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## Case Study

# Effect of running-in for delamination and friction properties of selfmating diamond-like carbon coatings in water



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## ABSTRACT

Two running-in processes, i.e., polishing a diamond-like carbon (DLC) coated disk and pre-sliding in ambient air, were suggested as a delamination suppression method for self-mating DLC coatings in water, and the effects of these methods were verified using a ball-on-disk tribometer. An a-C:H-type DLC coating was deposited on SUJ2 bearing steel balls and disks using plasma chemical vapor deposition. It was confirmed that delamination of DLC coated disk using the polishing process. The friction coefficient in that condition was reduced to about one-fifth of that of the untreated disk. The running-in process of pre-sliding in ambient air also suppressed delamination of DLC coatings. Moreover, the friction coefficient after this process was reduced to half of that with the other polishing process in the boundary lubrication condition. It was found that these effects have a relationship with surface smoothness and hydrophilicity.

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#### 1. Introduction

Water lubrication is considered one of the most promising ecofriendly technologies because it does not emit  $CO_2$  at the disposal, as does oil lubrication. This is also essential technology for foods and medical devices to prevent oil contamination. However, water has some issues, including low viscosity and oxidation action that hinder its use as a lubricant. In recent years, some studies on the water lubrication of ceramics such as SiC and Si<sub>3</sub>N<sub>4</sub> have been carried out focusing on tribochemical reactions with respect to these issues [1,2].

Diamond-like carbon (DLC) coatings have been considered suitable for water lubrication because they have good tribological properties, including surface flatness, high hardness, high wear resistance, and chemical inertness [3]. Ohana et al. reported that the delamination of DLC coatings occurs in water, in spite of the lower friction coefficient compared with ambient air conditions [4]. We reported the DLC delamination process in water and the formation of a smooth surface without delamination in air by detailed observation, Raman spectroscopy, and nanoindentation of

\* Corresponding author. E-mail address: yasunori\_niiyama@niche.tohoku.ac.jp (Y. Niiyama). wear scar on the ball. It is clear that delamination occurred by the propagation of microcracks generated by contact with droplets formed on the disk during mass production process due to carbon particle deposition. It was also confirmed that the DLC structure changed to an sp2-rich suppressed delamination by preventing the propagation of microcracks [5].

From a practical point of view, it is essential for the utilization of DLC coatings in water lubrication to suppress the delamination. Therefore, the purpose of this study is to verify a method of suppressing delamination of DLC and reducing friction in water. So the following two running-in processes as delamination suppression methods were suggested based on the DLC delamination mechanism and verified using a ball-on-disk tribometer in water.

- 1. Polishing a DLC coated disk: To remove droplets on the disk that generate microcracks, which are the origin of delamination.
- Pre-sliding DLC coated ball and disk in ambient air: To promote a DLC structure change to suppress the propagation of microcracks and form a smoother surface, based on previous our study.

In this study, friction and delamination properties were investigated by tribotests in water and observations of ball wear scars after applying the above two running-in processes. 2

## 2. Experimental procedure

## 2.1. Experimental apparatus

In this study, an inverted ball-on-disk type tribometer which resolution of friction force is 25 mN, was adopted, the same as in our previous study as shown in Fig. 1 [5]. To carry out experiments under water conditions, the ball and disk were submerged in a bath filled with purified water. The sliding speed was controlled by the motor rotation speed and the diameter of the disk wear track and varied between 0.01 and 1 m/s. The normal load varied between 1 and 20 N (initial contact pressure 365–991 MPa) by setting a dead weight and was calibrated using a load cell prior to the experiment. The ball holder that fixed the ball to the contact point was removable and could be positioned with micrometer accuracy. Therefore, the friction test could be restarted at approximately the same contact point while observing the wear process on the ball.

The wear scar on the ball was observed using a confocal microscope (Lasertec Optelics H1200) and a scanning electron microscope (SEM, Hitachi High-Tech-science SU8020) to detect microcracks in the wear scar and delamination.

## 2.2. Specimens

DLC coatings were deposited on a SUJ2 bearing steel disk with a diameter of 30 mm and a hardness of 920HV0.1 kgf that was an average measured at 5 points by nanoindenter (ELIONIX, ENT2100) and on a SUI2 bearing steel ball with a diameter of 8 mm and a similar hardness, using plasma chemical vapor deposition (CVD) in a system made by Tohken Thermo Tech Co., Ltd. The thickness of the DLC coating was approximately 3 µm. The DLC coatings were a-C:H type with a hardness of 2250 HV, which was calculated from the indentation hardness of 23.8 GPa, a Young's modulus of 215 GPa which was also measured by nanoindenter as an average at 5 points, and a hydrogen content of 25% that was measured by the Elastic Recoil Detection Analysis (Kobe Steel, Ltd., HRBS500). The CVD source gas was acetylene. An intermediate layer was formed by the graded layer of W and W-doped DLC, with a thickness of approximately 0.7 µm. The ball and disk specimens had a similar surface roughness, 20-25 nm Rq, while approximately 40 droplets with a height of  $1-5 \mu m$  were found in an area of 300  $\mu$ m  $\times$  300  $\mu$ m, as shown in Fig. 2. Microcracks on the ball wear scar were generated by contacting these droplets, as in the previous study.

Therefore, the disk was polished for 2 h by a lapping machine (Dr. Lap, ML-182, Maruto Instrument Co. Ltd.) using 1.0-um diamond slurry with a load of 4.2 N to remove the droplets on the disk as one running-in process for suppressing delamination. Fig. 3 shows 2D and 3D observations of the polished disk surface made using a confocal microscope. Only two droplets with heights of over 1 µm were detected in the same area as before polishing.

As another running-in process for suppressing delamination, pre-sliding in ambient air was carried out before sliding in water







Fig. 2. Optical images of droplets on the disk: 2D (a) and 3D (b).

according to a previous study in which the DLC structure changed and smooth wear scar was formed on the ball [5]. Pre-sliding condition was a normal load of 10 N, a sliding speed of 0.1 m/s, and a sliding distance of 180 m that was selected as a better condition to promote DLC structure change, to make smooth wear scar and not to wear out DLC coating on the ball. Sliding distance was calculated by multiplying the disk contact perimeter by the total number of disk rotations. Friction behavior, wear scar on the ball, and wear track on the disk are shown in Fig. 4. The friction coefficient increased gradually from 0.05. Wear scar was formed without delamination and cracks on the ball; on the other hand, there were some droplets on the disk wear track, as reported in a previous study. Tribotests were carried out in water using this combination of ball and disk after pre-sliding.

#### 2.3. Raman spectroscopy

The DLC structure change in the ball was investigated by Raman spectroscopy (Tokyo Instruments Inc., Nanofinder30). The laser wavelength applied was 532 nm, and the laser power was 5 mW. The exposure time of sampling was 1 s, and 128 spectra were accumulated. Five different points on the ball were analyzed, and all were within a 400- $\!\mu m$  diameter from the ball contact center.

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