



# CVD Diamond, DLC, and c-BN Coatings for Solid Film Lubrication

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# CVD DIAMOND, DLC, AND c-BN COATINGS FOR SOLID FILM LUBRICATION

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## SUMMARY

The main criteria for judging coating performance were coefficient of friction and wear rate, which had to be less than 0.1 and  $10^{-6}$  mm<sup>3</sup>/N·m, respectively. Carbon- and nitrogen-ion-implanted, fine-grain, chemical-vapor-deposited (CVD) diamond and diamondlike carbon (DLC) ion beam deposited on fine-grain CVD diamond met the criteria regardless of environment (vacuum, nitrogen, and air).

## INTRODUCTION

Diamond's excellent properties make it an ideal material for many tribological applications. Its extreme hardness, high abrasion resistance, good fatigue strength, high thermal conductivity, good radiation and temperature resistance, chemical and thermal inertness, high corrosion resistance, and environmental compatibility (refs. 1 and 2) can suit it for bearings, valves, and engine parts in the harsh environments of internal-combustion and jet engines. Oxidation is less rapid in air than in pure oxygen. The onset of oxidation has been shown to start at 500 °C (ref. 1). Any carbon material would be oxidized away under combustion conditions. Diamond burns brightly in an oxygen jet at 720 °C (ref. 1).

Modern diamond belongs to one of four distinct categories: natural diamond, high-pressure synthetic diamond, chemical-vapor-deposited (CVD) diamond, and diamondlike carbon (DLC). Both natural and high-pressure synthetic diamond have limited applications because of the crystals' small size and high cost. Another limiting factor for tribological applications is the separate operation required to bond the crystals to a substrate. CVD diamond, on the other hand, offers a broader potential, since size and, eventually, cost are less of a limitation. CVD diamond, available in planar film or sheet form, opens the door for design engineers and tribologists to take full advantage of diamond's intrinsic properties in wear, solid lubrication, erosion, and corrosion applications.

The major drawbacks of CVD diamond, which restrict its use as a tribological coating, are its low bending strength and its very high deposition temperature. These obstacles must be overcome before practical, reliable, and cost-effective diamond coatings will become available as wear-resistant, self-lubricating barriers for many moving mechanical assemblies. A process must be developed that will keep the deposition temperature below 400 °C and provide consistently satisfactory adhesion to metallic and nonmetallic substrates, including steel and silicon nitride (Si<sub>3</sub>N<sub>4</sub>).

Diamondlike carbon (DLC) coatings have properties similar to those of CVD diamond and, without the high-temperature substrate requirements and with little restriction on size, are easier to process. DLC has two disadvantages: low deposition rate and high internal residual stress. Unlike diamond it cannot be obtained as thick monolithic shapes, at least with the present technology (refs. 1 and 2).

The tribological applications of CVD diamond and DLC may be limited by their pronounced reactivity with iron and iron-based alloys. Cubic boron nitride (c-BN) is an alternative that overcomes this problem. Cubic boron nitride has the second highest hardness next to diamond, high thermal conductivity, and high chemical and thermal stability (ref. 3). Therefore, c-BN coatings are promising as wear-resistant, self-lubricating coatings in contact with iron and iron-based alloys. As for DLC the major disadvantage of c-BN coatings is high internal residual stress.

To determine their friction and wear behavior, sliding friction experiments were conducted with CVD diamond films, DLC films, and c-BN films in contact with a hemispherical (1.3-mm radius of curvature) natural diamond pin or with a hemispherical (1.6-mm radius of curvature) CVD diamond pin in humid air, in dry nitrogen, and in ultrahigh vacuum. The diamond films were produced by microwave-plasma-enhanced CVD (ref. 4) and hot-filament CVD (ref. 5) techniques; the DLC films, by an ion-beam deposition technique (ref. 6); and the c-BN films, by a reactive ion plating technique (ref. 7). These films were deposited on flat surfaces of (100) silicon,  $\alpha$ -silicon carbide (SiC), and Si<sub>3</sub>N<sub>4</sub>. Further, various analytical techniques—including Raman

spectroscopy, hydrogen forward scattering (proton recoil analysis), Rutherford backscattering (RBS), transmission (TEM) and scanning electron microscopy, x-ray photoelectron spectroscopy (XPS), and x-ray diffraction—were used to characterize the films.

Some earlier data and experimental details on this research are given in references 4 to 12.

## MATERIALS

### CVD Diamond

Four different surfaces of CVD diamond films were investigated: as deposited, fine grain; polished, coarse grain; carbon ion implanted, fine grain; and nitrogen ion implanted, coarse grain (ref. 12).

#### As-Deposited, Fine-Grain CVD Diamond

As-deposited, fine-grain diamond films were produced by microwave-plasma-enhanced CVD and were primarily polycrystalline (ref. 4). The diamond films consisted of carbon and some elements from the substrate material, silicon. The hydrogen concentration was estimated to be 2.5 at.%. The crystallites were oriented along the  $\langle 110 \rangle$  direction. Grain size ranged from 20 to 100 nm. The lattice constants calculated from transmission electron diffraction patterns matched well with diamond's known lattice constants (ref. 4). Crystallites had a granulated or spherulitic morphology. The surfaces contained spherical asperities ranging from 6 to 37 nm root mean square (rms). The XPS analysis indicated that the surfaces contained oxygen with carbon/oxygen ratios ranging from 8 to 12.

#### Polished, Coarse-Grain CVD Diamond

Polished, coarse-grain diamond films were produced by hot-filament CVD (refs. 5 and 12). They were primarily polycrystalline, with the crystallites primarily oriented along the  $\langle 111 \rangle$  direction. The grain size was approximately 10 000 nm (10  $\mu\text{m}$ ).

#### Carbon-Ion-Implanted, Fine-Grain CVD Diamond

Carbon ions were implanted into as-deposited, fine-grain diamond films with an ion implanter at 60 and 160 keV (refs. 9 and 10); the carbon ions penetrated to calculated mean depths of 88 and 198 nm, respectively. The diamond films implanted at 60 keV were used for tribological evaluation, and the diamond films implanted at 160 keV were used for microstructural characterization.

The surfaces of the resulting films contained oxygen with carbon/oxygen ratios ranging from 8 to 12, like those of the as-deposited diamond films. However, the carbon-ion-implanted, fine-grain diamond films were more conductive than the as-deposited diamond films. This increased conductivity indicates that carbon ion implantation alters the normally insulating diamond surface to an electrically conductive carbon surface and eventually to a graphitic surface. No significant changes in surface morphology and roughness resulted from the carbon ion implantation. The surface features of the carbon-ion-implanted, fine-grain diamond films were almost the same as those of the as-deposited, fine-grain diamond films. The only morphological effect of carbon ion implantation was the rounding of asperity edges. After implantation the formerly granulated, or spherulitic, surfaces of the fine-grain diamond films contained somewhat blunt, rounded asperities.

#### Nitrogen-Ion-Implanted, Coarse-Grain CVD Diamond

Nitrogen ions were implanted into a polished, coarse-grain diamond film with an ion implanter (ref. 12). The nitrogen ions penetrated to a calculated mean depth of 47 nm. No significant changes in surface morphology and roughness resulted from nitrogen ion implantation. The surface features of the nitrogen-ion-implanted diamond film were almost the same as those of the polished diamond film.

### Diamondlike Carbon

DLC films with a mean surface roughness  $R_{\text{rms}}$  of 40 nm were deposited on fine-grain CVD diamond films by the direct impact of an ion beam composed of a 3:17 mixture of argon and methane at a radiofrequency power of 99 W and ion energies of 1500 and 700 eV (refs. 6 and 11). DLC film thicknesses ranged from 520 to 660 nm. The compositions of the DLC films deposited at 1500 and 700 eV were, in atomic percent, C(59) H(36) Ar(1.8) and C(57) H(42) Ar(0.8), respectively. As indicated, the hydrogen concentration was higher in the DLC films deposited at 700 eV ( $\text{C}/\text{H} = 1.36$ ) than at 1500 eV ( $\text{C}/\text{H} = 1.64$ ).

### Cubic Boron Nitride

Cubic boron nitride films with an  $R_{\text{rms}}$  of 20 to 40 nm were deposited on silicon by magnetically enhanced plasma ion plating (ref. 7). A magnetic field parallel to the electric field was used in conjunction with a hot cathode to produce a plasma composed of a 9:1 mixture of argon and nitrogen at a radiofrequency power of 300 W. High-purity (99.9%) boron was evaporated with an electron beam.

### CVD Diamond Pin Specimens

The CVD diamond pin specimens were produced as follows: (1) a free-standing diamond film was produced by the hot-filament CVD technique (ref. 5); (2) the film was brazed onto one end of a steel pin; and (3) the CVD diamond tip of the pin was then ground with a diamond wheel and polished with diamond powder. The CVD diamond pin specimens were hemispherical, with a radius of curvature at the apex of approximately 1.6 mm.

## EXPERIMENT

Raman spectroscopy was used to characterize carbon bonding and structure. Transmission electron microscopy and electron diffraction were used to determine the microstructure and the crystalline state. Rutherford backscattering and hydrogen forward scattering were used to find the composition of the DLC films. Surface profilometry was used to determine the surface morphology, roughness, and wear of the films. Scanning electron microscopy was also used to determine surface morphology.

Rotating sliding friction experiments were performed in humid air at relative humidities to 40%, in dry nitrogen at relative humidities of less than 1%, and in ultrahigh vacuum at a vacuum pressure of  $10^{-7}$  Pa. All the experiments were conducted with the DLC films and the CVD diamond films in contact with the diamond pins under a load of 0.98 N (mean Hertzian contact pressure, approximately 2 GPa), at a constant rotating speed of 120 rpm (sliding velocity from 31 to 107 mm/s because of the range of wear track radii involved in the experiments), and at room temperature. The friction apparatus used in the investigation was mounted in a vacuum chamber. The apparatus can measure friction in humid air, in dry nitrogen, and in ultrahigh vacuum. The steady-state coefficient of friction and the wear rate are the average values obtained from two to four experiments at each sliding friction condition.

## RESULTS AND DISCUSSION

### Friction and Wear Properties of CVD Diamond and DLC Films

#### Coefficients of Friction and Wear Rates

*CVD diamond films.*—In humid air and in dry nitrogen (fig. 1(a)) as-deposited, fine-grain diamond films; polished, coarse-grain diamond films; and carbon- and nitrogen-ion-implanted diamond films had low coefficients of friction ( $<0.1$ ) and low wear rates ( $\leq 10^{-6}$  mm<sup>3</sup>/N·m). In this respect, the ion-implanted CVD diamond was similar to the as-deposited, fine-grain or polished, coarse-grain CVD diamond.

In ultrahigh vacuum (fig. 1(b)) both as-deposited, fine-grain diamond films and polished, coarse-grain diamond films had high coefficients of friction ( $>0.4$ ) and high wear rates ( $\leq 10^{-4}$  mm<sup>3</sup>/N·m), making them unacceptable for tribological applications. In ultrahigh vacuum the effect of carbon and nitrogen ion

implantation was significant: these films had low coefficients of friction ( $\leq 0.1$ ) and low wear rates ( $10^{-6}$  mm<sup>3</sup>/N·m), making them acceptable for tribological applications.

**Diamondlike carbon films.**—In dry nitrogen and in humid air the DLC films ion beam deposited on fine-grain CVD diamond had low steady-state coefficients of friction and low wear rates (fig. 2). In ultrahigh vacuum the ion-beam-deposited DLC films (like the ion-implanted CVD diamond films) also had low coefficients of friction and low wear rates (fig. 2) and provided adequate solid lubrication. Such enhanced tribological performance, coupled with a wider range of coating thicknesses, means that these DLC films would have longer endurance life and better wear resistance than ion-implanted diamond films. Thus, DLC films can be an effective wear-resistant, lubricating coating regardless of the environment.

In this investigation the main criteria for judging the performance of these hard carbon-based films were that the coefficient of friction and wear rate had to be less than 0.1 and  $10^{-6}$  mm<sup>3</sup>/N·m, respectively. Carbon- and nitrogen-ion-implanted, fine-grain CVD diamond films and ion-beam-deposited DLC films met the criteria regardless of environment. Why did these films meet the criteria? Let us investigate their characteristics and seek mechanisms for their low friction behavior.

### Characteristics of Ion-Implanted Diamond Films

**Bonding characteristics.**—The Raman spectrum of an as-deposited, fine-grain diamond film (fig. 3(a)) reveals three bands characteristic of CVD diamond films: (1) a sharp band centered near 1332 cm<sup>-1</sup>, (2) a broad band centered between 1500 and 1530 cm<sup>-1</sup>, and (3) an even broader band centered near 1320 cm<sup>-1</sup>. The sharp band centered near 1332 cm<sup>-1</sup> is characteristic of diamond's sp<sup>3</sup> bonding. The two broad Raman shift bands near 1320 cm<sup>-1</sup> and between 1500 and 1530 cm<sup>-1</sup> are characteristic of the nondiamond form of carbon. They are called the D band and the G band, respectively. The G-band Raman shifts are attributed to the sp<sup>2</sup>-bonded carbon, whereas the D-band Raman shifts are attributed to the disorder of the nondiamond carbon present in the diamond films (ref. 1). The as-deposited, fine-grain diamond films contained a considerable amount of nondiamond carbon.

The Raman spectrum of a polished diamond film (fig. 3(b)) reveals three bands: (1) a sharp band centered near 1332 cm<sup>-1</sup> (the sp<sup>3</sup> bonding of diamond), (2) a broad band centered between 1500 and 1530 cm<sup>-1</sup> (the sp<sup>2</sup> bonding of carbon), and (3) an even broader band centered near 1320 cm<sup>-1</sup> (the disorder of the nondiamond carbon).

The Raman spectrum of a carbon-ion-implanted, fine-grain diamond film (fig. 3(c)) reveals a very broad band with a peak centered between 1500 and 1530 cm<sup>-1</sup> and a shoulder near 1320 cm<sup>-1</sup>, indicative of the amorphous, nondiamond form of carbon. The characteristic diamond peak is absent.

The Raman spectrum of a nitrogen-ion-implanted diamond film (fig. 3(d)) reveals a very broad band with a peak centered between 1500 and 1530 cm<sup>-1</sup> and a shoulder near 1320 cm<sup>-1</sup>, indicative of the amorphous, nondiamond form of carbon. The characteristic diamond peak is absent.

**Microstructural characteristics.**—Transmission electron microscopy of cross sections of diamond films implanted by carbon ions at 160 keV revealed a layered structure containing an amorphous layer formed on the crystalline diamond layer (ref. 13). Figure 4 represents a typical selected area diffraction (SAD) pattern of the as-deposited, free-standing diamond film. Diffraction rings observed in the SAD pattern suggested the presence of randomly oriented small crystallites. The  $d$  spacings of the observed diffraction rings were evaluated, using the standard aluminum SAD pattern as a calibration standard, and matched well with the known diamond  $d$  spacings. Note that the  $d$  values of the observed diffraction rings can also be matched with those of highly oriented graphite (refs. 14 and 15). However, the Raman spectrum of the highly oriented graphite differed from that of diamond. The Raman spectrum of the CVD diamond film clearly showed a characteristic diamond peak at 1333 cm<sup>-1</sup>.

Figure 5(a) shows a cross-sectional, bright-field transmission electron micrograph of a diamond film ion implanted with carbon ions at 160 keV at a dose of  $6.7 \times 10^{17}$  ions/cm<sup>2</sup> (TRIM-88 calculated penetration range, 198 nm). Carbon ion implantation produced an amorphous surface layer 500 to 600 nm thick. The amorphous nature of the surface layer was confirmed by the SAD pattern. Note in figure 5(a) that the silicon substrate was sputtered away during ion milling. Figure 5(b) represents the SAD pattern of both the ion-implanted and unimplanted parts of the diamond film. The diffuse ring in the SAD pattern shows the amorphous nature of the implanted layer; the spotty diffraction rings are due to the randomly oriented crystallites of the underlying unimplanted part of the diamond film.

Thus, Raman and TEM analyses of ion-implanted diamond films revealed that amorphous, nondiamond carbon replaced diamond carbon as the top layer.

## Characteristics of Ion-Beam-Deposited DLC Films

Figure 6 presents Raman spectra of DLC films deposited on fine-grain CVD diamond at ion energies of 1500 and 700 eV. The Raman spectra indicate the presence of amorphous, nondiamond carbon. The characteristic diamond peak is absent. These spectra show that the disorder of the nondiamond carbon was more prevalent in the DLC film deposited at 1500 eV than in that deposited at 700 eV. These Raman spectra are similar to those for DLC films deposited on silicon substrates (ref. 11).

## Lubrication Mechanism of Ion-Implanted CVD Diamond and DLC Films

Bombarding diamond films with carbon ions at 60 keV or with nitrogen ions at 35 keV produced a thin, superficial layer of amorphous nondiamond carbon ( $<0.1\text{ }\mu\text{m}$  thick). The ion-beam-deposited DLC films also contained predominantly amorphous, nondiamond carbon (hydrogenated carbon). The presence of amorphous nondiamond in both ion-implanted diamond and DLC films greatly decreased their friction and wear in ultrahigh vacuum, without sacrificing their low friction and low wear properties in dry nitrogen and in humid air.

According to the Bowden-Tabor theory of metallic friction (refs. 16 and 17) or the relation between friction and the total surface energy in the real area of contact (ref. 18), reducing friction requires minimizing the shear strength of the interface, the total surface energy in the real area of contact, the area of contact, and the plowing contribution. Figure 7 illustrates how these minimizations can be achieved. Using a hard substrate reduces both the area of contact and the plowing; using an amorphous nondiamond carbon surface layer reduces the shear strength and surface energy in the real area of contact. In other words, the low friction of the ion-implanted diamond and ion-beam-deposited DLC films can be attributed to the combination of the low shear strength and low surface energy of the thin, amorphous nondiamond carbon surface layer and the small contact area resulting from the high elastic modulus and hardness of the underlying diamond film.

## Friction and Wear Properties of c-BN Films

Figure 8 presents average coefficients of friction in humid air, in dry nitrogen, and in ultrahigh vacuum for as-deposited c-BN films in sliding contact with CVD diamond pins as a function of number of passes. The friction data indicate that the steady-state coefficients of friction were generally low in the three environments and in the ascending order of ultrahigh vacuum, dry nitrogen, and humid air.

In ultrahigh vacuum the sliding action caused the c-BN film to break down, whereupon the coefficient of friction rapidly increased at approximately 1400 passes (fig. 8). The endurance life of c-BN film in ultrahigh vacuum was unstable between 50 and 1500 passes (i.e., the wear rate varied from  $10^{-4}$  to  $10^{-6}\text{ mm}^3/\text{N}\cdot\text{m}$ ). On the other hand, in humid air and in dry nitrogen the coefficient of friction remained constant for a long period without film breakdown even at 100 000 passes. The endurance life of c-BN films was 60 to 2000 times greater in dry nitrogen and in humid air than in ultrahigh vacuum.

## CONCLUDING REMARKS

In this investigation the main criteria for judging the performance of hard carbon-based films were coefficient of friction and wear rate, which had to be less than 0.1 and  $10^{-6}\text{ mm}^3/\text{N}\cdot\text{m}$ , respectively. The following films met the requirements regardless of environment:

1. Carbon- and nitrogen-ion-implanted, fine-grain CVD diamond
2. DLC ion beam deposited on fine-grain CVD diamond

Ion implantation produces a graded interface and is easily controlled by adjusting the operating variables of the accelerator, such as accelerating energy, current density, and time. A disadvantage of ion implantation technology is the shallow penetration depth of the implanted species (layer thickness,  $0.01$  to  $0.5\text{ }\mu\text{m}$ ) relative to conventional coatings. This shallow penetration depth may limit the tribological applications of ion implantation to light loads or short-term operations. In other words, the endurance life (wear life) of the ion-implanted layer, which contributes to tribological benefits, is limited.

Ion beam deposition produces uniform DLC films, a few micrometers thick, on large areas and curved surfaces. This greater range of DLC coating thicknesses, coupled with low friction and wear regardless of

environment, led to longer endurance life and improved wear resistance relative to the ion-implanted CVD diamond films. For example, DLC films with a coating thickness of 0.5  $\mu\text{m}$  on CVD diamond had approximately 20 times the endurance life of carbon-ion-implanted, fine-grain CVD diamond with an amorphous, nondiamond 0.05- $\mu\text{m}$ -thick carbon layer.

Lastly, c-BN in contact with CVD diamond exhibited a capability for lubrication (low adhesion and friction) regardless of environment, but especially in ultrahigh vacuum.

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Air	Nitrogen	Vacuum	Film	Pin
○	●	●	As deposited	Natural diamond
△	▲	▲	Polished	CVD diamond
□	■	■	Carbon ion implanted	Natural diamond
◇	◆	◆	Nitrogen ion implanted	CVD diamond

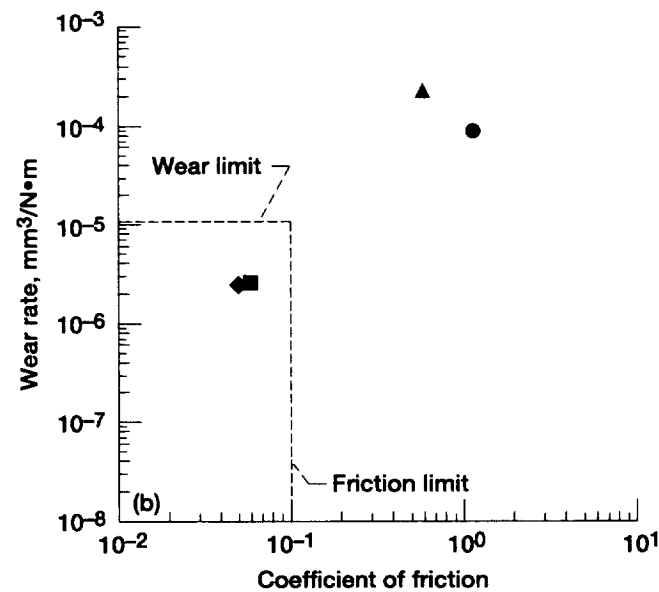
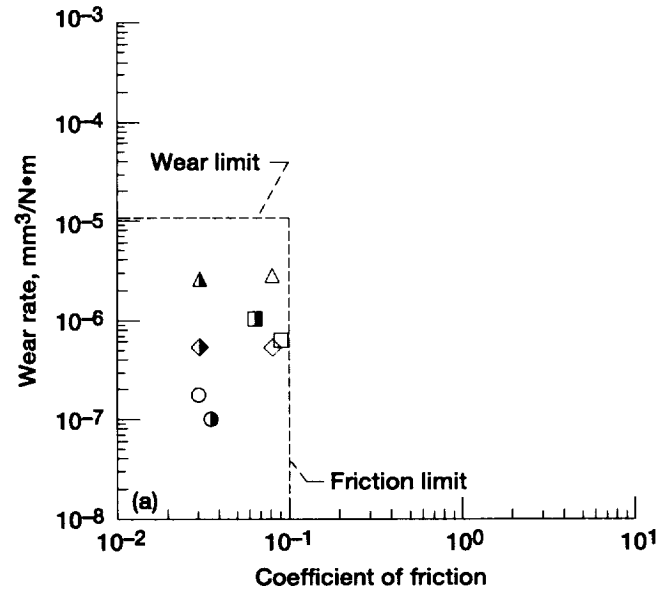


Figure 1.—Relationship between wear rate and coefficient of friction for chemical-vapor-deposited (CVD) diamond films. (a) Humid air and dry nitrogen environments. (b) Ultra-high-vacuum environment.

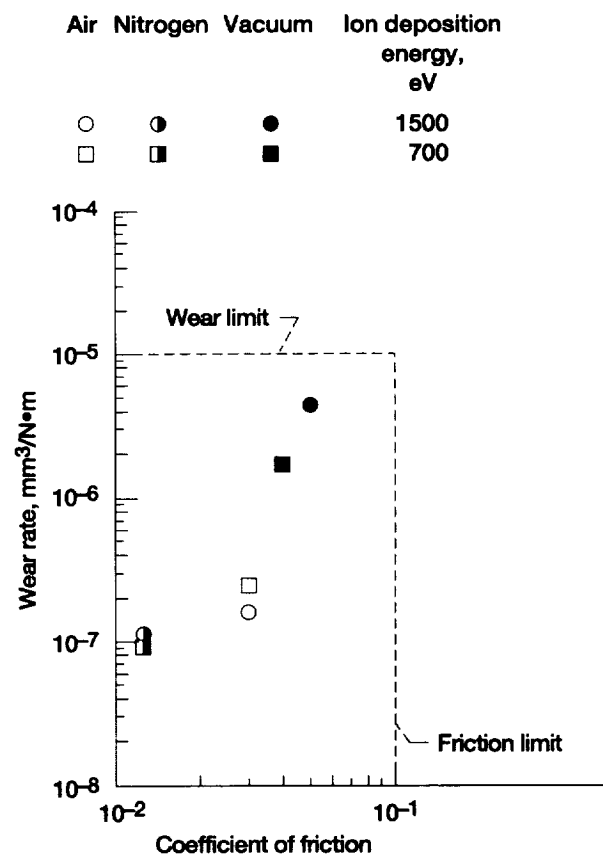


Figure 2.—Coefficients of friction and wear rates for diamondlike carbon (DLC) films deposited on fine-grain diamond at 1500 and 700 eV.

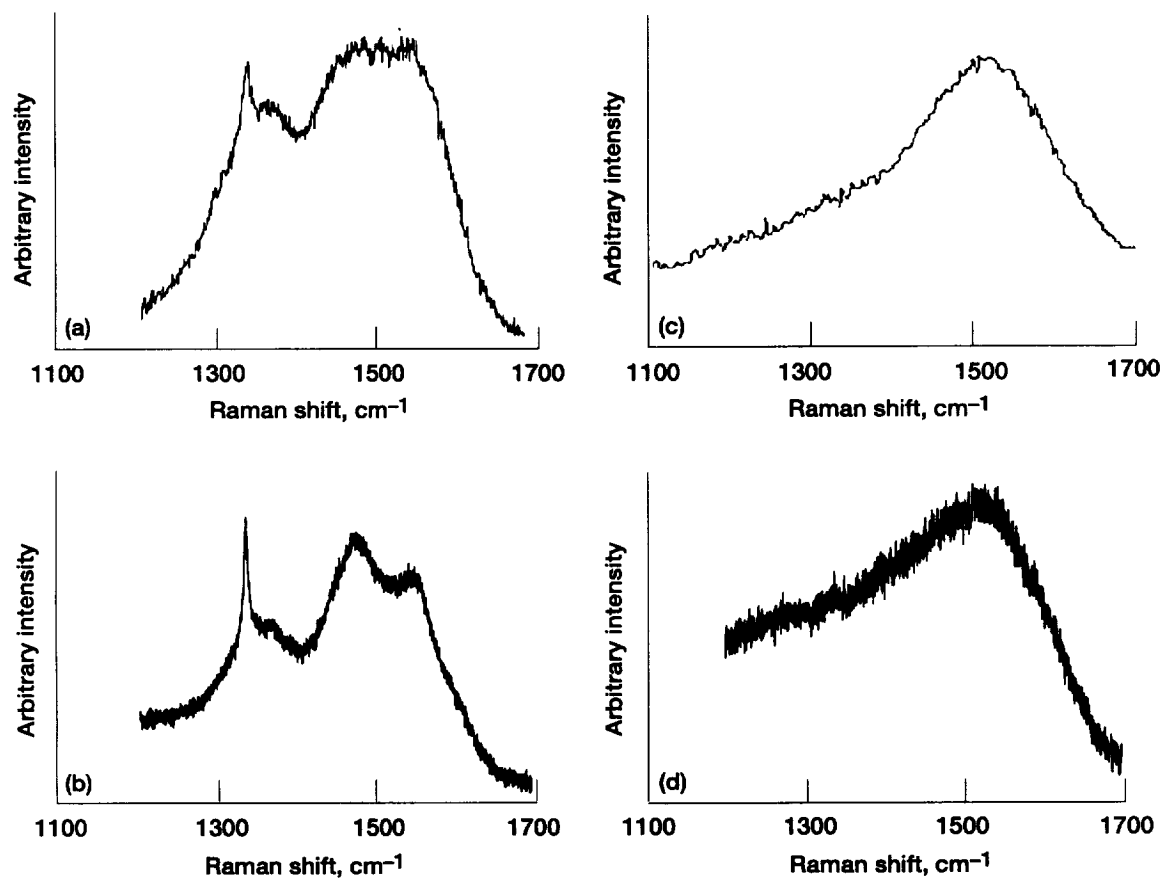


Figure 3.—Raman spectra of various CVD diamond films. (a) As deposited, fine grain (20 to 100 nm). (b) Polished, coarse grain (10  $\mu\text{m}$ ). (c) Carbon ion implanted, fine grain. (d) Nitrogen ion implanted, coarse grain.

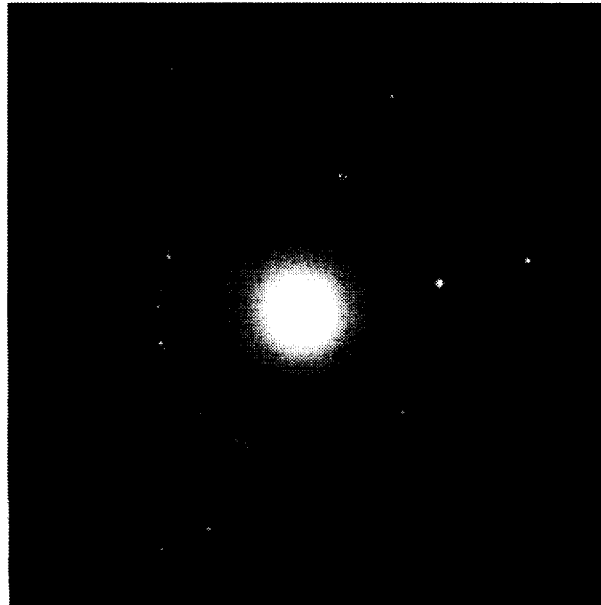


Figure 4.—Typical selected area diffraction pattern of as-deposited, free-standing diamond film.

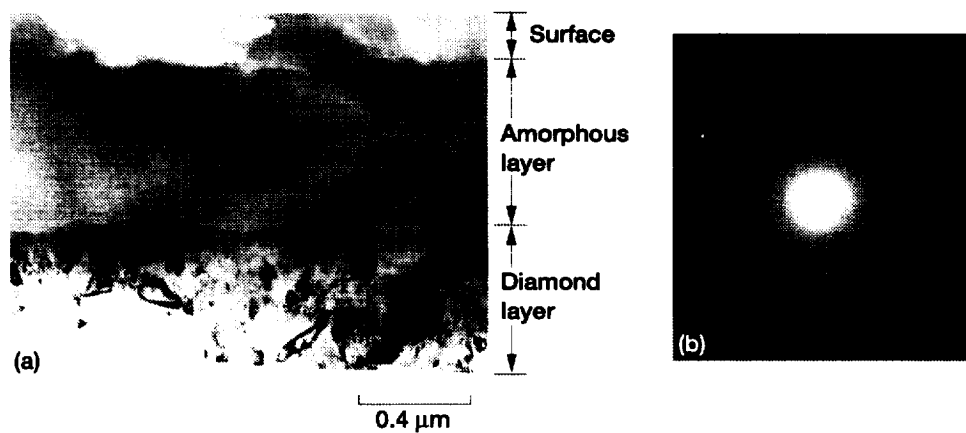


Figure 5.—Carbon-ion-implanted, fine-grain diamond film. Carbon ions at 160 keV at a dose of  $6.7 \times 10^{17}$  ions/cm<sup>2</sup>. (a) A cross-sectional TEM bright-field image. (b) Selected area diffraction pattern of both ion-implanted and unimplanted fine-grain diamond film.

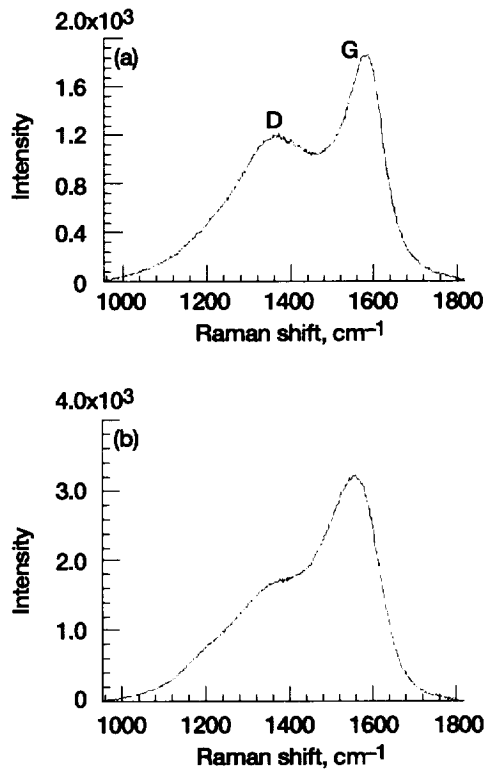


Figure 6.—Raman spectra of DLC films ion beam deposited on fine-grain diamond. (a) Ion energy, 1500 eV. (b) Ion energy, 700 eV.

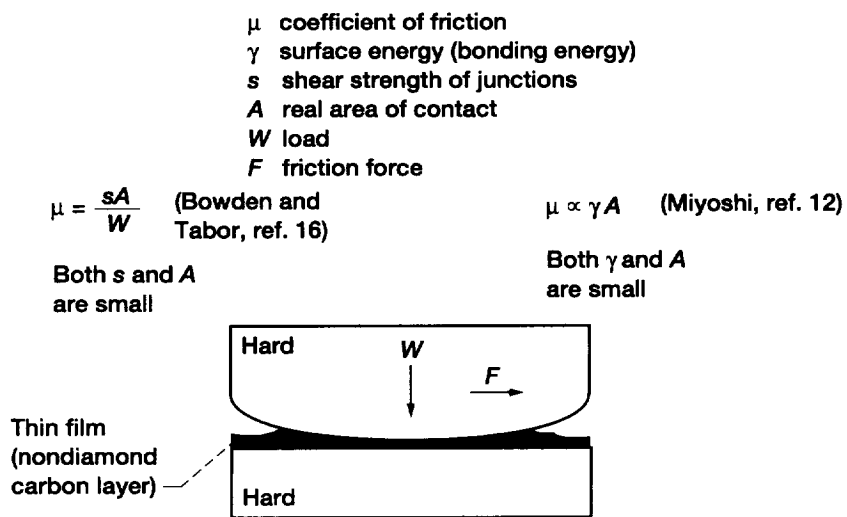


Figure 7.—Lubrication mechanisms.

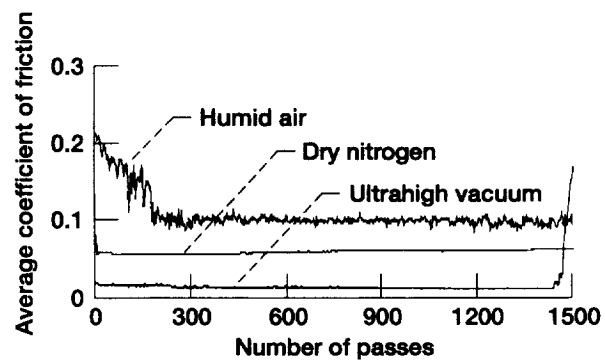


Figure 8.—Coefficients of friction for c-BN films in sliding contact with CVD diamond pins in humid air, dry nitrogen, and ultra-high-vacuum environments.



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