ANL/CP--72657

DE91 010816

Received by

APR 2 4 1991

A TRIBOLOGICAL INVESTIGATION OF THE GRAPHITE-TO-DIAMOND-LIKE BEHAVIOR OF AMORPHOUS CARBON FILMS ION-BEAM-DEPOSITED ON CERAMIC SUBSTRATES*

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March 1991

To be presented at the 18th International Conference on Metallurgical Coatings and Thin Films Conference, April 22-26, 1991, San Diego, CA

* Work supported by the U.S. Department of Energy, Office of Advanced Transportation Materials, under Contract W-31-109-Eng.-38.

MASTER

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A TRIBOLOGICAL INVESTIGATION OF THE GRAPHITE-TO-DIAMOND-LIKE BEHAVIOR OF AMORPHOUS CARBON FILMS ION-BEAM-DEPOSITED ON CERAMIC SUBSTRATES

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ABSTRACT

A tribological investigation was conducted on the graphite-to-diamond-like behavior of hard carbon films produced on SiC, Si_3N_4, and ZrO_2 substrates by means of ion-beam-deposition. Friction tests were performed on a ball-on-disk machine with pairs of various ceramic balls and disks coated with hard carbon films in dry and humid air, Ar, and N_2. The friction coefficients of carbon films sliding against Si_3N_4 and sapphire balls were in the range of 0.02 - 0.04 in N_2 and Ar but significantly higher ($\approx$ 0.15) in humid air. The wear rates of ceramic disks coated with carbon films were unmeasurable, and, depending on the test environment, the wear rates of counterface ceramic balls were reduced by factors of two to four orders of magnitude below those of balls that were slid against uncoated ceramic disks. Graphite disks were also tested, to obtain friction data that can help us understand the graphite-to-diamond-like tribological behavior of carbon films. Micro laser-Raman spectroscopy and scanning electron microscopy were employed to analyze the structure and chemistry of worn surfaces and to elucidate the graphite-to-diamond-like tribological behavior of amorphous carbon films.

INTRODUCTION

A unique combination of good chemical inertness, low static and dynamic friction, and high wear resistance of diamond-like carbon (DLC) films make them good prospects for a wide range of tribological applications. One example is the magnetic disk media where these films have already been used to achieve low friction and high wear resistance [1]. Nowadays, there exists a growing interest in expanding their applications to tooling and high-precision ball-bearing applications, and aerospace mechanisms.

DLC films are deposited by a variety of methods including DC and RF plasma assisted CVD, sputtering in a glow discharge, and ion beam deposition [2-5]. Carbon-bearing gases and/or graphite are commonly used as the source materials [2-5]. The deposition condition is such that the film growth takes place under continuous ionic/atomic bombardment. Loosely bound carbon atoms, that can lead to graphite precursors, are sputtered off. The deposition of DLC films can be realized at room temperature or at temperatures up to 350°C. The resultant DLC films are predominantly amorphous [2,5,6]. According to Weissmantel et al. [6], the amorphous structure of DLC films results from the rapid quenching of the thermal spikes and the collapse of the pressure spikes that are created by the high energy ions/atoms impinging constantly on the growing film surfaces. Recent studies have demonstrated that within the largely amorphous structure of DLC films, very short range ordered diamond (characterized by sp^3 type tetrahedral bonds) and graphite (characterized by sp^2 type trigonal bonds) phases also exist [1]. These films are often described as being degenerate or imperfect forms of diamond and/or graphite with properties varying from diamond-like to graphite-like. Depending on the type of deposition method, large amounts of hydrogen were also found in most DLC films [1,5]. Sometimes, these films are referred to as hydrogenated amorphous carbon films. Hydrogen is thought to play important roles in the stabilization of the sp^3 type bonds [1].

The primary purpose of this study is to investigate the graphite-to-diamond like tribological behavior of DLC films produced on various ceramic substrates by ion-beam-deposition. Moreover, the effects of load, environment, and counterface material on the friction and wear characteristics of DLC films are explored. Graphite, glassy carbon, and single crystal diamond are also incorporated in the experimental work to help understand the graphite-to-diamond like tribological behavior of DLC 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 sintered Si_3N_4 SiC, and yttria-stabilized ZrO_2. Selected properties of these materials are presented in Table 1. Volume porosity of these materials was less than 1%. The disk specimens, 50 mm in diameter by 6 mm thick, were diamond polished to a surface finish of better than 0.03 μm center-line average (CLA). Pyrolitic graphite, glassy carbon and single crystal diamond were also used in the experimental program.

The counterface balls were made of 9.5-mm diameter Si_3N_4, 6.3-mm diameter sapphire, and 1.6-mm diameter single crystal diamond balls with a surface finish of better than 0.05 μm CLA. They were firmly attached to the ball holder of a ball-on-disk machine to assume the vertical bal-on-disk configuration. Balls and uncoated 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 (IBD) of Diamond-Like Carbon

The IBD of thin DLC films (e.g., approximately 2 µm thick) was performed at room temperature in a vacuum chamber equipped with a broad beam ion source. A schematic illustration of this system is shown in Fig. 1. Methane gas was fed through the cylindrical ion source (see Fig. 1) and used as the carbon source. Methane was ionized by energetic electrons produced by a hot filament wire. Ionized species pass through a grid where they gain high acceleration energy and reach a hot wire filament emitting thermionic electrons which neutralize the incoming ions. Discharging of ions is important when using insulating ceramics as substrates.

Coatings were applied on substrates in either two or three step processes. Two step process was used for SiC and Si_3N_4 substrates. Specifically, the samples were positioned directly under the ion source and sputter cleaned with a 1 keV, 200 mA Ar ion beam for about 3 to 5 minutes. The DLC film was applied immediately after cleaning by increasing the methane flow through the cylindrical ion source. The operating conditions were adjusted to give an ion beam with an acceleration energy of 400 eV and a current density of ≈ 2.5 mA/cm_2. At this operating condition, the deposition rate was ≈ 3 µm/hr. Deposition was continued until obtaining a 2 µm thick DLC film.

For ZrO_2 substrates, a three step process was used to deposit DLC films. After sputter cleaning, an intermediate SiC layer was sputter deposited on these substrates. This layer was applied by rotating the sample out of the beam and into the sputter-coating position as shown in Fig. 1 after the sputter-cleaning step. Methane is bled into the vacuum chamber at this point and the 1 keV argon beam current is allowed to sputter the silicon target shown in the figure. The combination of the Si being sputtered onto the sample and C derived from the methane environment result in SiC. A 100 to 200 nm thick layer of SiC deposited on zirconia and was found to be sufficient for achieving excellent bonding between substrate and DLC coatings. At the conclusion of SiC step, the source was switched to methane operation, the sample was rotated back to the position directly beneath the beam, and the DLC coating was applied using the conditions described previously. Water or LN2 cooling of the samples via their mounting plates to keep their temperatures below 200°C during processing was found to be essential to the application of high quality films onto the samples.

Friction and Wear Tests

Friction and wear tests were performed with pairs of various balls and disks on a ball-on-disk tribometer. Tests were run in dry and humid air, and nitrogen and argon. A plexiglass cover was fitted over the pin-on-disk machine, permitting various environments to be created. A few pairs without the DLC films were wear-tested primarily to assess and compare their tribological performance to that of pairs with a DLC film. The dead weight applied on top of the balls ranged from 1 to 20 N which created initial mean Hertzian contact pressures of approximately 0.4 to 1.5 GPa. Frictional force was monitored with the aid of a linear variable-displacement-transducer and recorded on chart papers throughout the tests. The rotational velocity of disk specimens was kept between 8 to 15 rpm to provide the same sliding speed of 2.6 cm/s. The sliding distance was 165 m.

Wear-volume measurements on the balls were based on microscopic determination of the diameter of the circular wear scars. The wear of disk specimens was estimated from the traces of surface profiles across the wear tracks. For better accuracy and reproducibility, duplicate tests were run, and the average values are reported. Wear scars and tracks were examined with scanning electron microscopy and micro laser-Raman spectroscopy.

RESULTS

Friction and Wear

Figure 2 shows the effects of test environment and load on the steady-state friction coefficients of DLC films sliding against the Si_3N_4 and sapphire balls. As is clear, the friction coefficient decreased steadily with increasing load of up to 10 N in both the normal air (e.g., ≈40% relative humidity) and Ar atmosphere. When the load was increased to 20 N, the friction coefficients tended to increase. The DLC film that was deposited on Si_3N_4 substrate could not endure 20 N load. It was removed quickly from the sliding wear track during wear tests in normal air. As can be noted from Fig. 2, the friction coefficients in Ar is approximately an order of magnitude lower than that in normal air.

Figure 3 shows the friction coefficients of Si_3N_4 and sapphire balls during sliding against DLC-coated Si_3N_4 and ZrO_2 disks, respectively, in Ar, N_2, and in dry (≈0% RH), normal (50±5% RH), and humid air (90±5% RH). The friction coefficient of a bulk-diamond pin sliding against a Si_3N_4 disk is also included in this figure for comparison. As can be noted, the friction coefficients of Si_3N_4, sapphire, and diamond balls are significantly lower in Ar and N_2 than those observed in air. Also, the frictional behavior of diamond appears less dependent on environment.

Figure 4 shows the wear rates of Si_3N_4 balls during sliding against the uncoated and DLC-coated SiC substrates in various test environments under a 2 N load. As is clear, the wear rates of balls are reduced markedly when they are rubbed against the DLC-coated substrates, with the lowest wear occurring in Ar and N_2 environments. The measurement of the amount of wear on DLC-coated SiC was hard to achieve. We used a surface profilometer at vertical magnifications up to 50,000X, but could not record any measurable wear.

Figure 5 shows the friction coefficients of Si_3N_4 balls during sliding against the pyrolitic graphite and glassy carbon disks in different environments. The friction coefficients of balls sliding against both the amorphous carbon and graphite disks tend to decrease with increasing humidity. The frictional behavior of amorphous carbon appears less affected by humidity than that of graphite. The friction coefficients of balls sliding against the graphite disk are substantially high in Ar, N_2, and dry air.

Structural and Chemical Characteristics of DLC Films

Figure 6 shows a cross-sectional scanning electron micrograph of a DLC film deposited on a SiC substrate. The microstructure of DLC looks extremely dense and free of volume defects. Moreover, this film does not appear to possess a columnar morphology which is typical of most ceramic and metallic films produced by physical vapor deposition techniques. The interface between film and substrate is continuous and hard to discern, suggesting that the bonding between film and substrate is good.

Figure 7 summarizes the mirco laser-Raman spectroscopy results obtained from the surface of a DLC film before and after wear testing in dry and humid air. For comparison, spectra from graphite and glassy carbon specimens were also included.

The Raman spectrum of a virgin DLC surface is similar to that of a wear track formed during sliding in dry air. The main features of these two spectra are that there exist a strong peak at ≈1290 cm^{-1} and a broad peak at ≈1570 cm^{-1}. After wear test in humid air, the peak at ≈1290 cm^{-1} remains unchanged but the broad peak at 1570 cm^{-1} becomes flattened with a new peak at ≈1800 cm^{-1}. In the spectrum of the virgin DLC surface, one can notice a minor peak at 1334 cm^{-1} which may represent the crystalline diamond phase. After a wear test in dry air, this peak appears to have shifted to ≈1336 cm^{-1}, but becomes hard to discern on the wear track formed in humid air. In natural diamond, this peak is normally found at 1332 cm^{-1}, however, a slight shift can occur when the sp^3 bonds are subject to high internal stresses [10]. When further stressing occurs during wear test, the diamond line shifts further from 1334 to 1336 cm^{-1} as shown in Fig. 7. The pyrolitic graphite has a strong peak at 1580 cm^{-1} and a relatively weak one at 1355 cm^{-1}. Whereas, the glassy carbon is characterized by two broad peaks. One located at around 1350 and the other is at 1600 cm^{-1}.

DISCUSSION

The results of this study demonstrate that the DLC films can provide very low friction coefficients and wear rates to sliding ceramic surfaces (see Figs. 2 to 4). These results are consistent with those of the previous investigators [2,5,11]. During tests in air with increasing relative humidity, the friction and wear performance of DLC coatings degrades, suggesting that the gaseous species in the surrounding environment have profound effects on their tribological behavior. As shown in Fig. 3, the friction coefficients of the DLC films sliding against the Si_3N_4 and sapphire balls are comparable to the friction coefficient of a diamond ball sliding against a Si_3N_4 disk when tested in Ar and N_2. This observation suggests that the DLC films behave like diamond in such environments. However, a disparity occurs when tests run in air with increasing humidity.

Compared to that of diamond, the friction coefficients of the DLC films increase substantially.

Test results from graphite and glassy carbon differ considerably from those obtained from DLC films (see Fig. 5). The friction coefficients of balls sliding against the graphite disks decrease substantially with increasing humidity. Although not as pronounced, a similar trend is also seen for glassy carbon material. This may be due to the fact that the microstructure of glassy carbons is an aggregate of very small graphite particles [1]. Because of a very short-range atomic order, graphite particles can not behave like crystalline graphite.

Currently, there exist a few mechanistic explanation for the frictional behavior of policrystalline diamond and DLC films. These include: chemisorption of hydrogen and/or other chemically active species [7-9], double-layer structure and/or micro-graphitization [10,11], and transfer-layer formation [12]. Some of these mechanisms are highly hypothetical and are based on limited amount of analytical data obtained from a specific DLC film under very specific test conditions. Since the family of the diamond and DLC films is rather large and varies from more graphite-like to more diamond-like, the proposed mechanisms should not be considered universal. They fall short in explaining wide differences in reported results.

In general, a logical interpretation of the mechanisms that control the frictional behavior of the DLC films must take into account the structure and chemical nature of the microcontacts at sliding interfaces. Because of the dynamic nature of sliding tests, both the chemical and structural nature of these microcontact interfaces changes. For most soft metals and layered solids, low friction is due to interfilm and/or interlayer shear, respectively. However, with their high mechanical hardness and rigidity, DLC films can not undergo shear. We believe that in DLC films, easy shear occurs largely between the film and the counterface pin surfaces. Because of their high chemical inertness, the amount of chemical attraction between pin and disk sides causing adhesion is limited. The open, dangling bonds of the carbon atoms lying on the surface are largely passivated by hydrogen atoms that are present in these films. Hydrogen may have been picked up from surrounding atmosphere too. Recent tests by Pepper [7], Gardos [8], and Bowden [9] demosntrated that the removal of hydrogen from the top surface of DLC and diamond films, and single-crystal diamond resulted in an increased adhesion, hence friction.

The above explanation holds for the DLC films tested in this study. Laser-Raman spectroscopy indicated some evidence of diamond phase or sp^3 type bonding (characterized by a Raman line at 1334 cm^{-1}) in their predominantly amorphous structures (see Fig. 7). However, when the environment becomes humid, the friction coefficients of DLC films increase substantially during sliding against the ceramic pins, whereas, the friction coefficient of diamond against Si_3N_4 increases only marginally (see Figs. 2 and 3). For this type of disparity, we consider two possible mechanisms. One which was proposed by Miyoshi et al. [5] involves the saturation of microcontacts with water molecules causing increased bonding across the interfaces, and the other is micrographitization of contact interfaces due to mechanochemical interactions [10,11]. According to Miyoshi et al [5]., water molecules in humid air fill in the valleys and other surface irregularities. During sliding contact, water molecules exert residual bonds across the opposing surfaces, hence give rise to higher friction.

In the micrographitization case, mechanochemical interactions between sliding interfaces and the surrounding environments can lead to the graphitization of microcontact regions. As a result, DLC films behave much like graphite.

Furthermore, graphite-like carbon with an sp^2 type bonding may become increasingly active in humid environments. Tribological tests by Miyoshi et al [5] demonstrated that DLC films with more graphite-like and/or graphitized structures behaved much like graphite. Laser-Raman spectroscopy results in Fig. 7 suggest that some structural changes had occurred during sliding tests in humid air. Because, the spectrum from a wear track formed in humid air differs considerably from those of a wear track formed in dry air and undisturbed DLC film. One special feature of the Raman spectrum of the wear track formed in humid air is that the emergence of a flattened peak which covers the strongest Raman line of pyrolitic graphite (see Fig. 7). This suggests that perhaps a thin layer with a more graphite-like structure was formed on this sliding surface.

To explore this possibility further, we conducted two types of special tests. In one case, we purposely increased and decreased humidity within the test chamber and monitored the change in friction coefficients of DLC films during sliding against a sapphire ball. The result of this test is presented in Fig. 8. As can be seen, the friction coefficient of the DLC film increases with decreasing relative humidity. When the humidity is increased again, the friction coefficient starts decreasing. This type of behavior is typical of graphite. As can be seen in Fig. 8, this behavior is highly reproducible. In the second case, we slid a sapphire pin in Ar over a track that was formed in humid air. The other test conditions were kept the same. As can be realized from Fig. 9, the friction coefficient of sapphire pin was high initially, but decreased steadily during successive sliding cycles and stabilize at around 0.03 after about 100 cycles. Whereas the friction coefficient of a sapphire pin slid against a new track reached a steady-state value of ~0.02 after 10 sliding cycles. These observations suggest that there may have been a thin graphitic film formed on the contact interfaces of DLC films in humid air. The increase in friction with decreasing humidity in Fig. 8 is typical of graphite (see Fig. 5). The initially high friction coefficient in Fig. 9 further suggests that there was a thin layer of graphite on surface of DLC formed during a sliding test in humid air. As this film wears out, the friction coefficient decreases, because the sliding once again occurs between DLC film and counterface pin.

Formation of a transfer layer on the sliding surface of DLC films was observed by Hirvonen et al. [14]. They proposed that such transfer films play dominant roles in the friction and wear behavior of DLC films. In our studies, we could not found any transfer film on the sliding surfaces. Therefore, we discount this as a probable mechanism.

CONCLUSIONS

Based on the results presented above, we can draw the following conclusions.

1. In general, DLC films prepared by ion-beam deposition are capable of imparting low friction and wear to sliding ceramic surfaces.

2. The lowest friction and wear are achieved in Ar and N_2. The wear rates and the friction coefficients of ceramic balls sliding against DLC films tend to increase in air with increasing humidity.

3. The frictional behavior of DLC films is much like the bulk diamond in Ar and N_2, but deviates from that of diamond as in humid air.

4. In humid air, the DLC films behave much like bulk graphite.

ACKNOWLEDGEMENT

The authors wish to thank J. Parker of Argonne National Laboratory for performing the laser-Raman spectroscopy. This work was supported by the U.S. Department of Energy, Office of Transportation Materials, under Contract W-31-109-Eng-38.

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Table 1. Selected properties of test materials

Material	Density (g/cm^3)	Young's Modulus (GPa)	Hardness (GPa)	Fracture Toughness (MPa√m)
ZrO_2	6.0	206	13	6.7
Si_3N_4	3.2	294	17	5.7
Sapphire	3.9	470	22	-
SiC	3.2	431	-	5.6

FIGURE CAPTIONS

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

Fig. 2. Variation of friction coefficients of Si_3N_4 and sapphire balls during sliding against DLC-coated Si_3N_4 and ZrO^2 disks in Ar and normal air with increasing load.

Fig. 3. Friction coefficients of various pins during sliding against DLC-coated disks in different atmosphere.

Fig. 4. Wear rates of Si_3N_4 balls during sliding against DLC-coated and uncoated Si_3N_4 disks in different environments.

Fig. 5. Friction coefficients of Si_3N_4 balls during sliding against pyrolitic graphite and glassy-carbon in different environments.

Fig. 6. Cross-sectional scanning electron micrograph of a DLC film deposited on a SiC substrate.

Fig. 7. Laser-Raman spectra of a DLC film before and after wear tests in dry and humid air, and of pyrolitic graphite and glassy carbon.

Fig. 8. Variation of friction coefficient of a sapphire ball during sliding against a DLC-coated ZrO_2 disk as a function of relative humidity.

Fig. 9. Variation of friction coefficients of sapphire balls during sliding against a new wear track and against a track that was formed in humid air.

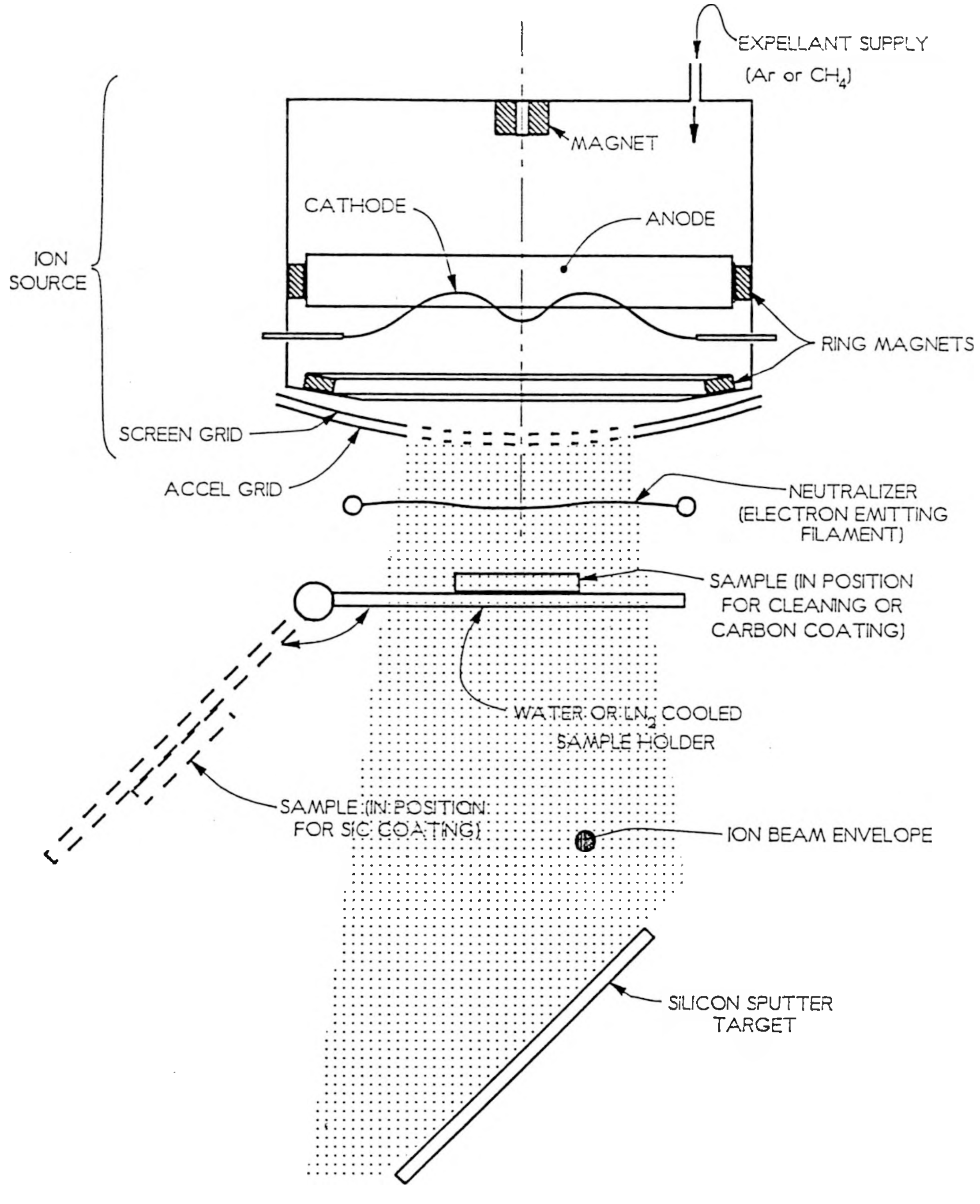

EXPELLANT SUPPLY
(Ar or CH_4)
MAGNET
CATHODE
ANODE
ION SOURCE
RING MAGNETS
SCREEN GRID
ACCEL GRID
NEUTRALIZER (ELECTRON EMITTING FILAMENT)
SAMPLE (IN POSITION FOR CLEANING OR CARBON COATING)
WATER OR LN_2 COOLED SAMPLE HOLDER
SAMPLE (IN POSITION FOR SiC COATING)
ION BEAM ENVELOPE
SILICON SPUTTER TARGET

Fig.1

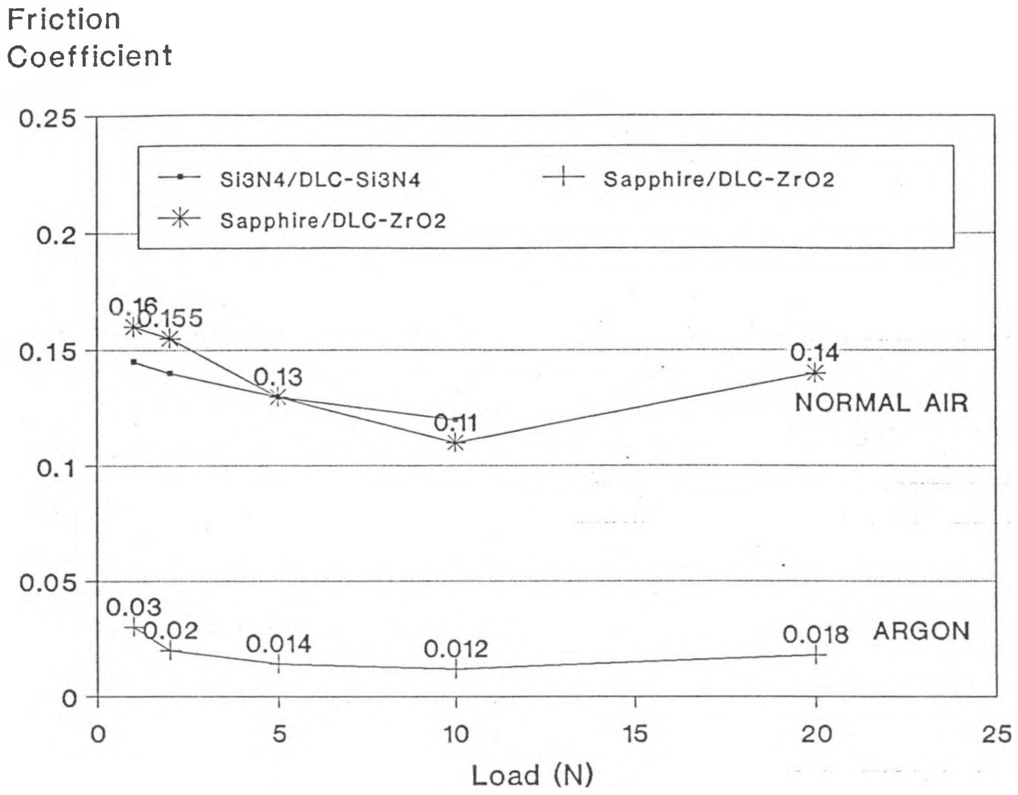

Friction Coefficient
0.25
0.2
0.15
0.1
0.05
0
Si3N4/DLC-Si3N4
Sapphire/DLC-ZrO2
Sapphire/DLC-ZrO2
0.16
0.155
0.13
0.11
0.14
NORMAL AIR
0.03
0.02
0.014
0.012
0.018
ARGON
0
5
10
15
20
25
Load (N)

Fig. 2

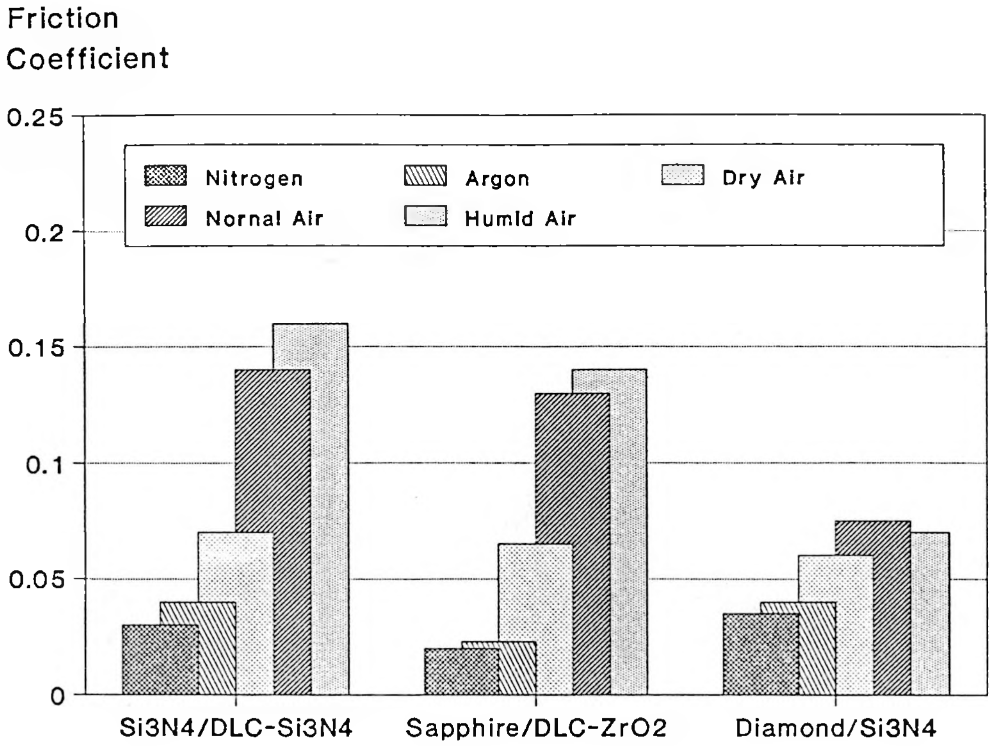

Friction Coefficient
0.25
0.2
0.15
0.1
0.05
0
Nitrogen
Argon
Dry Air
Nornal Air
Humid Air
Si3N4/DLC-Si3N4
Sapphire/DLC-ZrO2
Diamond/Si3N4

Fig. 3

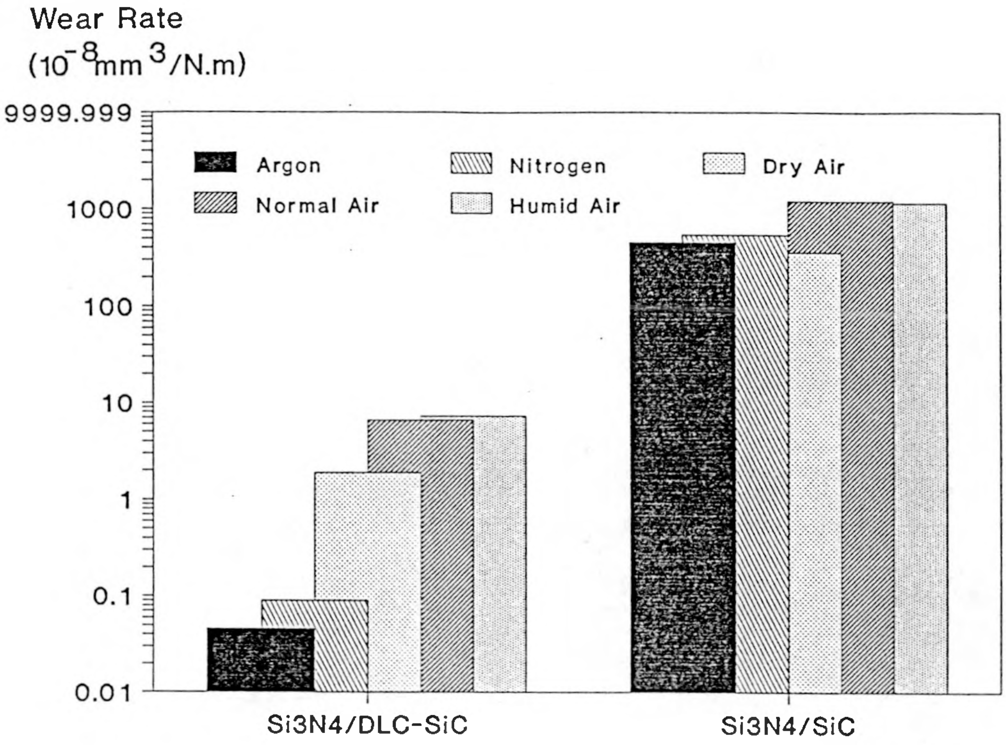

Wear Rate
(10^{-8} mm^3/N.m)
9999.999
1000
100
10
1
0.1
0.01
Argon
Nitrogen
Dry Air
Normal Air
Humid Air
Si3N4/DLC-SiC
Si3N4/SiC

Fig. 4

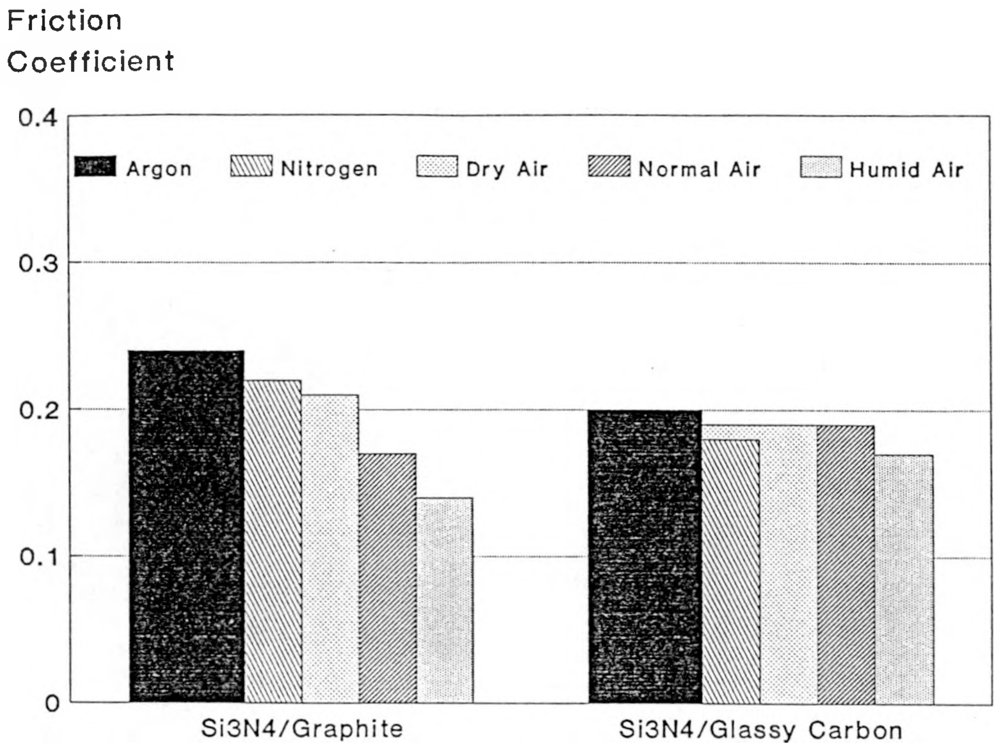

Friction Coefficient
0.4
0.3
0.2
0.1
0
Argon
Nitrogen
Dry Air
Normal Air
Humid Air
Si3N4/Graphite
Si3N4/Glassy Carbon

Fig. 5

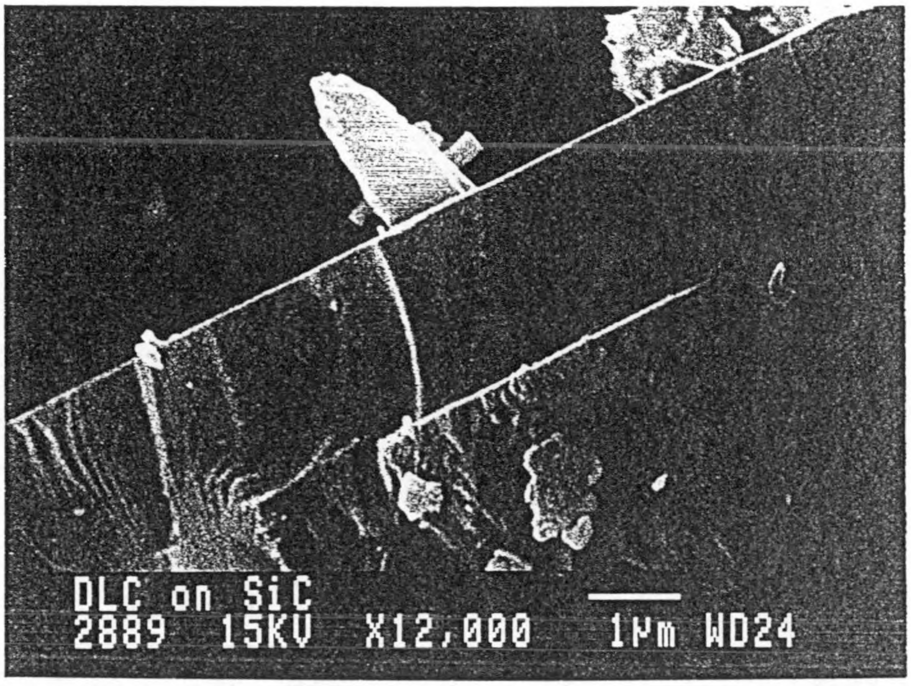


Fig. 6

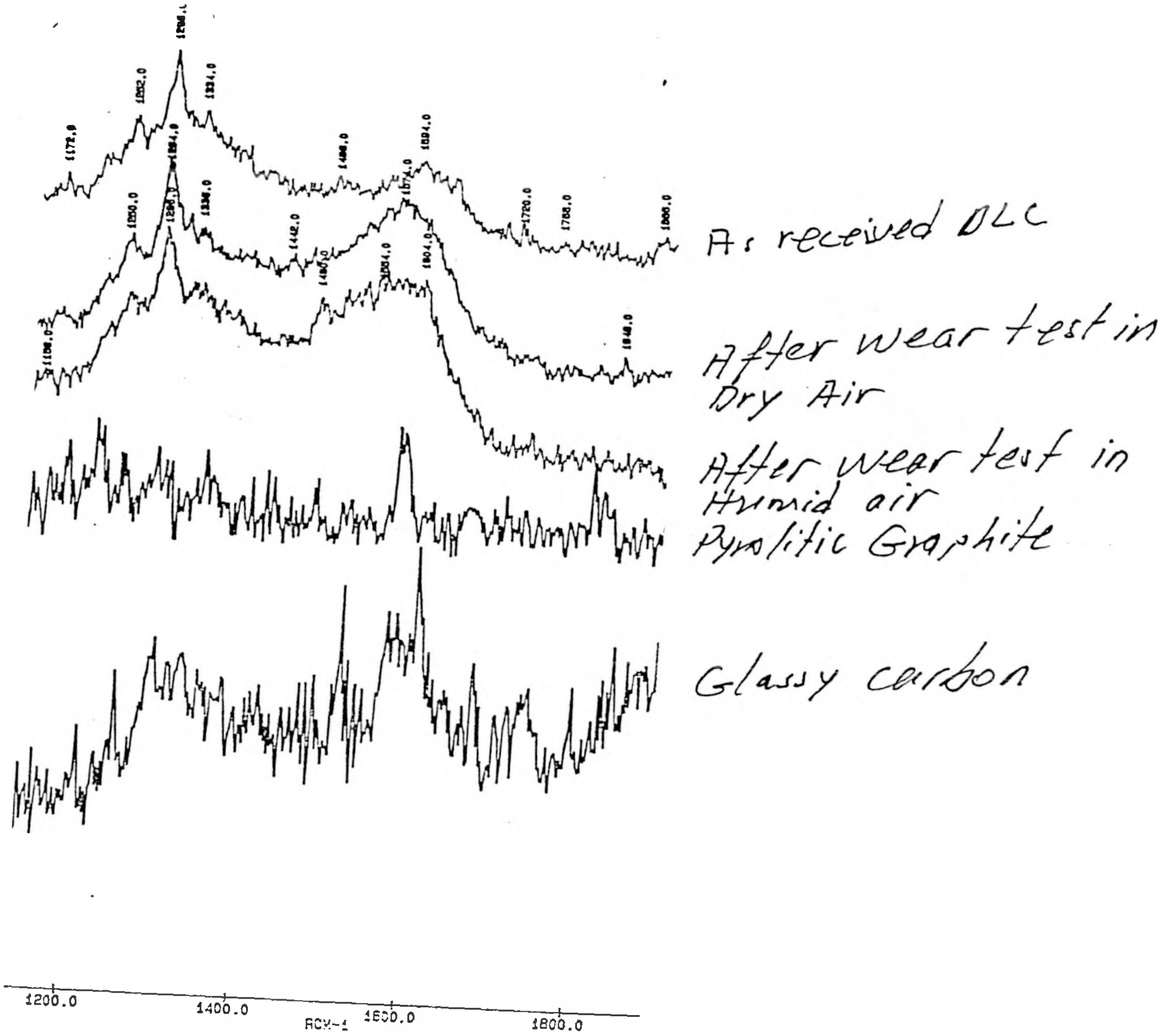
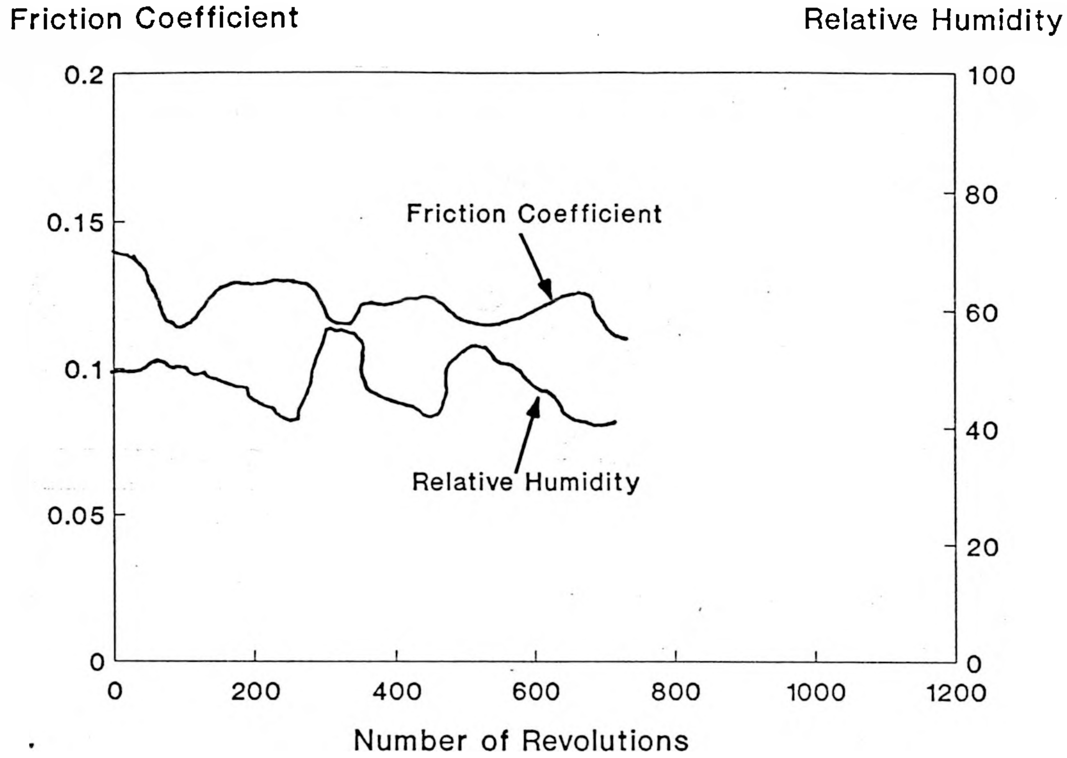

1172.0
1252.0
1296.1
1334.0
1486.0
1594.0
1574.0
1720.0
1788.0
1866.0
1250.0
1294.0
1336.0
1442.0
1604.0
1848.0
1106.0
1296.0
1480.0
1564.0
As received DLC
After wear test in Dry Air
After wear test in Humid air
Pyrolitic Graphite
Glassy carbon
1200.0
1400.0
RCM-1
1600.0
1800.0

Fig. 7



Friction Coefficient
Relative Humidity
0.2
0.15
0.1
0.05
0
100
80
60
40
20
0
Friction Coefficient
Relative Humidity
0
200
400
600
800
1000
1200
Number of Revolutions

Fig. 8

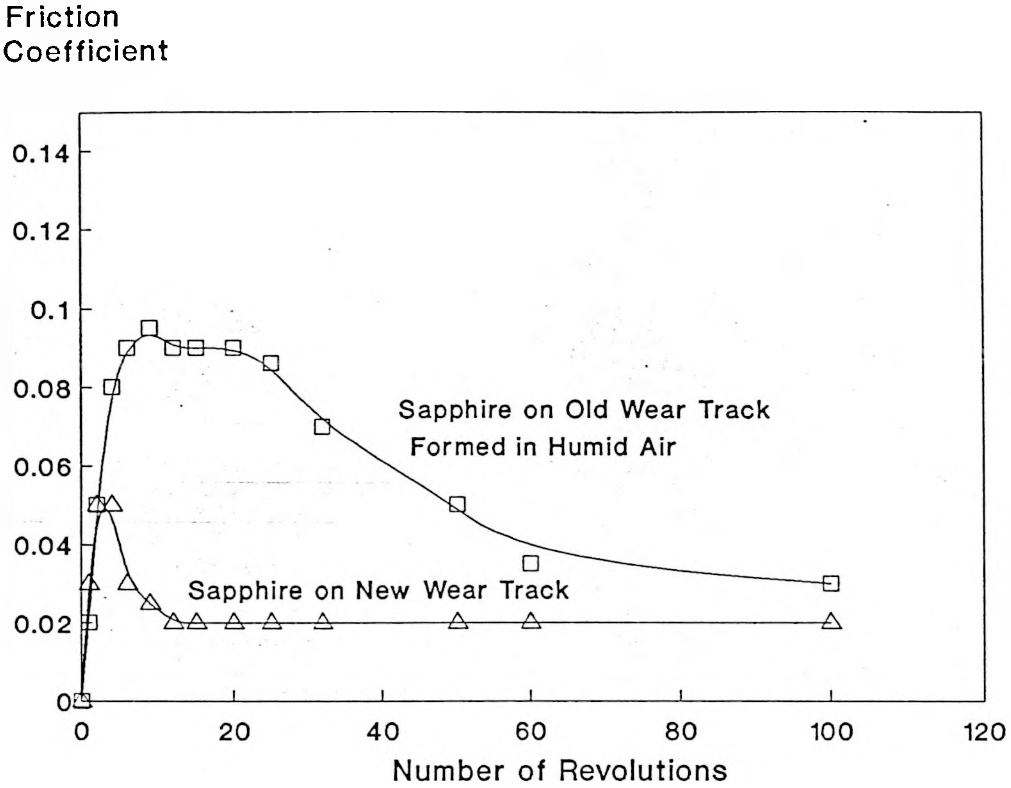

Friction
Coefficient
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
0
20
40
60
80
100
120
Number of Revolutions
Sapphire on Old Wear Track
Formed in Humid Air
Sapphire on New Wear Track

Fig. 9