

1N-27

NASA Technical Memorandum 107316

# Friction and Wear of Ion-Beam-Deposited Diamondlike Carbon on Chemical-Vapor- Deposited, Fine-Grain Diamond

Kazuhisa Miyoshi  
*Lewis Research Center*  
*Cleveland, Ohio*

Richard L.C. Wu and William C. Lanter  
*K Systems Corporation*  
*Dayton, Ohio*

October 1996



National Aeronautics and  
Space Administration



# FRICITION AND WEAR OF ION-BEAM-DEPOSITED DIAMONDLIKE CARBON ON CHEMICAL-VAPOR-DEPOSITED FINE-GRAIN DIAMOND

Kazuhisa Miyoshi  
National Aeronautics and Space Administration  
Lewis Research Center  
21000 Brookpark Road, Mail Stop 23-2  
Cleveland, Ohio 44135, U.S.A.

and

Richard L.C. Wu and William C. Lanter  
K Systems Corporation  
1522 Marsetta Drive  
Dayton, Ohio 45432, U.S.A.

## SUMMARY

Friction and wear behavior of ion-beam-deposited diamondlike carbon (DLC) films coated on chemical-vapor-deposited (CVD), fine-grain diamond coatings were examined in ultrahigh vacuum, dry nitrogen, and humid air environments. The DLC films were produced by the direct impact of an ion beam (composed of a 3:17 mixture of Ar and CH<sub>4</sub>) at ion energies of 1500 and 700 eV. Sliding friction experiments were conducted with hemispherical CVD diamond pins sliding on four different carbon-base coating systems: DLC films on CVD diamond; DLC films on silicon; as-deposited, fine-grain CVD diamond; and carbon-ion-implanted, fine-grain CVD diamond on silicon. Results indicate that in ultrahigh vacuum the ion-beam-deposited DLC films on fine-grain CVD diamond (similar to the ion-implanted CVD diamond) greatly decrease both the friction and wear of fine-grain CVD diamond films and provide solid lubrication. In dry nitrogen and in humid air, ion-beam-deposited DLC films on fine-grain CVD diamond films also had a low steady-state coefficient of friction and a low wear rate. These tribological performance benefits, coupled with a wider range of coating thicknesses, led to longer endurance life and improved wear resistance for the DLC deposited on fine-grain CVD diamond in comparison to the ion-implanted diamond films. Thus, DLC deposited on fine-grain CVD diamond films can be an effective wear-resistant, lubricating coating regardless of environment.

## INTRODUCTION

In an ultrahigh vacuum, a spacelike environment, both the coefficients of friction (0.4 to 2.0) and the wear rate ( $10^{-4}$  mm<sup>3</sup>/Nm) of chemical-vapor-deposited (CVD) diamond films are considerably higher than in air or dry nitrogen. It is clear that surfaces will have to be modified to provide acceptable levels of friction and wear properties before CVD diamond films can be used in vacuum for tribological applications such as vacuum wear parts in the semiconductor industry or tribocomponents in spacecraft (ref. 1).

Modifying the surface of CVD diamond films by carbon or nitrogen ion implantation produces a thin ( $\leq 0.5$ - $\mu$ m-thick) layer of amorphous, nondiamond carbon in the near surface region, which has low shear strength (refs. 2 to 4). The presence of an amorphous, nondiamond carbon layer on CVD diamond films decreases both friction and wear in ultrahigh vacuum, resulting in a low steady-state coefficient of friction ( $< 0.1$ ) and a low wear rate ( $\leq 10^{-6}$  mm<sup>3</sup>/Nm), respectively. Ion implantation provides acceptable levels of friction and wear for CVD diamond films regardless of the environment in which they are examined (humid air, dry nitrogen, or ultrahigh vacuum).

It is known that ion implantation does not create an interface of demarcation between the host material and implanted species (ref. 5). Instead, it produces a graded interface. The ion implantation process is easily controlled by adjusting the operating variables of the accelerator, such as the accelerating energy, current density, and time. One of the disadvantages of ion implantation technology is that the depth of penetration of the implanted species is

very shallow (thickness range of ion-implanted layers, 0.01 to 0.5  $\mu\text{m}$ ) compared with that of conventional coatings, which 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.

Since the thickness range of diamondlike carbon (DLC) films can be 0.1 to 5  $\mu\text{m}$ , which is one order of magnitude greater than that of the ion-implanted layer, the wear (endurance) life of DLC can be longer than that of the ion-implanted layer. Therefore, studying an amorphous DLC film coated on a fine-grain CVD diamond film is a logical approach to enhancing tribological properties, in particular to increasing the endurance of CVD diamond films.

This investigation examined the friction and wear behavior of ion-beam-deposited DLC films coated on fine-grain CVD diamond in sliding contact with CVD diamond pins in three environments: ultrahigh vacuum, dry nitrogen, and humid air. For a comparison of the friction and wear properties of the ion-beam-deposited DLC films on fine-grain CVD diamond, reference experiments also were conducted with three types of carbon-base films: ion-beam-deposited DLC films on silicon; as-deposited, fine-grain CVD diamond films on silicon; and carbon-ion-implanted, fine-grain CVD diamond films on silicon.

## MATERIALS

DLC films with a mean surface roughness ( $R_{\text{rms}}$ ) of 40 nm were deposited on fine-grain CVD diamond, and films with a mean  $R_{\text{rms}}$  of 22 nm were deposited on silicon by the direct impact of an ion beam composed of a 3:17 mixture of Ar and  $\text{CH}_4$  at an RF power of 99 W and ion energies of 1500 and 700 eV (ref. 6). DLC film thicknesses ranged from 520 to 660 nm.

As-deposited, fine-grain CVD diamond films with a mean  $R_{\text{rms}}$  of 31 nm on silicon were produced by microwave-plasma-assisted CVD (ref. 7). Carbon-ion-implanted, fine-grain CVD diamond films with a mean  $R_{\text{rms}}$  of 14 nm were produced by impacting carbon ions into the as-deposited, fine-grain CVD diamond films at an accelerating energy of 60 keV and a current density of 50  $\mu\text{A}/\text{cm}^2$  for approximately 6 min, resulting in a dose of  $1.2 \times 10^{17}$  carbon ions/ $\text{cm}^2$  that formed a ballistic layer less than 0.1- $\mu\text{m}$  thick (refs. 2 and 3).

The CVD diamond pin specimens were produced as follows: (1) a free-standing diamond film was produced via the hot-filament CVD technique (ref. 8); (2) the CVD diamond film was brazed on 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, and Rutherford backscattering (RBS) and hydrogen forward scattering were used to determine the compositions of the DLC films. Surface profilometry was used to determine the surface morphology, roughness, and wear of the films. Scanning electron microscopy was used to determine surface morphology.

Rotating sliding friction experiments were performed in humid air at a relative humidity to 40 percent, in dry nitrogen at a relative humidity less than 1 percent, and in ultrahigh vacuum at a vacuum pressure of  $10^{-7}$  Pa. All experiments were conducted with the DLC films and CVD diamond films in contact with the CVD diamond pins (radius, 1.6 mm) with a load of 0.49 N (mean Hertzian contact pressure, around 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.

## RESULTS AND DISCUSSION

Figures 1(a) to (c) present Raman spectra of the as-deposited, fine-grain CVD diamond films and the DLC films deposited on fine-grain CVD diamond at ion energies of 1500 and 700 eV. The fine-grain CVD diamond films contained the diamond form of carbon at  $1332 \text{ cm}^{-1}$  as well as the G- and D-band nondiamond forms of carbon (ref. 9). These results are consistent with those from the Raman analysis conducted in the previous study (ref. 7). Raman spectra of the DLC films on fine-grain CVD diamond indicate the presence of amorphous, nondiamond carbon. The

characteristic diamond peak is absent from the micro-Raman spectra of the ion-beam-deposited DLC films. These spectra showed that the disorder of the nondiamond carbon is more prevalent in the DLC film deposited at 1500 eV than at 700 eV. Raman spectra of the DLC films deposited on silicon substrates are similar to those shown in figures 1(b) and (c) for the DLC deposited on fine-grain CVD diamond substrates.

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, there is a higher concentration of hydrogen in the DLC film deposited at 700 eV ( $C/H = 1.36$ ) than at 1500 eV ( $C/H = 1.64$ ).

Figures 2 (a) to (f) show typical friction traces in the three environments examined (ultrahigh vacuum, dry nitrogen, and humid air) for DLC films deposited on fine-grain CVD diamond films and for as-deposited, fine-grain CVD diamond films. The traces show closely spaced irregularities. In general, the heights of the irregularities in the friction traces investigated strongly depended on environment. Heights were lowest for dry nitrogen, increased for humid air, and were the highest for ultrahigh vacuum.

In ultrahigh vacuum, the mean coefficients of friction obtained for the DLC films on CVD diamond were less than 0.1—values which were lower than those obtained for the as-deposited, fine-grain CVD diamond films by a factor of 5 or greater. Both in dry nitrogen and in humid air, the mean coefficients of friction for the DLC films were slightly lower than those for the as-deposited, fine-grain CVD diamond films.

Figures 3(a) and (b) present the steady-state (equilibrium) coefficients of friction and wear rates in ultrahigh vacuum, dry nitrogen, and humid air for DLC films deposited on fine-grain CVD diamond and on silicon at ion energies of 1500 and 700 eV. Both the steady-state coefficients of friction and the wear rates of the DLC films depended on the environment. Values were lowest for dry nitrogen, increased for humid air, and were the highest for ultrahigh vacuum.

The ion energy influenced the wear rate of DLC films in ultrahigh vacuum: for DLC films deposited at 1500 eV on fine-grain CVD diamond or on silicon, wear rates were 2.6 times greater than for those deposited at 700 eV. This may be attributable to the higher hydrogen concentration in the films deposited at 700 eV than at 1500 eV. On the other hand, the ion energy had little influence on the coefficients of friction and wear rates of DLC films in dry nitrogen and in humid air. The coefficient of friction and wear rate of DLC films deposited on fine-grain CVD diamond or silicon at 1500 eV were similar to those for films deposited at 700 eV.

Figure 4 presents the steady-state (equilibrium) coefficients of friction and wear rates obtained in ultrahigh vacuum, dry nitrogen, and humid air for DLC films deposited on fine-grain CVD diamond at ion energies of 1500 and 700 eV; for as-deposited, fine-grain CVD diamond; and for carbon-ion-implanted, fine-grain CVD diamond. The data indicate a marked difference in friction and wear due to the combination of materials and environmental conditions.

In ultrahigh vacuum, both the steady-state coefficient of friction and the wear rate of the DLC films were relatively low and were similar to those of the carbon-ion-implanted CVD diamond films. In contrast, the bare, as-deposited, fine-grain CVD diamond films had a higher coefficient of friction and a greater wear rate. Thus, the DLC films deposited on fine-grain CVD diamond films, like the carbon-ion-implanted, fine-grain CVD diamond, provide solid-lubrication for CVD diamond films in ultrahigh vacuum. In humid air and in dry nitrogen, both the steady-state coefficient of friction and the wear rate of the as-deposited DLC films were slightly lower than those of the as-deposited, fine-grain CVD diamond films and the carbon-ion-implanted, fine-grain CVD diamond films.

Ion beam deposition of diamondlike carbon (DLC) films on chemical-vapor-deposited (CVD) diamond films significantly decreased sliding friction and wear rate in ultrahigh vacuum. It also reduced the coefficients of friction and wear rates in dry nitrogen and in humid air. Furthermore, ion beam deposition made it possible to produce uniform DLC films, a few micrometers thick, on large areas and curved surfaces (ref. 6). 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 than for the carbon-ion-implanted CVD diamond films. For example, the endurance life of DLC films with a coating thickness of 0.5  $\mu\text{m}$  on CVD diamond was approximately 20 times greater than that of carbon-ion-implanted, fine-grain CVD diamond with an amorphous, nondiamond 0.05- $\mu\text{m}$ -thick carbon layer.

## CONCLUSIONS

The presence of a DLC film produced by direct ion beam deposition at ion energies of 1500 and 700 eV greatly decreased both the friction and wear of fine-grain CVD diamond films in ultrahigh vacuum, without sacrificing

the low friction and low wear properties in dry nitrogen and in humid air. Thus, a DLC film deposited on a fine-grain CVD diamond film can be an effective wear-resistant, lubricating coating in all three environments investigated.

#### ACKNOWLEDGMENTS

This study was supported by U.S. Air Force Scientific Research through Wright Laboratory under Contract F33615-95-2551 with K Systems Corporation. The authors thank Drs. M. Murakawa, S. Miyake, S. Watanabe, and S. Takeuchi of the Nippon Institute of Technology for producing the CVD diamond pin specimens; Dr. Susan L. Heidger for the Raman analysis; Ronald E. Miller for the experimental setups; and Duane J. Dixon for the scanning electron microscopy.

#### REFERENCES

1. K. Miyoshi, R.L.C. Wu, A. Garscadden, P.N. Barnes and H.E. Jackson, NASA TM-105926 (1993) and J. Appl. Phys. 74 (1993) 4446-4454.
2. R.L.C. Wu, K. Miyoshi, A.L.K. Both, A. Garscadden and P.N. Barnes, Surf. Coat. Technol. 62 (1993) 589-594.
3. K. Miyoshi, S. Heidger, A.L.K. Both, D.T. Jayne, P. Herrera-Fierro, B. Shogrin, P. J. Wilbur, R.L.C. Wu, A. Garscadden and P. N. Barnes, NASA TM-106682 (1994).
4. R.L.C. Wu, A.K. Rai and K. Miyoshi, Appl. Phys. Lett. 68 (1996) 1054-1056.
5. B. Bhushan and B.K. Gupta, *Handbook of Tribology* (McGraw-Hill, Inc. (1991) 12.11-12.13).
6. R.L.C. Wu, W. Lanter, K. Miyoshi, S.L. Heidger, P. Bletzinger and A. Garscadden, Mat. Res. Soc. Symp. Proc. 354 (1995) 63-68.
7. R.L.C. Wu, A.K. Rai, A. Garscadden, P. Kee, H.D. Desai and K.J. Miyoshi, Appl. Phys. 72 (1992) 110-116.
8. M. Murakawa and S. Takeuchi, Mat. Sci. Eng. A140 (1991) 759-763.
9. H.O. Pierson, *Handbook of Carbon, Graphite, Diamond, and Fullerenes. Properties, Processing, and Applications* (Noyes Publications, Park Ridge, NJ (1993) 245-247).

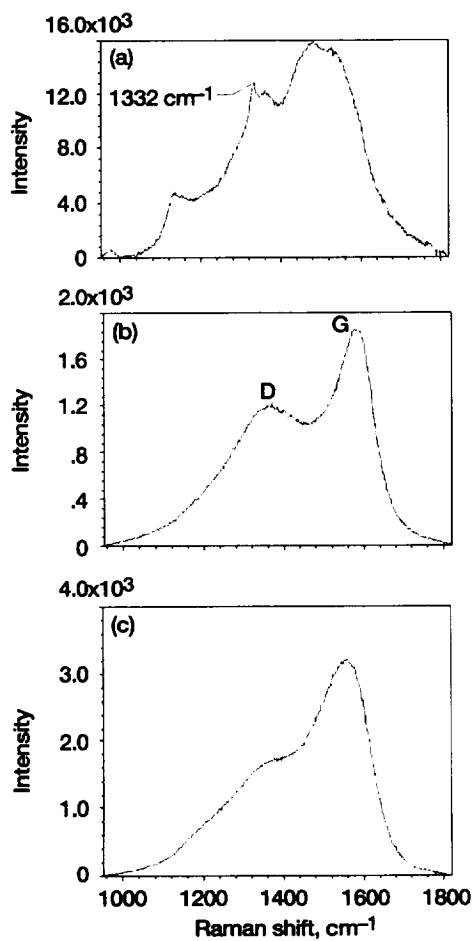


Figure 1.—Raman spectra. (a) Fine-grain diamond film. (b) Diamondlike carbon film on fine-grain diamond at an ion energy of 1500 eV. (c) Diamondlike carbon film on fine-grain diamond at an ion energy of 700 eV.

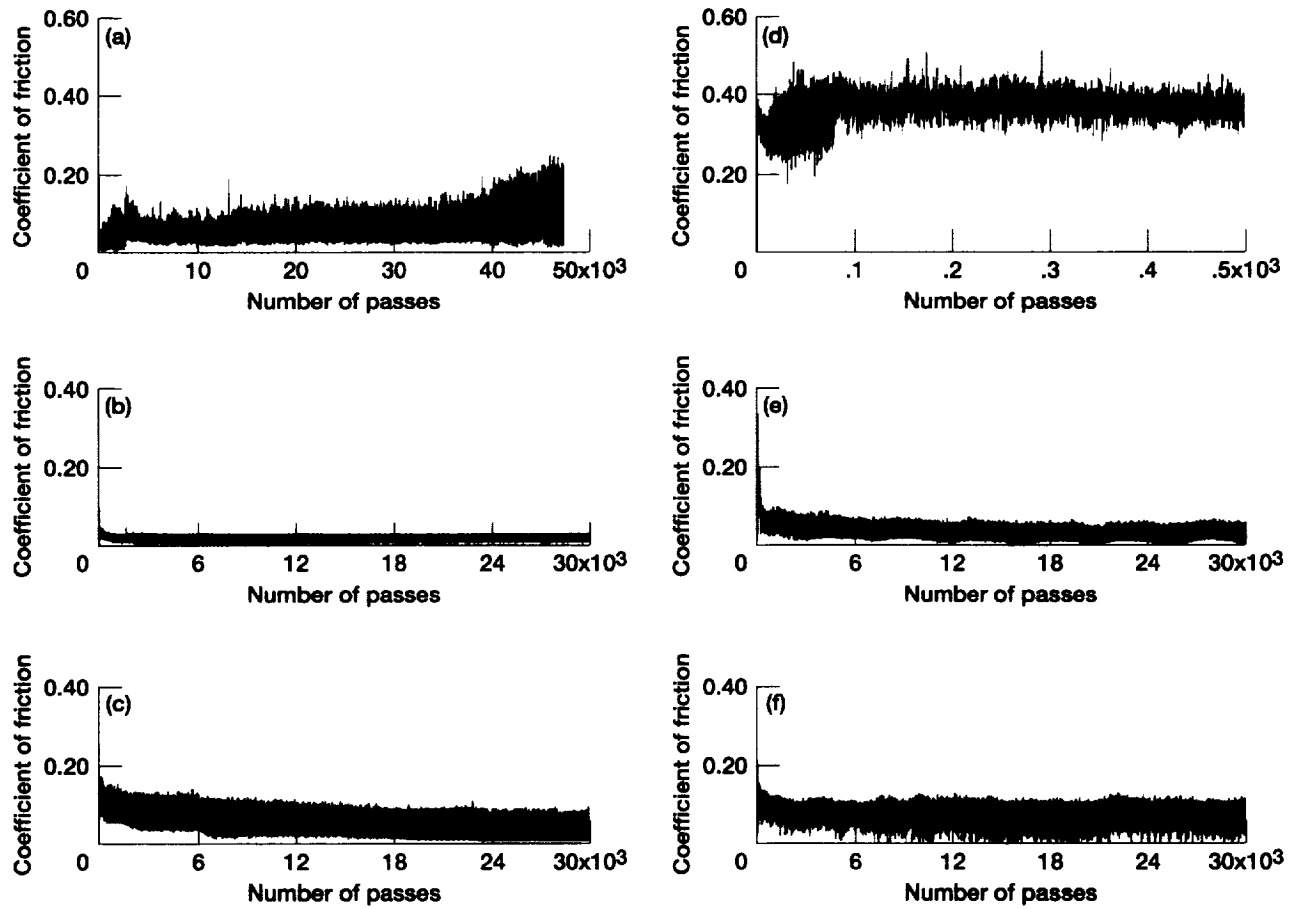


Figure 2.—Typical examples of friction traces for films in sliding contact with chemical-vapor-deposited diamond pins in ultrahigh vacuum, dry nitrogen, and humid air environments at room temperature. (a) Diamondlike carbon (DLC) film in ultrahigh vacuum. (b) DLC film in dry nitrogen. (c) DLC film in humid air. (d) Fine-grain diamond film in ultrahigh vacuum. (e) Fine-grain diamond film in dry nitrogen. (f) Fine-grain diamond film in humid air.



Ion energy used to deposit  
diamondlike carbon,  
eV

○ 1500  
□ 700

Open symbols indicate humid air environment  
Half-open symbols indicate dry nitrogen environment  
Solid symbols indicate ultrahigh vacuum environment

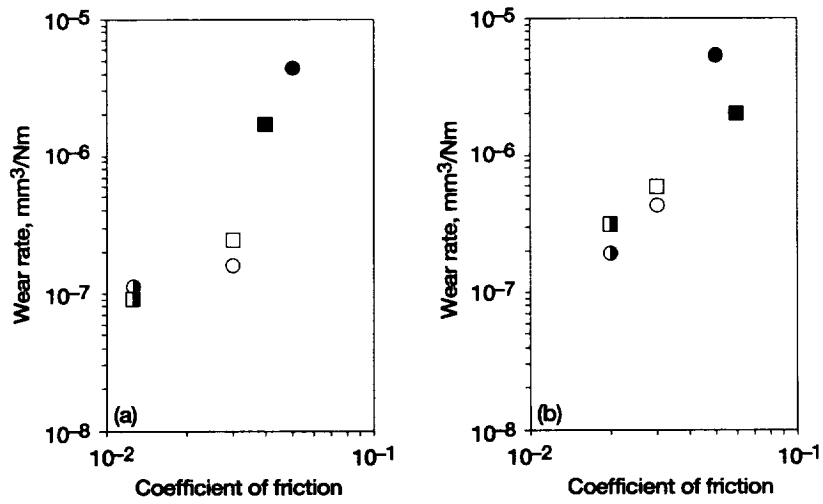


Figure 3.—Coefficients of friction and wear rates of diamondlike carbon.  
(a) Deposited on fine-grain diamond. (b) Deposited on silicon at ion energies  
of 1500 and 700 eV.

- DLC deposited on fine-grain diamond at 1500 eV
  - △ Fine-grain diamond
  - ◇ Carbon-ion-implanted, fine-grain diamond
- Open symbols indicate humid air environment  
 Half-open symbols indicate dry nitrogen environment  
 Solid symbols indicate ultrahigh vacuum environment

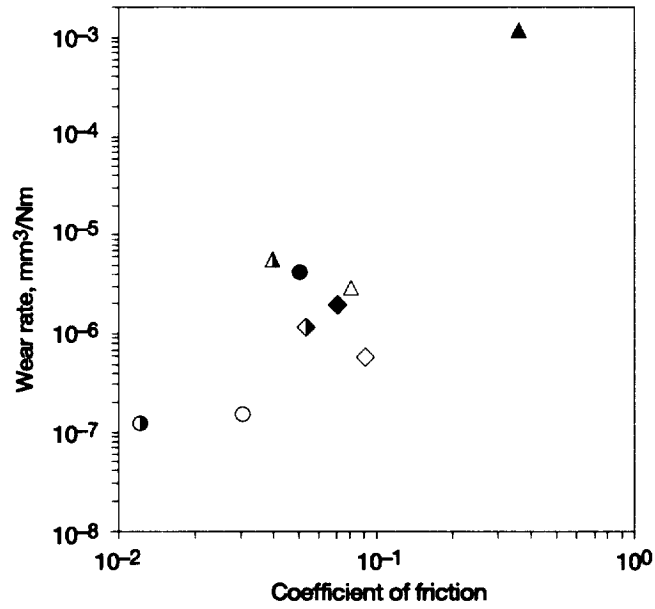


Figure 4.—Coefficients of friction and wear rates of diamond-like carbon deposited on fine-grain diamond, as-deposited, fine-grain diamond, and carbon-ion-implanted, fine-grain diamond films in sliding contact with chemical vapor deposited diamond pins in ultrahigh vacuum, dry nitrogen, and humid air environments.



# REPORT DOCUMENTATION PAGE

Form Approved  
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

<b>1. AGENCY USE ONLY (Leave blank)</b>		<b>2. REPORT DATE</b> October 1996	<b>3. REPORT TYPE AND DATES COVERED</b> Technical Memorandum	
<b>4. TITLE AND SUBTITLE</b> Friction and Wear of Ion-Beam-Deposited Diamondlike Carbon on Chemical-Vapor-Deposited, Fine-Grain Diamond			<b>5. FUNDING NUMBERS</b>  WU-505-63-5A	
<b>6. AUTHOR(S)</b>  Kazuhisa Miyoshi, Richard L.C. Wu, and William C. Lanter				
<b>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</b>  National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135-3191			<b>8. PERFORMING ORGANIZATION REPORT NUMBER</b>  E-10320	
<b>9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)</b>  National Aeronautics and Space Administration Washington, D.C. 20546-0001			<b>10. SPONSORING/MONITORING AGENCY REPORT NUMBER</b>  NASA TM-107316	
<b>11. SUPPLEMENTARY NOTES</b>  Kazuhisa Miyoshi, NASA Lewis Research Center; Richard L.C. Wu and William C. Lanter, K Systems Corporation, 1522 Marsetta Drive, Dayton, Ohio 45432. Responsible person, Kazuhisa Miyoshi, organization code 5140, (216) 433-6078.				
<b>12a. DISTRIBUTION/AVAILABILITY STATEMENT</b>  Unclassified - Unlimited Subject Category 27  This publication is available from the NASA Center for AeroSpace Information, (301) 621-0390.			<b>12b. DISTRIBUTION CODE</b>	
<b>13. ABSTRACT (Maximum 200 words)</b>  Friction and wear behavior of ion-beam-deposited diamondlike carbon (DLC) films coated on chemical-vapor-deposited (CVD), fine-grain diamond coatings were examined in ultrahigh vacuum, dry nitrogen, and humid air environments. The DLC films were produced by the direct impact of an ion beam (composed of a 3:17 mixture of Ar and CH <sub>4</sub> ) at ion energies of 1500 and 700 eV and an RF power of 99 W. Sliding friction experiments were conducted with hemispherical CVD diamond pins sliding on four different carbon-base coating systems: DLC films on CVD diamond; DLC films on silicon; as-deposited, fine-grain CVD diamond; and carbon-ion-implanted, fine-grain CVD diamond on silicon. Results indicate that in ultrahigh vacuum the ion-beam-deposited DLC films on fine-grain CVD diamond (similar to the ion-implanted CVD diamond) greatly decrease both the friction and wear of fine-grain CVD diamond films and provide solid lubrication. In dry nitrogen and in humid air, ion-beam-deposited DLC films on fine-grain CVD diamond films also had a low steady-state coefficient of friction and a low wear rate. These tribological performance benefits, coupled with a wider range of coating thicknesses, led to longer endurance life and improved wear resistance for the DLC deposited on fine-grain CVD diamond in comparison to the ion-implanted diamond films. Thus, DLC deposited on fine-grain CVD diamond films can be an effective wear-resistant, lubricating coating regardless of environment.				
<b>14. SUBJECT TERMS</b>  Diamondlike carbon coatings; CVD diamond coatings; Tribology			<b>15. NUMBER OF PAGES</b> 10	
			<b>16. PRICE CODE</b> A02	
<b>17. SECURITY CLASSIFICATION OF REPORT</b> Unclassified	<b>18. SECURITY CLASSIFICATION OF THIS PAGE</b> Unclassified	<b>19. SECURITY CLASSIFICATION OF ABSTRACT</b> Unclassified	<b>20. LIMITATION OF ABSTRACT</b>	