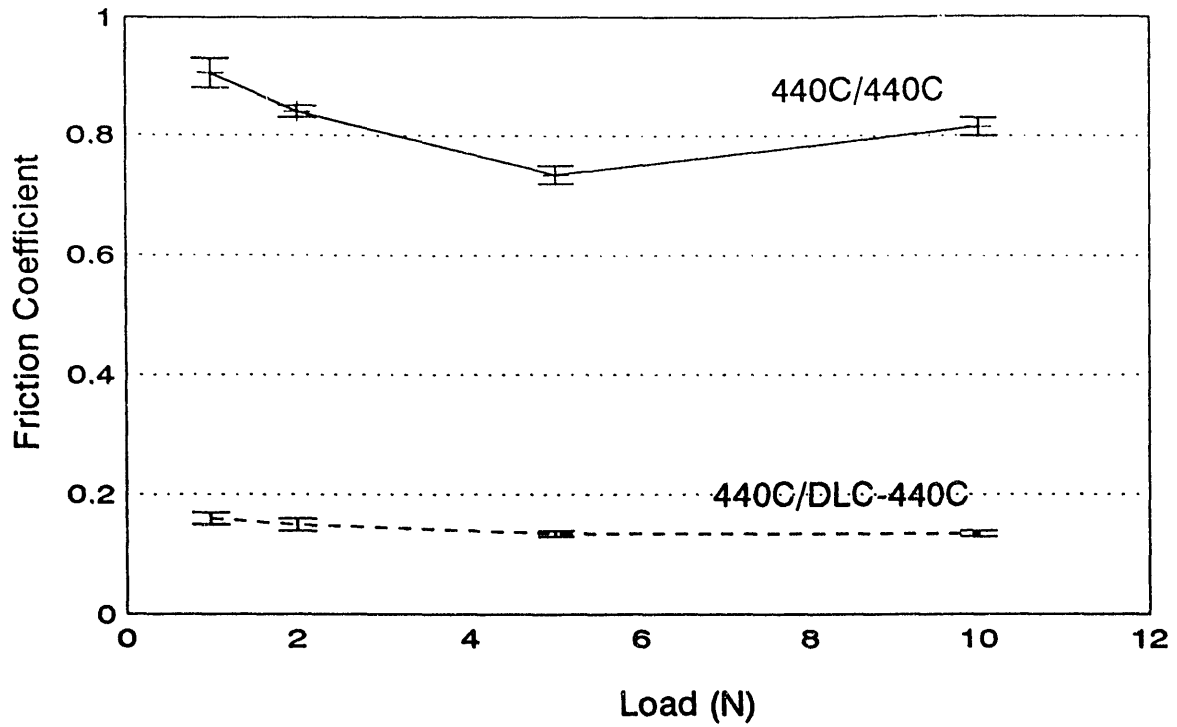
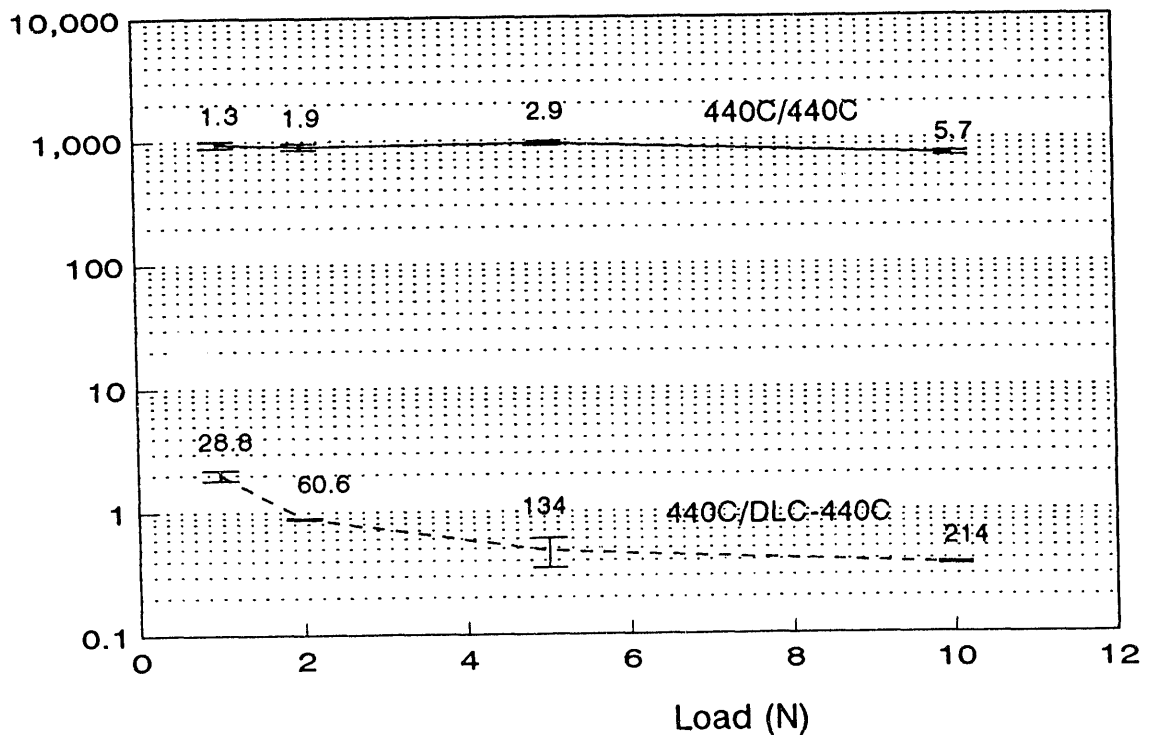


Fig. 3. Micro-laser-Raman Spectrum of a DLC film used in this study



(a)

Average Wear Rate  
( $\times 10^{-8} \text{ mm}^3/\text{N.m}$ )



(b)

Fig. 4. Variation of (a) friction coefficient and (b) average wear rate of 440C balls during sliding against DLC-coated and uncoated 440C disk with increasing load (the numbers near the data points show the final nominal bearing pressures in MPa). The sliding velocity was 0.5 m/s.

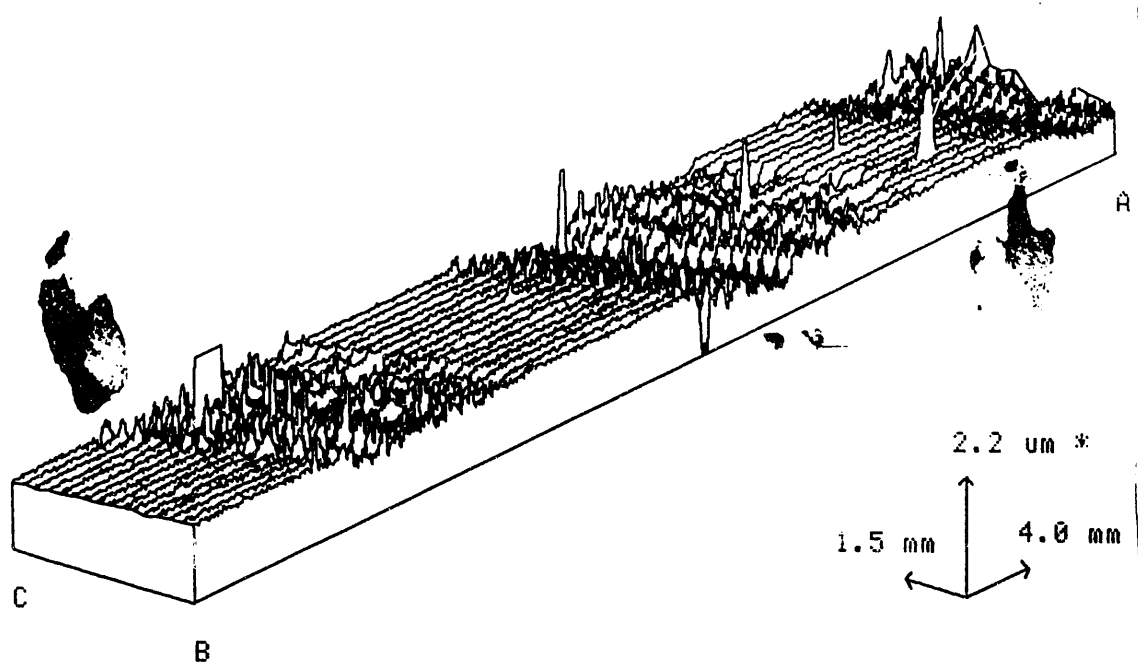


Fig. 5. 3-D surface map of wear tracks formed on uncoated 440C during sliding against 440C ball under 10-N load at sliding velocity of 0.5 m/s.

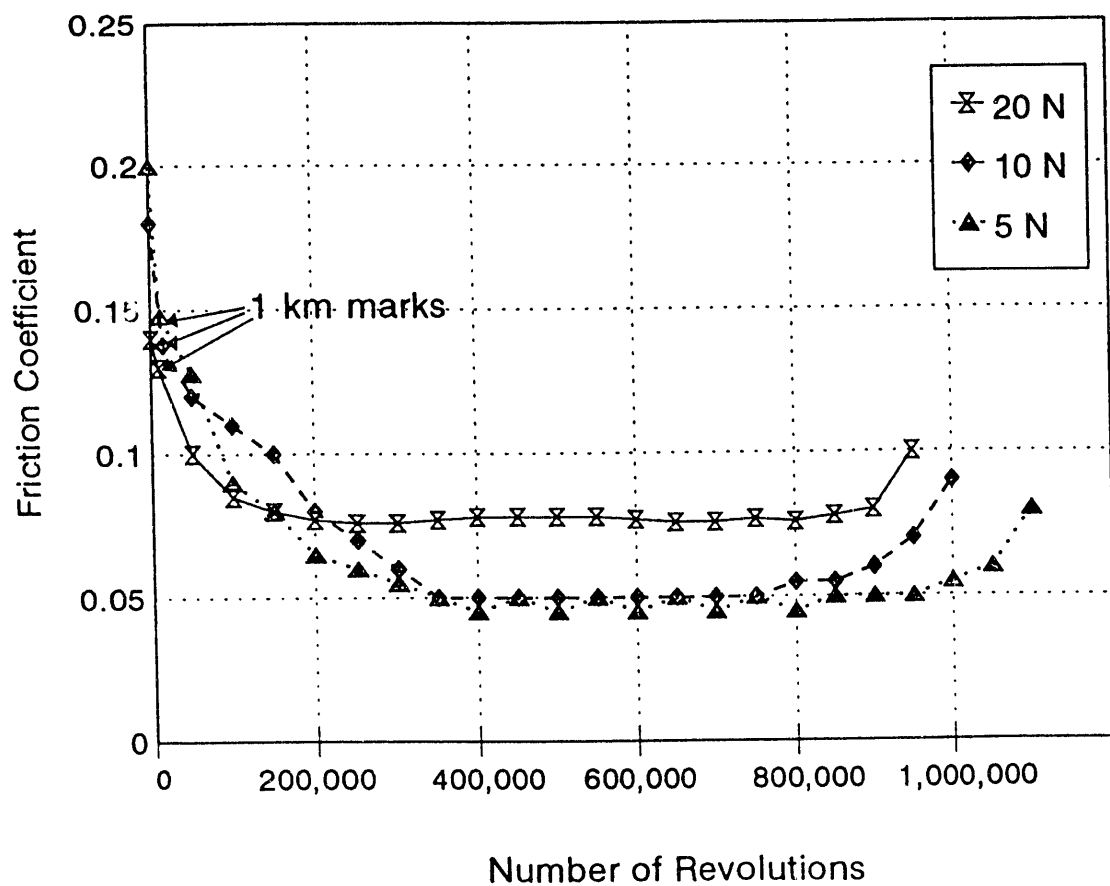


Fig. 6. Variation of friction coefficient of 440C balls during sliding against DLC-coated 440C disk in lifetime tests under 5, 10, and 20N loads with wear-track diameters of 36, 41, and 34 mm, respectively. Sliding velocity was 0.5 m/s.

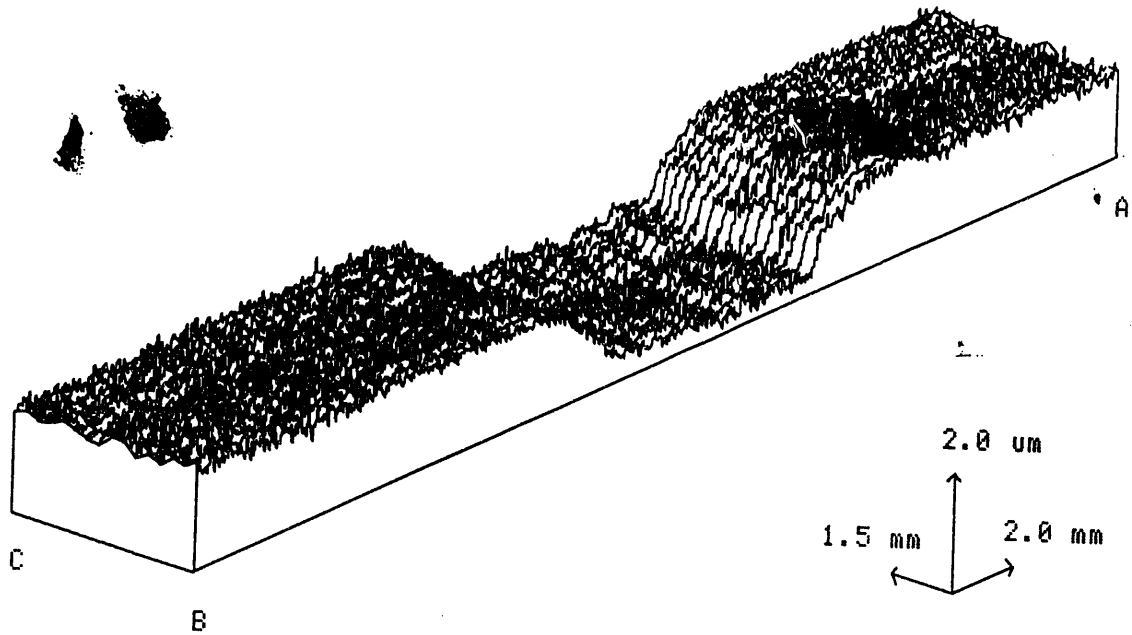
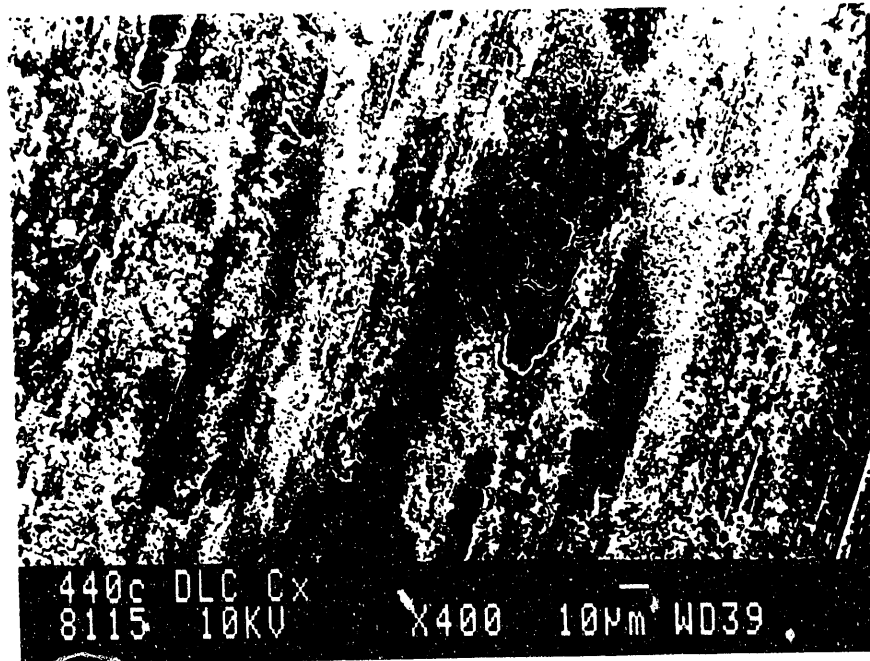
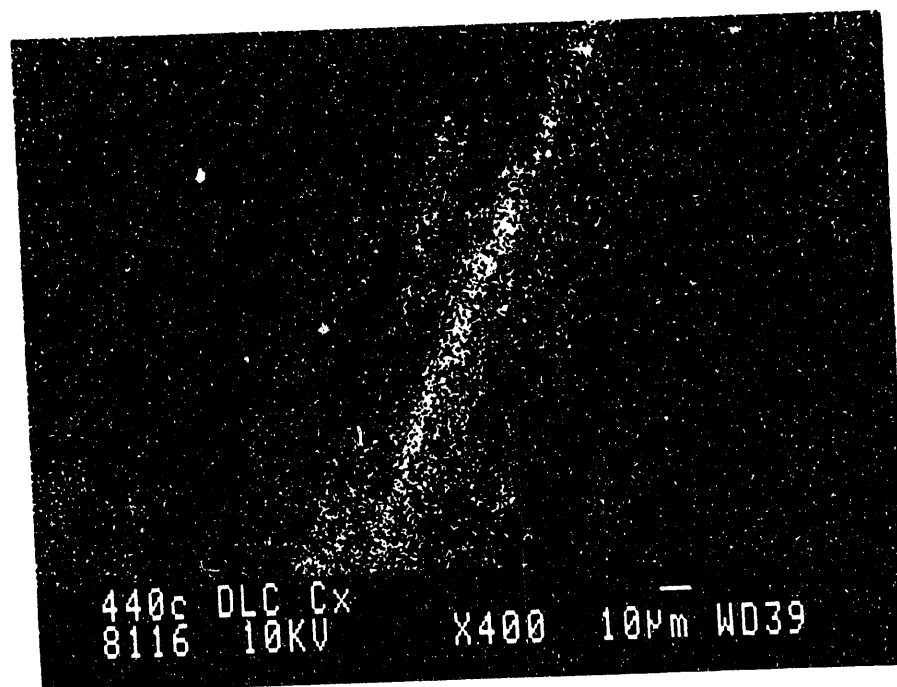


Fig. 7. 3-D surface map of wear track formed on DLC-coated 440C disk during lifetime test under 20N at sliding velocity of 0.5 m/s.



(a)



(b)

Fig. 8. (a) Scanning electron micrograph and (b) corresponding carbon X-ray map revealing the formation of a C-rich transfer film on a wear scar formed during an interrupted lifetime test under 10 N load at 0.5 m/s sliding velocity.

**END**

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