Depth Profile of Oxygen of Diamond-Like Carbon Sliding under Pressurized Hot Water

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The wear mechanism of diamond-like carbon (DLC) in hot, pressurized water has been studied by the oxygen depth profile of DLC films pre and post a sliding test. The sliding test between DLC films coated on chromium molybdenum steel (JIS SCM420) pins and austenitic stainless steel (JIS SUS316) plates was conducted in a water environment by using high temperatures of 100, 200, 250 and 300°C and a high-pressured (10 MPa) autoclave friction tester. The dissolved oxygen concentration (DO) was 0.52 mg/L for oxygen-poor water and 40 mg/L for oxygen-rich water. The experimental results showed that the specific wear rate of DLC was strongly related to the water temperature and the DO concentration. The specific wear rate in both cases of oxygen-poor and oxygen-rich water showed a temperature dependency. When the temperature was below 200°C, the specific wear rate of DLC did not depend on the DO concentration. On the other hand, when the water temperature was more than 200°C, the specific wear rate drastically increased with the DO concentration. In order to prove the effect of oxidation on the wear rate of DLC, the depth profile of the oxygen concentration was compared to that of the carbon concentration on the inside and outside of the wear scar of DLC after sliding tests in hot, pressurized water. When the concentration of DO in the water was low at 0.52 mg/L, the O/C ratio was not affected by the water temperature. On the other hand, when the concentration of DO in water was high at 40 mg/L, the O/C ratio of the top surface increased from 0.05 to 0.18 with an increase in the water temperature on the inside of the wear scar of DLC, where the O/C ratio on the inside of the wear scar was higher than that on the outside of the scar. On the basis of the estimated rate of reaction between DLC and oxygen, determined from a linear relationship between ln (Oxidation wear volume) and the inverse temperature, it was concluded that the oxidation of DLC governed the wear of DLC in hot, pressurized water. Friction was found to enhance the oxidation wear of DLC from the estimated activation energy.

Keywords: DLC, pressurized hot water, wear, oxygen concentration, oxygen depth profile

1. Introduction

Several studies have proved that the presence of hydrogen, oxygen and other environmental species in diamond-like carbon (DLC) testing environments affects the tribological behaviour of DLC wear in water [1-7]. The hydrogen content of DLC coatings shows low friction in inert environments due to very low adhesive forces between the fully hydrogen-terminated sliding surfaces of these carbon coatings which, as proposed by Erdemir [1,3], are not applicable when DLC slides in a water environment.

J. Andersson et al. reported that the friction of hydrogenated DLC under varying water pressure is controlled by the water molecules that are adsorbed onto the DLC surfaces, which could change the interaction to a dipole-like one, increasing the adhesive forces and hence, the friction [5]. Studies conducted by J. Anderson et al. in a high vacuum environment with various added gases also found that the presence of oxygen molecules slightly increased the friction coefficient of hydrogenated carbon films as compared to that in the hydrogen-free environment.
DLC [2], indicating the sensitivity of hydrogenated DLC to water and oxygen.

For the development of metallic hydraulic pumps, valves and cylinders, M. Uchidate studied the effect of environmental parameters namely, load, temperature, dissolved oxygen and water pressure on the tribological properties of a-C:H DLC against stainless steel at 20, 50 and 80°C in distilled water. They reported that the friction and wear of DLC are more dependent on the temperature than on the dissolved oxygen concentration in water (DO) and water pressure. The depth profile of the wear scar by AES revealed that the formation of a beneficial tribofilm, consisting mainly of C, O and Fe, on the steel surface prevented direct contact between DLC and a steel ball. As the temperature increased, the thickness of the C-layer decreased with increments in the O-layer [7].

Other researchers have conducted thermogravimetric analyses (TGA) to measure the critical temperature for oxidation [8-10]. In case of dry oxidation of Si-DLC, a loss in the weight of the Si-DLC was observed when the temperature reached 200°C due to the evaporation of volatile species. The carbon was seen escaping from the film, probably as CO or CO$_2$ gas, while oxygen penetrated into the film during oxidation. It was also reported by Wang Yang Ni et al. that the oxidation of the hydrogenated DLC coating on the Si-wafer started at 440°C where the recorded mass loss was indicative of carbon oxidization owing to the carbon dioxide formation [8].

A previous study explored the possibility of using a-C:H DLC in more severe environments at temperatures ranging from 23 to 300°C and pressures ranging from 0.1 to 30 MPa, in water with high dissolved oxygen (DO) concentration. The DO concentration significantly affected the wear of the sliding a-C:H against SUS316 at 300°C and 10 MPa. By varying the DO concentration in water to 0.73, 16.3 and 47.5 mg/L at 300°C, the wear rate increased 2.9 times from 5.9 to $16.8 \times 10^{-7}$ m/mm/Nm. The wear mechanism of DLC in hot, pressurized water was considered on the basis of a Raman spectroscopy analysis, XPS analysis, AES analysis and AFM scratch test, where a graphitized soft top layer (generated at a water temperature of 300°C after oxidation) was removed [11]. The oxidation was governed by the activation energy, as reported by [12].

Further analysis is needed to have a better understanding of the oxidation resistance of DLC in hot, pressurized water. As hydrogenated DLC is very sensitive to water and/or oxygen, the oxidation resistance varies when the DLC is tested in a dry environment.

This paper studies the effect of friction on the oxidation of DLC in hot, pressurized water with varied concentrations of DO was proposed thereafter.

2. Experiment

2.1. Experimental details

A schematic diagram of the high-temperature and high-pressure autoclave friction tester, designed and fabricated for this study, is shown in Fig. 1. This autoclave chamber had a capacity of 0.6 L, diameter of 80 mm, height of 130 mm, a maximum operating temperature of 300°C and a maximum working pressure of 30 MPa. Figure 2 shows three SCM420 pins coated with DLC as the sliding component arranged in a triangle. SUS316 stainless steel flat plate with a thickness of 1 mm was used as the mating material. The average roughness of the SUS316 stainless steel flat plate surface was 11.2 nm ($R_a$). The curvature radius of the pin head was 1 mm and the pin length was 7 mm. DLC films were deposited onto the SCM420 pins through the CVD deposition process. The thickness of the DLC film, $h$, was approximately 2 μm. The mass of the slider and pins

![Fig. 1 Schematic diagram of high-temperature and high-pressure autoclave friction tester](image1)

![Fig. 2 Photo of (a) DLC pins and SUS316 plate arrangement with base holder and (b) Pin slider and DLC pins dimension in mm. The DLC pin slides in a reciprocating motion](image2)
was 16.9 g and the normal applied load, \( N \) was 0.056 N per pin at rest. Around 7% of difference when the normal load at maximum inclination angle 20° and load with the buoyancy effect. The initial average Hertzian’s contact pressure was 329 MPa. In the chamber, the DLC pin specimen was made to slide on the SUS316 in a reciprocating motion as the autoclave chamber moved like a pendulum driven by an electric motor within a range of 2-20 rpm, as shown in Fig. 3. The inclination angle ranged from −21 to 21°. Figure 3 shows the actual photos of high-temperature and high-pressure autoclave friction tester used for conducting the experiment.

There were two types of experimental environments: oxygen-poor water (0.52 mg/L) and oxygen-rich water (40 mg/L). The concentration of \( DO \) in water was measured by dissolved oxygen meter after a friction test at room temperature and 0.1 atm. The effect of temperature on \( DO \) could not be evaluated as the operating temperature for the \( DO \) meter was around 60°C and this temperature could have altered the \( DO \). The reciprocal sliding wear test was conducted in the autoclave chamber at 20 rpm for around 2700 cycles for 200 m of the sliding distance. The test was executed at 100, 200, 250 and 300°C at 10 MPa of pressurized water. ¾ of the autoclave chamber volume was filled with distilled water. Nitrogen gas (\( N_2 \)) was then inserted into the chamber up to 5 MPa through the inlet pipe. The gas was then bubbled out until 0.1 MPa to prevent ambient air from entering the chamber through the outlet pipe. The outlet valve was closed and the chamber was pressurized with 10 MPa of \( N_2 \) in the oxygen-poor water. The water temperature was controlled by the heater power, according to temperatures shown in Table 1. The excess pressure inside the chamber was controlled by the gas outlet valve.

In case of the oxygen-rich water, oxygen gas (\( O_2 \)) was used for the bubbling process and pressurization. The amount of gas for bubbling and pressurization was the same for both the environments. The amount of \( DO \) was recorded using a \( DO \) meter after each test at room temperature. The amount of \( DO \) was 40 mg/L for the oxygen-rich water and 0.52 mg/L for the oxygen-poor water. Before the sliding tests, the specimens were cleaned thoroughly in an ultrasonic bath with acetone solvent for 15 minutes.

After the friction test, an optical microscope was used to observe the worn surfaces of the DLC film. Assuming that the DLC wear scar was flat, its specific wear rate was calculated from the wear diameter through the optical microscope. Auger electron spectroscopy with argon gas sputtering was used in order to examine the depth profile of the C and O elements in DLC. Atomic force microscope (AFM) nano scratch test was used to evaluate the change in nano hardness on the top surface of DLC after the friction test. The structural properties of DLC after the test were evaluated by X-ray photoelectron spectroscopy (XPS) with monochromated Al-K irradiation at a pass energy of 29.4 eV.

### Wear rate of DLC

The wear volume, \( V \) (mm³) of the DLC film was measured by using the worn width and depth \( H \) (mm) of the DLC pin, as shown in equation (1).

### Table 1 Test conditions

| Pin Specimen              | DLC(2 µm) coated on SCM420 pin (ϕ2 mm) |
|---------------------------|----------------------------------------|
| Plate specimen            | SUS316 plate (60 × 40 × 1) mm          |
| Normal load               | 0.056 N/pin                            |
| Contact pressure          | 329 MPa/pin                            |
| Sliding distance          | 200 m                                  |
| Sliding time              | 130 min                                |
| Environment               | Water                                  |
| Rotation speed            | 20 rpm                                 |
| Temperature and pressure in chamber | 100°C and 10 MPa
| 200°C and 10 MPa          | 250°C and 10 MPa                       |
| 300°C and 10 MPa          | Dissolved oxygen (\( DO \))            |
| Oxygen-poor water         | 0.52 mg/L                              |
| Oxygen-rich water         | 40 mg/L                                |
\[ V = \pi \int_{-\frac{d}{2}}^{\frac{d}{2}} (1 - x^2)^{\frac{1}{2}} dx \]  

(1)

The wear depth, \( H \) (mm) of the DLC pins was calculated by using equation (2),

\[ H = 1 - \sqrt{1 - \left(\frac{d}{2}\right)^2} \]  

(2)

where \( d \) is the wear loss diameter of the DLC pins (mm). The specific wear rate, \( W_s \) (mm\(^3\)/Nm) of the DLC pins was calculated from the Archard wear equation as represented by equation (3), where \( W \) is the applied load (N) and \( L \) is the sliding distance (m).

\[ W_s = \frac{V}{W \cdot L} \]  

(3)

3. Results and discussion

3.1. Surface morphology of DLC pins

Optical images of the worn surfaces of DLC pins after sliding against the SUS316 are shown in Figs. 4 and 5. In case of oxygen-poor water (Fig. 4), the worn surface area did not reveal any significant damage. It was clearly seen that the worn surface was very smooth without any scratch marks on the DLC pin wear scar area at all temperatures. In case of oxygen-rich water (Fig. 5), the worn surface area shows a smooth wear scar except when the temperature reaches 250 and 300°C, where the DLC has peeled off.

3.2. Specific wear rate of DLC

Figure 6 shows the specific wear rate of the DLC pins in oxygen-rich and oxygen-poor water. The rates under both types of water indicated a significant increase with increasing temperature. In case of the oxygen-poor water, the increase in specific wear rate was linearly related to the temperature. The specific wear rate varied in a range of 1.8–4.39×10\(^{-7}\) mm\(^3\)/Nm and it was 1.2–1.1 times lower than that of the oxygen-rich water DLC. Below 200°C, the difference in specific wear rates between both environments was comparatively small. Thus, there was no strong relationship between the amount of oxygen concentration in water and the wear of DLC below 200°C.

As the temperature rose above 200°C, the wear rate grew drastically steep in a linear form. At this stage, the wear rate of DLC was greatly affected by the high amount of dissolved oxygen in water. Thus, it can be concluded that the wear of DLC is extremely sensitive to the concentration of DO in water.

3.3. O/C ratio depth profile

Except for the oxygen-poor water environment, the specific wear rate of DLC had a strong correlation with the depth profile of oxygen against the carbon of DLC films, as shown in Figs. 7 and 8. There was no notable change in the O/C ratio of DLC on the inside and outside of the wear scar except for a slight increase in the O/C ratio at 250 and 300°C when the DO concentration was low at 0.52 mg/L. Thus, the O/C ratio for DLC in

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oxygen-poor water did not have a clear depth dependence through all the temperatures, as shown in Fig. 7. The oxygen depth profile of DLC will be discussed in greater detail in terms of the O/C ratio.

On the other hand, the oxygen concentration in DLC increased with an increase in the water temperature after the sliding test when the DO concentration was high at 40 mg/L. The oxygen concentration on the inside of the wear scar was nearly twice that on the outside of the wear scar of DLC under various water temperatures. On comparing the penetrative depth of oxygen in DLC on the inside and outside of the wear scar, it was found that the oxygen penetrated deeper into the DLC at higher water temperatures, and it was more pronounced when the water temperature rose above 200°C. The penetrative depth of oxygen on the outside of the wear scar increased from 1 to 1.5 min of sputter depth time when the water temperature reached 300°C. On the other hand, the oxygen penetrated deeper into the inside of the DLC wear scar from 2 min of sputter depth time at 200°C to 3 min of sputter depth time at 300°C of water temperature. Thus, it can be said that oxygen penetrates more easily into the inside than the outside of a wear scar of weakened DLC. The layer of DLC that was weakened by friction could have possibly enhanced the oxidation of DLC in hot, pressurized water.

3.4. Tribo-oxidation wear of DLC in hot, pressurized water

From the experimental results, it can be deduced that the concentration of DO in hot, pressurized water has a less significant effect on the specific wear rate of DLC when the DO concentration is low at 0.52 mg/L at all temperatures. As the DO concentration in water is increased up to 40 mg/L, no noticeable differences in the DLC wear rates are found below 200°C. However, a remarkable difference is indicated when the temperature exceeds 200°C for high DO concentrations. A steep linear increment in the wear rate of DLC is observed above 200°C. This could be attributed to the initiation of oxidation wear at 200°C that activates the entry of more
A reaction is believed to have occurred between the DLC and oxygen in hot water to form CO and CO$_2$. This phenomenon can be explained by the Arrhenius equation - predicting the reaction rate constant, $k$ that determines the speed of reaction for a reactant or product dependent on temperature. The rate of chemical reaction is expressed by the following equation, $A$ is the collision frequency, which is determined experimentally; $E_a$ is the activation energy for oxidation wear; $R$ is the gas constant; $T$ is the absolute temperature in Kelvin:

$$k = A \exp\left(-\frac{E_a}{RT}\right)$$  \hspace{1cm} (4)

In order to estimate the $E_a$ of the oxidation wear, $\ln$ (Oxidation wear volume) versus inverse ambient temperature i.e. $1/T$ is plotted. The oxidative wear volume is determined by the difference between the wear volume of oxygen-rich and oxygen-poor water (Fig. 9). The oxidative wear in Fig. 9 is a difference in the wear volume corresponding to the wear loss due to oxygen atoms into the DLC.

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**Fig. 8** Depth profile of O/C ratio on the inside and outside of the wear scar of DLC pin specimens under oxygen-rich water DO: 40 mg/L at (a) 100°C, 10 MPa (b) 200°C, 10 MPa (c) 250°C, 10 MPa (d) 300°C, 10 MPa

**Fig. 9** Wear volume of DLC after friction in oxygen-rich water and oxygen-poor water
the chemical reaction between DLC and oxygen. Figure 10 shows the Arrhenius plot of ln (Oxidation wear volume) against 1/T. The fitted line showed a good linear relation between ln (Oxidation wear volume) and 1/T. Since the slope of the fitted line is equal to −Ea/R, it is possible to estimate the activation energy, Ea, by substituting the R value. The activation energy for the tribo-oxidation reaction in DLC at a high temperature and in pressurized water is 50.6 kJ/mol. From the Ea value, the relationship between the normalised reaction rate constant, k and the temperature, T is obtained by substituting the value of A, R and T in equation (4), as shown in Fig. 11. The value of collision frequency A is determined from the value of interception at −22.118 which equals to ln (A). Therefore, the oxidation wear indicated a good relation with the normalised reaction rate constant, as shown in Fig. 11. The oxidation wear is intensified in the vicinity of 200°C.

Furthermore, the result of the O/C on the top surface of the DLC in oxygen-rich water revealed that friction increased the value of the O/C ratio on the inside of the DLC wear scar when temperature above 200°C, as shown in Fig. 12. This friction enhanced the oxidation reaction in the friction surface where the tribochemical reaction proceeded chemically. The oxygen easily penetrated into the inside of the DLC wear scar as the surface was damaged by friction. J. Xu et al. reported that the activation energy of a chemical reaction at the friction surface is 1/6-1/8th of the static oxidation. Thus, the chemical reaction in the present study strongly enhanced by friction [13]. The increase of the O/C ratio on the inside of DLC wear scar (friction test made between 100 to 300°C) can be explained by the activation energy of oxygen diffusion in DLC. In general, the diffusivity of oxygen atoms in DLC film is greater through less restrictive structural regions. It is believed that the top surface of DLC is damaged during friction in hot, pressurized and oxygen rich water. Figure 13 shows the theoretical values of the diffusion rate constant (D) against temperature to allow diffusion of oxygen into DLC under the said conditions. Low activation energy corresponds to diffusion of oxygen inside the wear scar, and high activation energy is for diffusion at the outside of the wear scar. Taking this information into consideration, we found that friction favors the diffusion rate of oxygen into the DLC film under the said conditions.

3.5. Effect of oxidation on the outer surface of the sliding area

The concentration of oxygen in water plays an important role in oxidation reaction. A higher DO concentration leads to greater mobility of oxygen and deeper penetration into DLC. Introduction of more oxygen in water increases the odds of collision between the C and O atoms with the right orientation to form CO or/and CO2. The volume loss on the outer wear scar was
not as obvious as that on the inside of the scar even for rich DO concentrations. The reaction for oxidation was strongly accelerated by friction where tribo-oxidation activation energy for silicon nitride in water was 1/6-1/8th of static oxidation [13]. It was assumed that the oxidation activation energy for DLC in high temperature and pressurized water on the outer wear scar was larger than that on the inner sliding area. This means that the critical temperature for oxidation on the outer surface of the wear scar could have been larger. In this case, the critical oxidation temperature in the inner sliding area for rich DO was above 200°C. The highest temperature used in this study was not enough to promote visible oxidation at the outer area of the wear scar to cause any significant loss of DLC volume. This is based on the findings using the thermo-gravimetric analysis (TGA) from other researches which revealed that oxidation without friction occurs above 300°C [8,14]. On the basis of thickness reduction from oxidation, the coating thickness did not record any change after 1 hour annealing up to 300°C [14]. This suggests that visible oxidation may not occur up to 300°C at the outer sliding area of DLC.

3.6. Proposed wear mechanism of DLC in hot, pressurized water

Previous researches have proposed several wear mechanisms on DLC at elevated temperatures. M. Uchidate et al. studied a-C:H vs. steel in water from room temperature to 100°C. The authors claimed that a beneficial tribolayer composed of C-rich layer formed on the steel ball prevented direct contact between DLC and steel. However, the beneficial tribolayer thickness reduced as the temperature increased [7]. This led to high wear of DLC in hot, pressurized water due to direct contact between the DLC and the counterpart. Reduction of the beneficial tribolayer was more critical at higher temperatures i.e. above 200°C, possibly due to vapourization of the tribolayer. The tribolayer consisting C, vapourized as gas species when reacting with oxygen and water at higher temperatures.

Another possible mechanism was the degradation of mechanical properties of DLC coating when exposed to high temperatures [9]. It was found that the micro hardness of DLC dropped as annealing temperature increased from room temperature to 300°C, owing to the graphitization and oxidation effects. Nano hardness measurement was carried out using AFM nano scratch on DLC sliding area after wear test at 300°C with different DO concentrations in water at 3000 nN scratch load. Nano hardness values showed clear dependence on the DO concentration at 300°C. The oxygen inside the DLC made the film top layer softer. Softening of the top surface of DLC at high temperatures could be associated to the transformation of sp² C-C to more sp³ C-C. Figure 14 shows the sp²/sp³ ratio of DLC at 300°C with different concentrations of DO in water measured by XPS. This soft layer was easily abraded by the counterpart, resulting in high wear of DLC at high temperatures (Fig. 15).

The mechanism of accelerated wear of DLC in high temperatures and pressurized water may be related to the...
removal of oxidation layer from the top surface of DLC [14]. C-O bonds may be generated on this surface due to the termination of C-C bonds during friction at high temperatures [13]. The amount of C-O bonds could have increased as oxygen penetrated deeper into the DLC film for water with high DO. The soft layer could have been caused by the C-O bonds formed in DLC. As the temperature increased above 200°C, tribo-activation energy, sufficient for soft C-O bonds in solid phase, vapourized as CO or CO₂ [9,13]. The distinct wear difference between low and high DO above 200°C could be attributed to the large amount of soft oxide layer removed from the DLC film when sliding with the counterpart and vapourizing as gas species simultaneously.

4. Conclusion

In this study, the depth profile of oxygen against the carbon of DLC was evaluated by AES, XPS and AFM nano scratch test analyses to understand the effect of friction on the oxidation of DLC under hot, pressurized water. The primary conclusions that can be drawn from the results are as follows:

• The specific wear rate of DLC showed temperature dependency in both low and high DO concentrations in water. Below 200°C, there was no distinct difference in the wear rates between low and high concentrations of DO in water. A slight increase was noted above 200°C for low DO and a steep linear increase of wear rate was found for high DO concentrations in water.

• When the DO concentration was small at 0.52 mg/L, mobility of the oxygen atoms into DLC was very limited even when the water temperature reached 300°C. On the other hand, when the DO concentration was high at 40 mg/L, the amount of oxygen in the DLC increased with the water temperature after the sliding test.

• After comparing the oxygen concentration between the inside and the outside areas of the wear scar under various water temperatures with AES, it was found that the oxygen concentration on the inside of the wear scar of DLC was almost twice that on the outside of its wear scar. It was also found that the oxygen penetrated deeper into the inside area of the wear scar as compared to its outside area at high temperatures. It was easier for the oxygen to penetrate into the inside of the wear scar of the weakened DLC. Hence, the layer of DLC weakened by friction enhanced the oxidation of DLC in hot water.

• The temperature dependency of wear of DLC was almost the same as the temperature dependency of the oxidation wear rate on the basis of the Arrhenius plots. From this result, it can be said that the wear of DLC in hot, pressurized water was governed by the chemical reaction between DLC and oxygen. The increase in wear rate was not only due to graphitization, softening of the topmost DLC layer and transformation of sp³ to sp² carbon, but also due to the oxidation of carbon following the reaction of C + O₂ → CO₂ which was enhanced by the friction.

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