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Friction and Wear of Diamond-Like Carbon Films Produced by Plasma-Assisted CVD Technique

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Diamond-like carbon (DLC) films were deposited on silicon substreates by using an RF plasmaassisted CVD apparatus; the effects of deposition conditions such as CH_4 gas pressure and substrate bias voltage on DLC film friction and wear were examined in both friction and scratch tests. In friction tests critical loads at which the friction coefficient increases abruptly depend on substrate bias voltages: critical loads deposited at a bias voltage of -100 V exceed those deposited at other bias voltages. Critical loads are correlated with DLC film hydrogen content. Critical DLC film loads in scratch tests depended considerably less than in friction tests. The friction coefficient of DLC films depends on neither substrate bias voltage nor CH_4 gas pressure.

Key words : Diamond-like carbon (DLC) film, RF plasma-assisted CVD, Friction, Wear, Critical load, Hydrogen content, Scratch test

I. Introduction

Diamond-like carbon (DLC) films are characterized by high hardness, low friction, high wear resistance, high corrosion resistance, and so on. DLC films have therefore been applied as protective films for metal molds for making aluminum cans and electronic components such as magnetic hard disks and video tape recorder capstans. Despite much research,[1-10] however, the effects of deposition techniques and conditions on the tribological properties of DLC films are not well known. The mechanisms behind low DLC film friction and wear also remain to be clarified.

In the present study, DLC films were produced by RF plasma-assisted chemical vapor deposition (CVD0 technique and the effects of deposition conditions on the tribological properties of DLC films were investigated

II. Details of Experiments

1. DLC films

DLC films were deposited on silicon wafers by using a RF plasma-assisted CVD apparatus (Fig. 1, Table 1). All films were prepared using CH_4 gas; the gas pressure was varied from 1.3 to 11.3 Pa. Films were deposited at substrate bias voltages from -100 to -1000 V. Film thickness was 400~500 nm, irrespective of deposition conditions.

Surface roughness, hardness, and hydrogen content were determined for the deposited DLC films. Surface roughness was measured by a noncontact 3-dimensional surface profile meter. Hardness was measured by a sultra-light load indentation hardness tester at a load of 9.8 mN. The content of hydrogen incorporated in DLC films was determined by elastic recoil detection analysis.

2. Friction and wear experiments

A ball-on-disk friction tester housed in an airtight vessel was used for friction and wear experiments (Fig. 2). A DLC film sample was mechanically fixed on a disk while a 3/16-inch diameter ball made of ceramics was used as the counterpart. Experiments were conducted in dry air, humid air, and ambient air with uncontrolled humidity (Table 2).

3. Scratch tests

Scratch tests were conducted using a commercialized scratch tester (Fig. 3). A cartridge with a diamond stylus having a radius of 15 μm was driven to swing parallel to the surface. A DLC film sample was mounted on a X-Y translator with a tiltable table and moved horizontally in a direction normal to the swing. As the sample moved, the stylus gradually pressed down on the sample.The scratch test was continued until the friction force abruptly increased.

III. Results and Discussion

1. DLC film characterization

DLC films have extremely smooth surfaces with roughness measurable in nm (Fig. 4). Bias voltage and CH_4

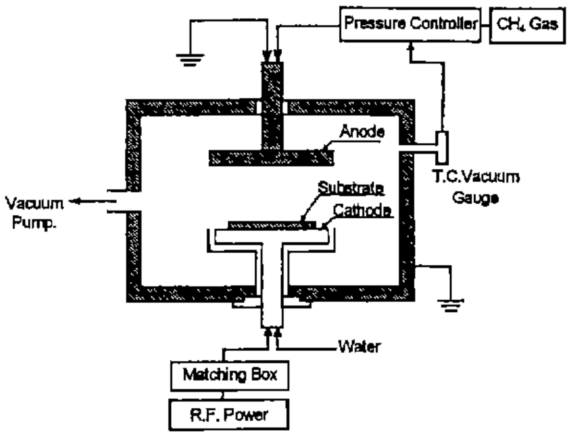

Fig. 1. RF plasma-assisted CVD apparatus.

Table 1. Deposition Conditions

Substrate	Si wafer
Gas	CH_4
Gas pressure	1 3, 6.7, 11.3 Pa
Bias voltage	-100, -400, -700, -1000 V
Thickness	400~500 nm

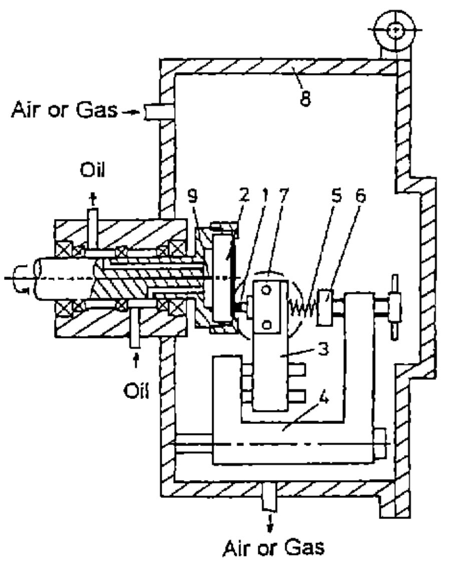

Fig. 2. Ball-on-disk friction tester.

gas pressure had little effect on surface roughness.

The hardness of DLC films deposited at different conditions is maximized at a bias voltage of -400 V, although the difference in hardness between -400 V and -700 V is low (Fig. 5). CH_4 gas pressure affects hardness only slightly.

In the measurement of hydrogen content in DLC films deposited under different conditions (Fig. 6), hydrogen content at a bias voltage of -100 V is definitely greater than at bias voltages of -400 V and -700 V, irrespective

Table 2. Conditions of Experiments

Normal load	2~55 N
Sliding velocity	14.9 cm/s (300 rpm)
Atmosphere	Ambient air (r.h.: 40~60%)
	Dry air (r.h. < 5%)
	Humid air (r.h.: > 85%)
Mating ball	SiC (Hv: about 27 GPa)
	Si_3N_4 (Hv: about 17 GPa)

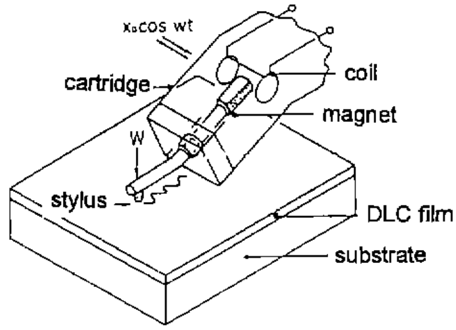

Fig. 3. Scratch tester.

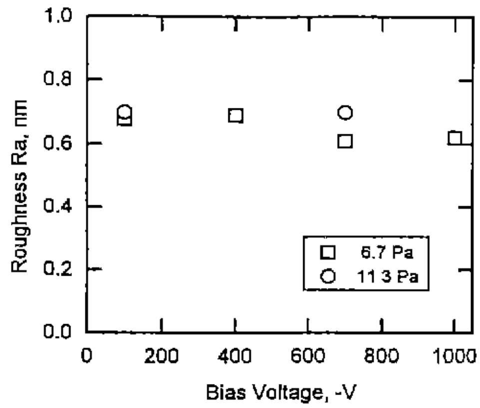

Fig. 4. Relationship between surface roughness of DLC films and bias voltage.

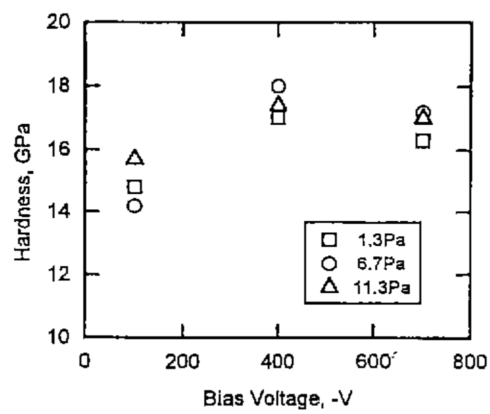

Fig. 5. DLC film hardness under different deposition conditions.

of CH_4 gas pressures. No difference is manifest between -400 V and -700 V.

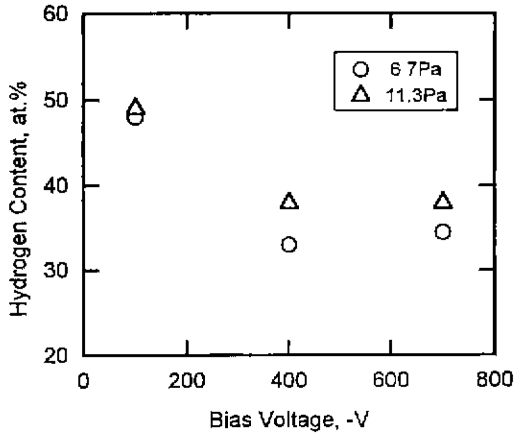

Fig. 6. Hydrogen content of DLC films under different deposition conditions.

2. *Critical loads in friction experiments*

First, we determined the critical loads at which the friction oefficient abruptly increased, i.e., at which DLC film wears off or ruptures. Experiments were conducted by increasing a normal load of 2 or 3 N until the friction coefficient abruptly increased. The friction coefficient decreased with increasing constant value if films do not wear off or rupture (Fig. 7). Similar frictional behavior was also observed in DLC films deposited under other conditions. As shown, the friction coefficient is nearly independent of bias voltage, and is scarcely affected by CH_4 gas pressure (data not shown).

DLC films deposited at a bias voltage of -100 V (Fig. 8) have considerably larger critical loads than those deposited at other bias voltages, irrespective of the type of ball material. Critical loads were, however, scarcely affected by CH_4 gas pressure. When the dependence of critical load on bias voltage is compared to that of DLC film hydrogen content on bias voltage (Fig 6), it is found that they are fairly similar. This suggests that the critical load of DLC films depends on hydrogen content. This

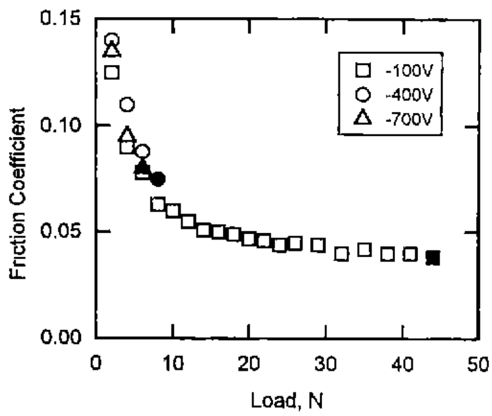

Fig. 7. Relationship between friction coefficient of DLC films deposited at gas pressure of 6.7 Pa and normal load (Closed symbols designate abrupt increase in friction. Mating ball: SiC).

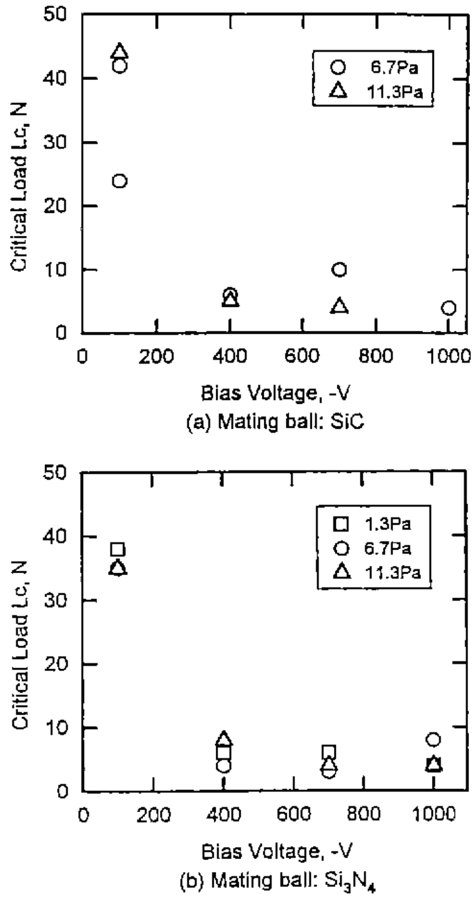

Fig. 8. Relationship between critical load of DLC films and deposition conditions.

correlation between tribological properties and hydrogen content is supported by the experiments of Wu *et al.*,[11] showing that DLC films wear depends strongly on hydrogen content, although the friction coefficient does not change significantly.

DLC films deposited at a bias voltage of -100 V have larger critical loads than those deposited at higher bias voltages (Figs. 5 and 8), even though the hardness of filmsdeposited at -100 V is slightly lower. This may suggest that DLC films deposited at -100 V are less brittle than those deposited at higher bias voltages.

3. *Critical loads in scratch tests*

In scratch test, when load was increased, the friction force abruptly increased at a certain load, designated as critical load Lcs. The relationship between Lcs and deposition conditions (Fig. 9) is like critical loads Lc (Fig. 8). The Lcs of DLC films deposited at a bias voltage of -100 V exceeds that of films deposited at higher voltages. The difference, however, of Lcs between -100 V and -400 V and -700 V is considerably smaller compared to Lc in friction experiments, although why is unknown. The effect of CH_4 gas pressure on critical load was not seen in scratch tests.

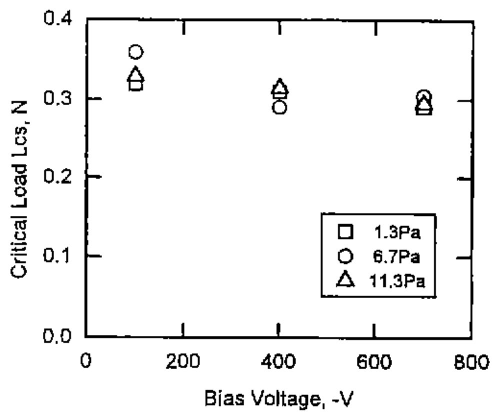

Fig. 9. Relationship between critical load of DLC films and deposition conditions in scratch tests.

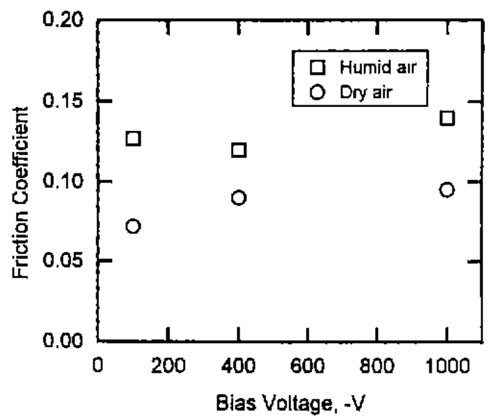

Fig. 11. Relationship between friction coefficient of DLC films deposited at gas pressure of 6.7 Pa and bias voltage.

4. Friction and wear in controlled atmosphere

Friction and wear experiments were conducted in both dry air and humid air (Fig. 10). Irrespective of atmosphere, the friction coefficient decreased with increasing the number of revolutions in the early stage and then attained to a constant value.

Relationship between friction coefficient and negative bias voltage is shown in Fig. 11. Irrespective of bias voltage, the friction coefficient in dry air is considerably lower than that in humid air. Similar results were obtained in DLC films deposited at other CH_4 gas pressures.

Wear scar in dry air is rather little, although it has irregular shape and locally deep valley (Fig. 12). Wear scar in humid air, in contrast, is considerably large, although the shape is smooth. It is interesting that the dependence of DLC film friction and wear on humidity in air is opposite to that of graphite friction and wear.

As above-mentioned, friction and wear of DLC film are intensely sensitive to environmental factors such as humidity in air. However, when DLC films are rubbed in dry air, friction coefficient can attain to a low value of about 0.05. This suggests that DLC film is essentially a very excellent tribomaterial.

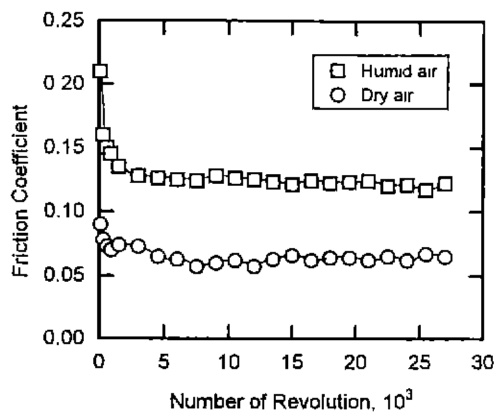

Fig. 10. Relationship between friction coefficient of DLC films deposited at gas pressure of 6.7 Pa and number of revolution.

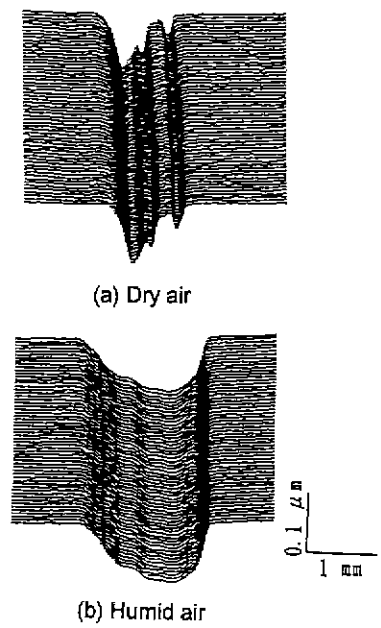

Fig. 12. Wear scar of DLC films.

IV. Conclusions

DLC films were deposited on silicon substrates by using an RF plasma-assisted CVD apparatus; the effects of deposition conditions such as CH_4 gas pressure and substrate bias voltage on DLC film friction and wear were examined in both friction and scratch tests. The following results were obtained.

1. DLC film critical load depends on substrate bias voltages: critical load for films deposited at -100 V is larger than for those deposited at other bias voltages. The critical load is correlated with DLC film hydrogen content in DLC film. CH_4 gas pressure, in contrast, had almost no effect on critical load in the present study.

2. DLC film critical load in scratch tests is also dependent on substrate bias voltage. The dependence of critical loads in scratch tests is considerably smaller than that in friction tests.

3. The friction coefficient of DLC films does not depend on either substrate bias voltage or CH_4 gas pressure.

4. DLC film friction and wear in dry air is considerably lower than that in humid air

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References

1 S. Miyake, S. Takahashi, I. Watanabe and H. Yoshihara, Proc. JSLE Int. Tribology Conf., 407 (1985).
2. K. Miyoshi, J. J. Pouch and S A. Alterovitz, Materials Science Forum. **52/53**.
3. 645 (1989).
4. A. Grill, V. Patel and B. Meyerson, Surface.
5. J. K. Lee, M. Smallen, J. Enguero, H. J. Lee and A. Chao, IEEE Trans. on Magnetics, **29**, 276 (1993).
6. S. Miyake, S. Watanabe, H Miyazaki and M. Murakawa, 2nd Int Conf. on Applications of Diamond Films and Related Materials, 649 (1993).
7. T Yamamoto, K. Seki and M. Takahashi, Surface and Coatings Techn., **62**, 543 (1993)
8. A. K. Gangopadhyay, W. C. Vassell, M. A. Tamor and P. A. Willermet, Trans ASME, J. Tribology, **116**, 454 (1994).
9. S R. P. Silva, A. Kapoor and G A. J. Amaratunga, Surface and Coatings Techn., **73**, 132 (1995)
10. E. Mounier, P. Juliet, E. Quesnel and Y. Pauleau, Surface and Coatings Techn., **76/77**, 548 (1995).
11. D. Yin, N Xu, Z. Liu, Y. Han and X. Zheng, Surface and Coatings Tchn., **78**, 31 (1996).
12. R L C. Wu, K. Miyoshi, R Vuppuladhadium and H E. Jackson, Surface and Coatings Techn., **54/55**, 57 (1992).