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Tribolgical Characteristics of DLC Film using Substrates with Varying Hardness

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Abstract: DLC (Diamond Like Carbon) films have predominant tribological properties like a high hardness, low friction and high chemical resistance; therefore, DLC films are applied in a wide range of industrial fields. This paper evaluated the characteristics of DLC films deposited on bearing steel with different hardness by RF-PECVD (Radio Frequency - Plasma Enhanced Chemical Vapor Deposition) method. Si-interlayer was deposited on bearing steel to improve adhesion strength by RF-Sputtering method. The DLC film structures were analyzed with Raman spectra and Gaussian function. Adhesion strength of DLC films was measured with a scratch tester. Friction and wear test were carried out with a ball-on –disc type to investigate the tribological characteristics. Experimental results showed that DLC films deposited on bearing steel under same deposition condition have typical structure DLC films regardless of hardness of bearing steel. Adhesion strength of DLC film is increased with a hardness of bearing steel. Friction coefficient of DLC film showed lower at the high hardness of bearing steel.

Keywords: *DLC film, adhesion strength, bearing steel, tribology, raman spectra, friction coefficient*

1. Introduction

DLC films have predominant tribological properties like a high hardness, low friction and high chemical resistance. That's why DLC films are used in the variety of industrial field[1-5]. Most reported papers related to tribological problems of DLC films have generally been evaluating and analyzing tribological behaviors of DLC films deposited on Si-wafer[6-9]. Recently the application of DLC film is increasing tendency to steel mechanical components for long service life of tribosystem. Therefore we can find published papers evaluating tribological properties between DLC films and metal[10-17].

However, in view of application of DLC film to steel tribo-components many tribological problems are remained. Bearing steel is mainly used in contact materials of functional machine elements and tribo-components. Recently bearing steel is also applied to tribo-component of fuel pump in severe operating conditions. In this paper we evaluated the tribological characteristics of DLC films deposited on bearing steel.

2. Experimental details

2.1. Coating device

Figure 1 shows the diagram of hybrid coating system used in this study. The Hybrid coating system consisted of a vacuum chamber, a vacuum system, a gas system and a power system. The vacuum system is consisted of a rotary pump and a diffusion pump. The substrate connected to the upper rotation gear with the power system in vacuum chamber. The power system consisted of a power supply which is able to control a bias voltage. Ar gas and C_2H_2 gas are respectively put into vacuum chamber with the device which is applied to MFC (Mss Flow Control) system. Hybrid coating system is operated to create DLC film on bearing steels with different hardness by RF- PECVD method.

2.2. Specimen preparation

We made the three kinds of bearing steels which have a cylindrical shape with 5 mm thickness and 20 mm diameter and different hardness of 200 Hv, 350 Hv and 650 Hv by heat treatment. The bearing steel of 350 Hv was quenched at

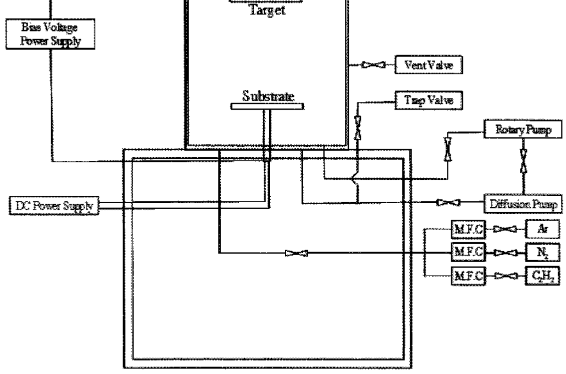

Fig. 1. Schematic diagram of hybrid coating system

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Table 1. The chemical composition of bearing steel

C	Si	Mn	P	S	Cr
0.95~1.10	0.15~0.35	<0.5	<0.25	<0.25	1.3~1.6

800°C(oil cooling) for 1 minute and tempered at 700°C And The bearing steel of 650 Hv was quenched at 800°C(oil cooling) for 5 minutes and tempered at 350°C. The bearing steel of 200 Hv wasn't performed heat treatment. Table 1 shows the chemical composition of bearing steel.

At first the bearing steels were cleaned with ultra-supersonic device for 20 minutes and then put on substrate in vacuum chamber. Si was deposited on the bearing steels by RF-sputtering method to improve the adhesion strength between DLC films and surface of the bearing steels. The DLC films deposited on bearing steels were made to a thickness of 500 nm by controlling the deposition time. We performed a process of Ar - etching under the degree of vacuum 1.2×10^{-2}torr, room temperature, Ar gas 30sccm and RF-bias -600 V for 15 minutes to remove the oxidation layer on bearing steels, followed by a process of interlayer deposition under the degree of vacuum 1.2×10^{-2}torr, room temperature, Ar gas 30sccm and RF-bias -250 V for 5 minutes to improve adhesion strength between DLC films and bearing steels. And then DLC films were deposited on the bearing steels with different hardness by RF-PECVD method under the degree of vacuum 3.0×10^{-2}torr, room temperature, Ar gas 15sccm, C_2H_2 15sccm and RF-bias -300 V for 20 minutes. Table 2 shows the production method and condition and also Figure 2 shows the specimen of bearing steel deposited DLC film. Figure 3 shows AFM images

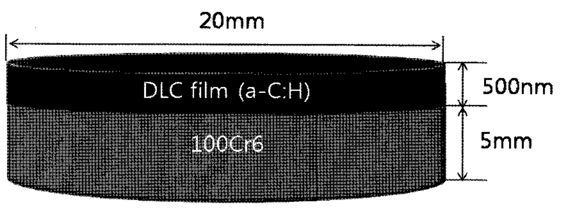

Fig. 2. Dimensions of specimen of bearing steel deposited DLC film.

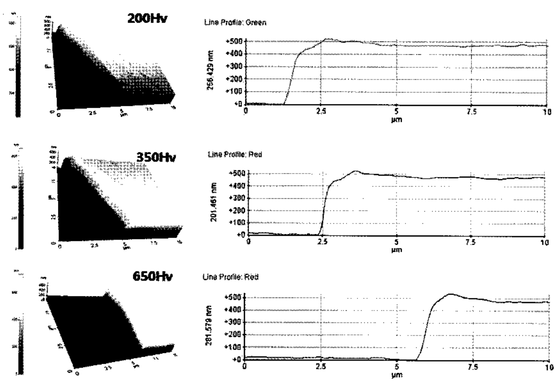

Fig. 3. AFM images showing the thickness of DLC films deposited on bearing steel with different hardness.

Table 2. Process of DLC films deposition.

Process	Working Pressure (torr)	RF-bias(V)	Gas(sccm) Ar	Gas(sccm) C_2H_2	Time (min)	Temp.
Ar-etching	1.2×10^{-2}	-600	30	-	15	Room Temp.
Interlayer	1.2×10^{-2}	-250	30	-	5	
DLC	1.2×10^{-2}	-350	15	15	20	

showing the thickness of DLC films deposited on bearing steel with different hardness.

2.3. Measurements & analysis of DLC films

Raman spectroscopy is a fast and non-destructive method for the characterization of carbon materials. All carbons show common features in their Raman spectra in the 800-2,000 cm^{-1} region, the so-called G and D peaks, which lie at around 1,560 and 1,360 cm^{-1}, respectively for visible excitation. The G peak is due to the bond stretching of all pairs of sp^2 atoms in both rings and chains. The D peak is due to the breathing modes of sp^2 atoms in rings. [18]

Adhesion strength of DLC film on the surface of bearing steel was evaluated by scratch test[19,20].

Scratch test had been proposed by Heavens in 1950s. After, it was theoretically completed by Benjamin and Weaver. Scratch test property is very simple. There is a diamond stylus with fixed radius in Scratch tester. It moves at a fixed speed on straight line, at the same time, a normal load is continually increased on substrate. When the scratch mark made on substrate is investigated with FE-SEM, we can know the critical load(Lc1) by a destroyed coating layer. We used RST S/N: 27-0510 to measure adhesion strength. This equipment can detect acoustic emission which occurs when a coating layer is destroyed.

This study used tribo-meter made by ourselves to measure the friction coefficient of DLC films. Tribo-meter is a wear equipment of ball on disk type and a low load version. It can also perform a wear test under a maximum load of 30N and maximum speed of 600 rpm. The ball is an Al_2O_3, with 3.1 mm diameter. The dimension of the disk is 20mm diameter and 5 mm thickness, and it is bearing steel with different hardness. Three kinds of bearing steel with different hardness (200 Hv, 350 Hv, 650 Hv) were made by heat treatment. Figure 4 shows Friction tester of ball on disk type. We controlled the roughness of disk surface by polishing to make fixed roughness values (Ra 0.01~0.02). Table 3 shows the experimental condition of friction test. And then we observed

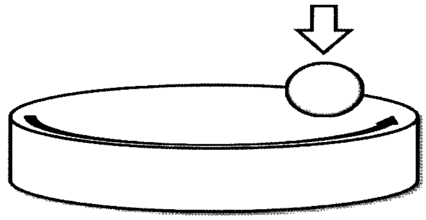

Fig. 4. Friction tester.

Table 3. Experimental condition of friction test.

Condition		Counter body	
Type	Ball on disk	Type	Ball
Normal Load(N)	3	Material	Al_2O_3
Speed(rpm)	100	Radius	3.1

the transfer layer on ball.

3. Experimental Results

3.1. Raman spectra

Figure 5 shows the results of Raman spectra peak. Raman spectra peaks of DLC film have D-peak and G-peak. G-Peak is around 1530 m^{-1} and shows sp^2(graphite-like). D-Peak is around 1350 m^{-1} and shows sp^3(diamond-like). Since DLC films have Sp^2 and Sp^3, we analyzed the structure of DLC films from Raman spectra peak applied to Gaussian function. We analyzed the structure of DLC films deposited on bearing steels with the hardness of 200 Hv, 350 Hv and 650 Hv by Raman spectra peak applied to Gaussian function.

As the results show in Figure 5, the DLC films deposited on bearing steels with different hardness have a typical DLC structure regardless of hardness.

3.2. Adhesion strength

We used Micro-scratch tester to measure and evaluate the adhesion strength between DLC films and bearing steel. Figure 6 shows the adhesion strength due to the hardness of bearing steels with different hardness by Acoustic emission signal graphs.

Adhesion strength test was respectively performed three

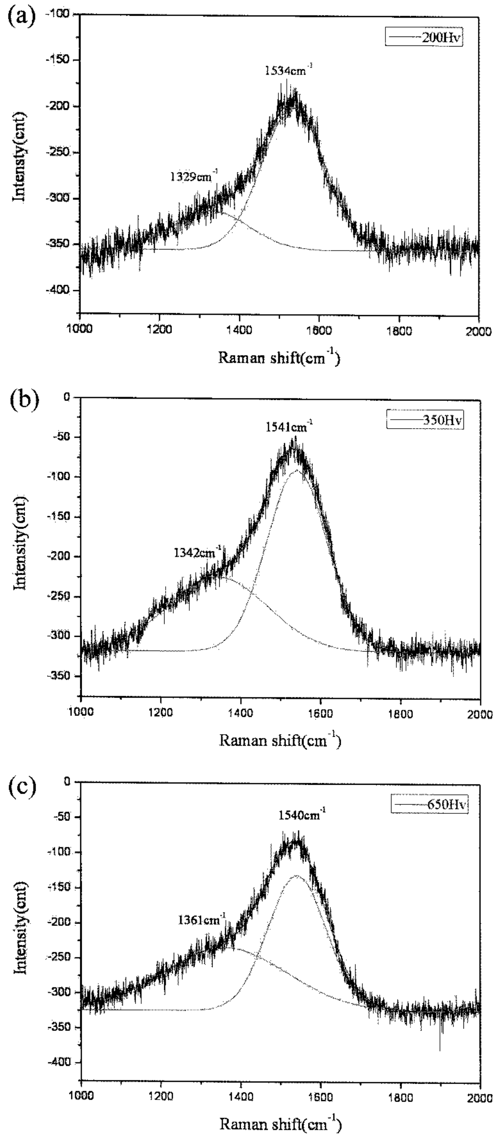

Fig. 5. The Gaussian line shape analysis of the DLC films deposited on bearing steel with hardness of (a)200 Hv, (b)350 Hv and (c)650 Hv.

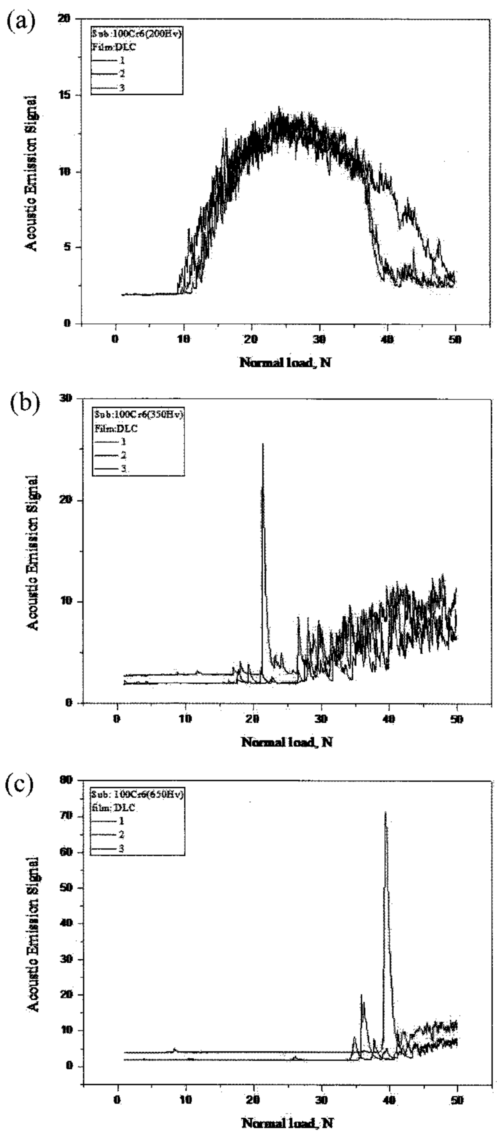

Fig. 6. Acoustic emission graph of scratch at DLC films deposited on bearing steel with hardness of (a)200 Hv, (b)350 Hv and (c)650 Hv.

Table 4. Adhesion strength values of DLC films deposited on bearing steel with different hardness.

Hardness Number	200 Hv	350 Hv	650 Hv
1	10.44 N	26.31 N	33.9 N
2	12.12 N	26.57 N	34.96 N
3	11.22 N	23.83 N	35.86 N

times. Table 4 shows the results of adhesion strength test due to hardness of bearing steel.

The results indicate that when the hardness of bearing steel is higher, the adhesion strength is increases; therefore, although DLC film was deposited under same deposition condition, the adhesion strength between the DLC film and a bearing steel is different according to the hardness of bearing steel.

3.3. Friction coefficient

Figure 7 shows the graph of friction coefficient with sliding time. From the Figure 7, hardness of bearing steel is higher, the friction coefficient is lower. And the friction coefficient of the DLC film deposited on the bearing steel with 650 Hv keeps steady state and very low. Otherwise, the friction coefficient of the DLC film deposited on the bearing steel with 200 Hv and 350 Hv are increasing with sliding time. Some cases, the DLC films were destroyed because of the very thin thickness (500 nm) of the DLC films. And the friction coefficient of bearing steel with 200 Hv is higher than bearing steel with 350 Hv; therefore, as the hardness of bearing steel is lower, the friction coefficient is higher.

Figure 8 shows worn surface and transfer layer on ball due to the hardness of bearing steel. As the hardness of bearing steel is lower, the area of transfer layer is wider. Because, as the area of transfer layer is wider, the area of real contact is wider. therefore, the friction and wear characteristics different due to the hardness of bearing steel.

4. Discussion

Figure 9 shows the elastic deformation of bearing steel and DLC film by bending stress. The elastic deformation is

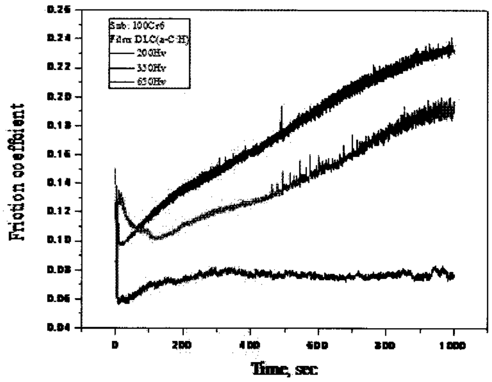

Fig. 7. Friction behaviors of DLC film deposited on bearing steel with different hardness.

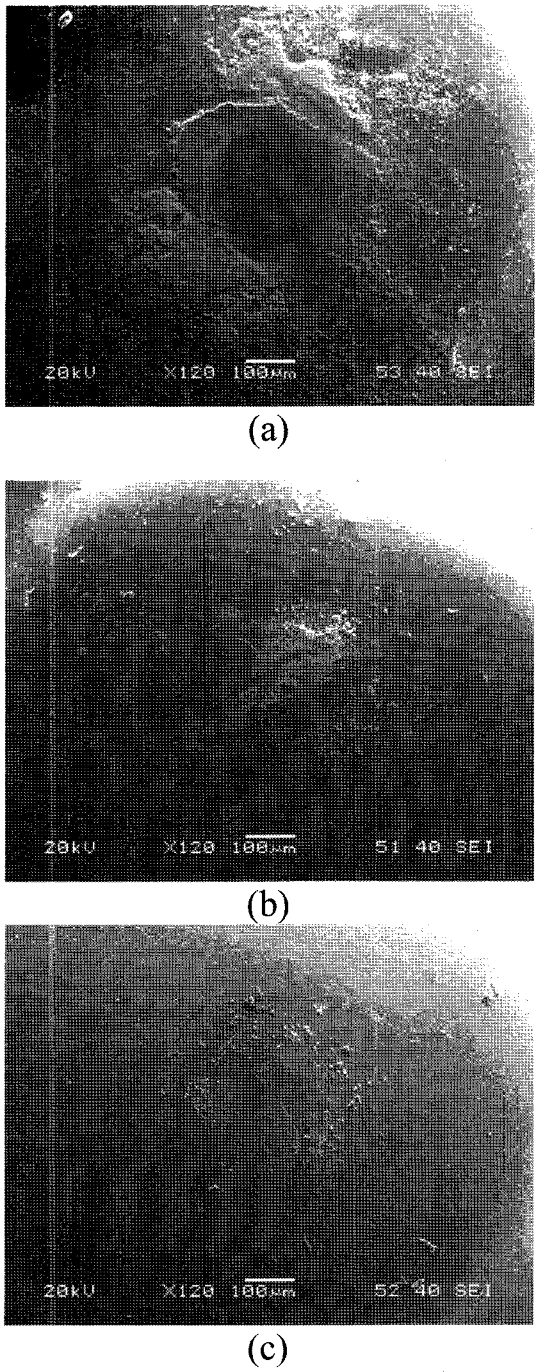

Fig. 8. The images of carbon films transferred from DLC films deposited on bearing steel with hardness of (a) 200 Hv, (b) 350 Hv and (c) 650 Hv on Al_2O_3.

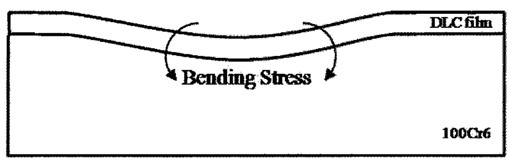

Fig. 9. Schematic illustration of elastic deformation of bearing steel and DLC film by bending stress.

changed due to the hardness of bearing steel. As the hardness of bearing steel is lower, the elastic deformation is bigger. So, the DLC film gives more bending stress. In scratch test, the diamond indenter of scratch tester moves on the surface of DLC film in straight line at the same time. It gets the load regularly increased; therefore, the DLC film deposited on the bearing steel with a low hardness has a low adhesion strength, because it gets much more bending stress than others. Also, the DLC film deposited on the bearing steel with a low hardness has a higher friction coefficient because the area of transfer layer is wider on the ball used as the counter body of it[10]. We thought that the characteristics of DLC film are different by the elastic deformation of substrates.

5. Conclusions

DLC films were deposited on bearing steel with a different hardness under the same deposition condition.

The deposited DLC films have typical structure of DLC films (a-C:H) regardless of hardness of bearing steel. Adhesion strength of DLC film deposited on bearing steel increases with hardness of the bearing steel. Friction coefficient of DLC film shows lower value at the higher hardness of bearing steel.

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