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Extended Abstract

INTRODUCTION

Diamond's outstanding properties, extreme hardness, chemical and thermal inertness, and high strength and rigidity, make it an ideal material for a tribological part. For example, as is well known, diamond is one of the slipperiest materials, having a coefficient of friction in air similar to that of polytetrafluoroethylene.

Modern diamonds belong to one of four distinct categories: natural, high-pressure synthetic, chemically vapor deposited (CVD), and diamondlike carbon (DLC). The tribological applications of natural and high-pressure synthetic diamonds are limited because of their small size and high cost. Also, these crystals have to be bonded to a substrate in a separate operation. This requirement, coupled with their high cost, limits the general use of diamonds in tribological applications. On the other hand, CVD diamond and DLC have a greater potential because size is, and eventually cost will be, less a limitation. CVD diamond and DLC, available in relatively large-area, planar films or sheets, alter the surface properties of a substrate, thus opening the door to tribology technology that can take full advantage of diamond's intrinsic properties. For example, its low coefficient of friction (<0.1) in air makes it suitable for applications such as wear, solid lubrication, erosion, and corrosion.

The major disadvantage of CVD diamond is that its high deposition temperature restricts its substrate materials in tribological coatings. Although DLC is processed without the high-temperature substrate requirements, its three disadvantages are low deposition rate and high internal stress, and, unlike diamond, it cannot be obtained as thick monolithic shapes, at least not with the present technology.

For the tribological use of CVD diamond and DLC, we must have a good understanding of these coatings, the counterfacing material, and the type of environment and operation. Several characterization techniques are available for studying bulk and surface conditions of the materials. These techniques provide different types of information, and in due course, it should be possible to coordinate them and provide a coherent uniform description of the bulk and surface.

This paper deals with the ways in which physical characterization techniques can be usefully applied to study the friction and wear of CVD diamond and DLC coatings. The emphasis is to relate friction and wear to reactions at the sliding interface and to the composition of the interface. The physical and tribological characteristics of as-deposited, fine- and coarse-grain CVD diamond films and as-deposited DLC films are reviewed. Some earlier data and experimental details on this study are given in references 1 to 4.

EXPERIMENT

The diamond films were produced on the flat surfaces of silicon (Si), silicon carbide (SiC), and silicon nitride (Si₃N₄) substrates using 99.999 percent hydrogen (H₂), 99.97 percent methane (CH₄), and 99.6 percent oxygen (O₂) in the microwave-source plasma deposition system. The DLC films were formed on the flat surfaces of Si substrates using CH₄ in the direct ion beam deposition system with a Kaufman ion source.

Several analytical techniques were used to characterize the as-deposited diamond and DLC films: scanning and transmission electron microscopy (SEM and TEM) to determine surface morphology and grain size measurements; Rutherford backscattering spectroscopy (RBS) to identify impurities in the films and to determine carbon and impurity concentrations; Raman spectroscopy and Fourier transform infrared spectroscopy (FTIR) to characterize diamond quality and structure; proton recoil detection (PRD) to measure the hydrogen concentration; x-ray photoelectron spectroscopy (XPS) to characterize surface chemistry; and x-ray diffraction to determine the crystal orientation of the films.

Reciprocating sliding friction experiments were performed in humid air at a relative humidity of 40 percent and in dry nitrogen at a relative humidity of less than 1 percent. Rotating sliding friction experiments were conducted in ultrahigh vacuum at a pressure of 10^{-7} Pa. Experiments were conducted with the diamond films in contact with a natural bulk diamond pin (1.3-mm radius) in the three environments. Also, experiments were conducted with the DLC films in contact with Si_3N_4 pins in the three environments.

RESULTS AND DISCUSSION

Diamond Films

The grain size and surface roughness of the as-deposited diamond films ranged from 20 to 3300 nm and from 15 to 160 nm root-mean-square (rms), respectively. The surface roughness increased as the grain size increased. The morphologies of the as-deposited diamond films are two types: smooth fine-grain and rough coarse-grain. The grain size and surface roughness of the as-deposited, fine-grain diamond films ranged from 20 to 100 nm and from 6 to 37 nm rms, respectively. The grain size and surface roughness of the as-deposited, coarse-grain diamond films ranged from 1000 to 3300 nm and from 48 to 160 nm rms, respectively.

The hydrogen concentration was estimated to be 2.5 at.% in the as-deposited, fine-grain diamond films and less than 1 at.% in the as-deposited, coarse-grain diamond films. The as-deposited, fine-grain diamond films contained considerably more nondiamond carbon than the as-deposited, coarse-grain diamond films.

Most of the crystallites in the as-deposited, fine-grain diamond films were oriented along (110) planes whereas those of the as-deposited, coarse-grain diamond films were oriented along (111) planes.

We found that the tribological characteristics of diamond films, similar to those of monolithic diamond, varied with the environment (e.g., fig. 1). As-deposited diamond films indeed have friction and wear properties similar to those of natural diamond in the three environments. In humid air and in dry nitrogen, the surface roughness of as-deposited diamond films can appreciably influence initial friction: the greater the initial surface roughness, the higher the initial coefficient of friction. When repeated sliding produced a smooth groove or a groove with blunted asperities on the rough surface of the coarse-grain diamond films, the coefficient of friction was low and the initial surface roughness effect became negligible. In humid air and in dry nitrogen, the as-deposited, fine-grain diamond film can be effectively used as a wear-resistant, self-lubricating coating having low coefficients of friction (0.02 to 0.04) and a low wear rate (10^{-4} $\text{mm}^3/\text{N}\cdot\text{m}$).

In ultrahigh vacuum, the as-deposited, fine- and coarse-grain diamond films have high coefficients of friction (1.5 to 1.7) and a high wear rate (10^{-4} $\text{mm}^3/\text{N}\cdot\text{m}$), which are not acceptable for tribological applications. In ultrahigh vacuum, removing the contaminant surface layer from the contact area of diamond films results in a stronger interfacial adhesion between the diamond pin and diamond films and raises the coefficient of friction. The increase in friction was attributed to removing the absorbed contaminants from the surface by rubbing or sliding in ultrahigh vacuum at room temperature.

The wear mechanism of diamond films similar to monolithic diamond is that of small fragments chipping off the surface. The size of wear particles is related to the extent of wear rates. The larger the wear particle size, the higher the wear rate.

Diamondlike Carbon Films

The as-deposited DLC films were amorphous and pin-hole free with uniform and smooth surfaces; the density was 1.7 ± 0.2 g/cm^3 and the surface roughness ranged from 10 to 18 nm rms. The DLC films contain 30 at.% hydrogen and 70 at.% carbon.

The Raman spectrum of the DLC film consists primarily of two very broad overlapping bands, the D- (disorder) band at 1340 cm^{-1} and the G- (graphite) band at 1539 cm^{-1} , designated as such because of their similarity to the first-order Raman spectrum of disordered sp^2 bonded carbon and polycrystalline graphite.

We found that the friction and wear characteristics of DLC films are similar to those of fine-grain diamond in humid air and in dry nitrogen, but they are different from those of fine-grain diamond in ultrahigh vacuum (fig. 1). In humid air and in dry nitrogen, the as-deposited DLC films have low coefficients of friction (0.02 to 0.16) and a low wear rate (10^{-4} $\text{mm}^3/\text{N}\cdot\text{m}$). In ultrahigh vacuum, the as-deposited DLC films have a low

coefficient of friction (<0.1) but have a high wear rate (10^{-4} $\text{mm}^3/\text{N}\cdot\text{m}$), which is not acceptable for tribological applications.

CONCLUSIONS

Both as-deposited, fine-grain diamond films and DLC films can be effective wear-resistant, self-lubricating coatings in humid air and in dry nitrogen, but they are not effective in ultrahigh vacuum. For the use of diamond and diamondlike carbon films in ultrahigh vacuum, film modifications (e.g., ion implantation) that provide acceptable levels of friction and wear properties are necessary.

REFERENCES

1. Wu, R.L.C., et al.: Synthesis and Characterization of Fine Grain Diamond Films. *J. Appl. Phys.*, vol. 72, no. 1, July 1992, pp. 110–116.
2. Miyoshi, K., et al.: Friction and Wear of Plasma-Deposited Diamond Films. *J. Appl. Phys.*, vol. 74, no. 7, Oct. 1993, pp. 4446–4454.
3. Wu, R.L.C., et al.: Physical and Tribological Properties of Rapid Thermal Annealed Diamond-Like Carbon Films. *Surf. Coat. Technol.*, vol. 55, 1992, pp. 576–580.
4. Miyoshi, K., et al.: Friction and Wear of Diamond and Diamond-Like Carbon Coatings. *Surf. Coat. Technol.*, vol. 55, 1992, pp. 428–434.

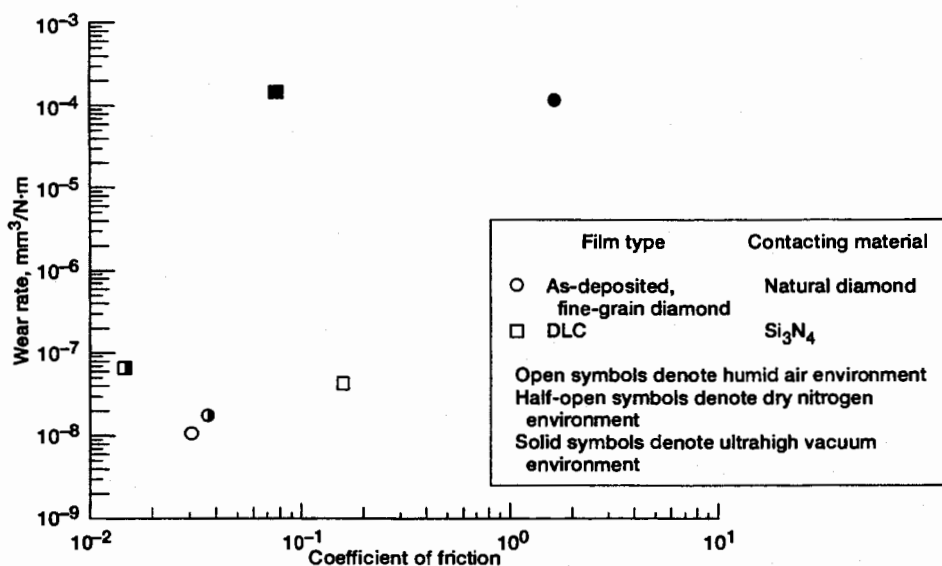


Figure 1.—Coefficient of friction and wear rate for fine-grain diamond films and diamondlike carbon films.