Collecting micrometer-sized wear particles generated between DLC/DLC surfaces under boundary lubrication with an electric field

Takayuki TOKOROYAMA*, Toru KAMIYA**, Nazeef Afifi Bin Hafiz AFMAD* and Noritsugu UMEHARA*

*Department of Mechanical and Aerospace Engineering, Graduate School of Micro-Nano Mechanical Science and Engineering, Nagoya University
Furo-cho, Chikusa-ku, Nagoya-shi, Aichi 464-8603, Japan
E-mail: takayuki.tokoroyama@mae.nagoya-u.ac.jp

**Tribology Section, Material Engineering R&D Department, Research & Development Headquarters, JTEKT Corporation
24-1 Kokubuhiganjo-cho, Kashiwara-shi, Osaka 582-8588, Japan

Received: 21 February 2018; Revised: 31 August 2018; Accepted: 16 December 2018

Abstract
Carbonaceous coatings such as Diamond-Like Carbon (DLC) are attractive candidates for reducing friction under boundary lubrication. However, the wear particles generated from the DLC are believed to form hard slurries that scratch surfaces like an abrasive, and they can shorten the lifetime of the DLC itself. Generally, in conventional automobiles, these wear particles are collected by oil filters. Modern engine bearings are required to reduce the friction coefficient to low levels so that low-viscosity lubricants are used to reduce resistance to flow. However, using a low-viscosity lubricant result in direct solid contact between surfaces, which disrupts the hydrodynamic lubrication and generates numerous wear particles. In this study, we carried out friction tests between DLC/DLC surfaces with kerosene as a boundary lubricant at room temperature. The friction tests were conducted over 12,000 cycles, and we replaced the lubricant after every 2000 cycles without changing the contact pair. Wear particles were collected from the lubricant using an electric field with the electrophoresis effect. The wear particles gathered near the positive electrode and fell onto a glass plate. Finally, after the kerosene evaporated, scanning electron microscope and laser optical microscope observations showed the relations between particle size and shape, the number of wear particles, and the friction coefficient.

Keywords: DLC, Boundary lubrication, Wear particles, Electric field collecting system, SEM, Laser optical microscope

1. Introduction

Mechanical parts that slide or roll against other parts should do so with low friction and should have a long life time. For transportation to achieve low energy consumption, reducing the friction coefficient between various mechanical parts in the engine, transmission, gears, and bearings is important. Carbonaceous coating such as Diamond-Like Carbon (DLC) are good candidates for reducing the friction coefficient in automobiles, since they have high hardness and a low friction coefficient (Kano et. al., 2005; Tasdemir et. al., 2013; Erdemir et. al., 2000; Donnet and Erdemir, 2004; Fontaine et. al., 2004). For sliding under lubrication, the gap between the two surfaces is determined by the surface roughness, normal load, sliding velocity, and the viscosity and temperature of the lubricant (Bowden and Tabor, 2001). Modern engine bearings are required to reduce the friction coefficient to low levels, so low-viscosity lubricants are used to reduce resistance to flow. However, using a low-viscosity lubricant result in direct solid contact between surfaces, which can disrupt the hydrodynamic lubrication and generate numerous wear particles. The particles are assumed to be abrasive; therefore, it is better to eliminate them from the contact region and from oil reservoirs. In general, these particles are thought to accelerate wear since they can scratch the surfaces, causing oil starvation by particle stagnation. In an automobile, it is common to trap the particles in an oil filter. The pore diameters of a conventional filter are around 50
μm. Although particles smaller than 50 μm are thought to accelerate wear in automobiles, using an oil filter that can trap such small particles causes a deterioration in performance because of the high resistance to flow due to the small pores. If the oil filter becomes damaged by the particles, oil passes through it, and it does not work anymore. Conventional ferrography cannot collect wear particles from nonmagnetic materials. Therefore, it is necessary to use another collecting method. In a previous work (Ohara et al., 2014), we found a major problem in using a conventional paper filter to trap wear particles with a diameter of approximately 1 μm: it was difficult to distinguish between powder from the paper and wear particles on the paper filter when we observed it using a scanning electron microscope (SEM). In this work, to avoid this problem, we tried to collect the wear particles that were smaller than 10 μm using a collecting system with an electric field. It has been confirmed that the diamond particles from a dispersion fluid can be moved by an AC electric field (Akagami and Umehara, 2006). After several friction tests with DLC/DLC contacts and a lubricant, we applied an electric field between positive and earth electrodes to collect wear particles. We classified them using a SEM and a laser optical microscope. The SEM can reveal the details of the surface features of the wear particles, such as the shape and size, and the laser optical microscope can be used to determine the volume. We investigated the relation between the shape and size of the wear particles and the friction coefficient of the DLC/DLC contact.

2. Experimental

2.1. Trapping wear particles

Wear particles were collected at six different time points during the friction test, which is described in more detail in Section 2.2. They were generated from the contact point of DLC/DLC sliding under boundary lubrication. It was hypothesized that all of the particles were generated from the contact point. Therefore, all of the collected particles consisted of only DLC. The wear particles produced under a lubricant are assumed to be charged positively or negatively. A positive electrode and an earth electrode were immersed in the lubricant, which was kerosene, to collect the particles. The particles should move toward the electrodes by electrophoresis (Doh and Cho, 2005). A schematic of the electric field collection system is shown in Fig. 1. The maximum applied voltage was 1.0 kV. The kerosene lubricant was then washed away with acetone. We observed the particles using a laser optical microscope. Finally, a thin coating of osmium oxide was applied onto the glass plate to allow us to observe the surface of the particles using SEM.

![Fig. 1 Schematic of the apparatus used to collect wear particles from the lubricant. The positive and earth electrode were held in place with insulators. The wear particles gathered near the positive electrode. When the voltage was switched off, the particles fell onto the glass plate. Finally, the kerosene lubricant was allowed to evaporate.](image)

2.2. Cylinder-on-a-disk friction tests and lubricant replacement

The friction test with a cylinder-on-a-disk (Fig. 2) was carried out under kerosene at room temperature. The viscosity of the kerosene was 1.7×10^−3 Pa·s at 23 °C. The normal load was approximately 10 N and applied using a dead weight to produce approximately 208 MPa of average Hertzian contact pressure. The sliding speed was 125.7 mm/s. From the Hamrock and Dowson equations, the λ (lambda) ratio was calculated to be 0.026, which is lower than 1. Therefore, this was clearly a boundary lubrication condition. The cylinder and the disk were coated by ion-plating-type DLC on bearing steel SUJ2 substrates. The diameter and length of the cylinder were both approximately 5.0 mm. The diameter of the disk was approximately 22.5 mm, and its thickness was 4.0 mm. Each coating were deposited by physical vapor deposition;
therefore, those coatings did not include hydrogen. The indentation hardness was approximately $30.0 \pm 5.0$ GPa.

The experimental procedure is shown in Fig. 3. The friction test run for 2000 cycles using a DLC-coated cylinder sliding against a DLC-coated disk, as shown in Figs. 3(a) and (b). After every 2000 cycles, the friction test was stopped, and all of the lubricant was poured out of the oil bath for testing. The lubricant was replaced, the cylinder and the disk specimen were reset in their initial positions, and we restarted the friction test. In total, there were 12,000 cycles, Figs. 3-(c) and (d)).

![Schematic of the cylinder-on-a-disk friction test](image1)

**Fig. 2** Schematic of the cylinder-on-a-disk friction test. (a) Top view, (b) side view, and (c) enlargement of the cylinder specimen and its holder sliding against the disk. The cylinder and disk were coated with DLC. The friction tests were finished before the substrate appeared; therefore, all wear particles were hypothesized to be DLC. The friction test was conducted with kerosene as a boundary lubricant at room temperature. The normal load was a dead weight. The friction force was measured by the load cell as strain that was converted to a voltage that was recorded on a PC.

![Schematics of the experimental procedure](image2)

**Fig. 3** Schematics of the experimental procedure. (a) Before the friction test, (b) cylinder-on-a-disk friction test under lubrication, (c) after 2000 test cycles, and (d) cylinder-on-a-disk test continued from the initial test position. The lubricant was replaced every 2000 cycles. New kerosene was added to the oil bath, and the friction test was continued for the next 2000 cycles. The friction test was repeated six times for a total of 12000 cycles.

### 2.3. Classification of wear particles using a laser optical microscope

Before measuring the size and amount of wear particles on the glass plate, it was necessary to verify that the laser optical microscope was able to measure the volume of wear particles. For this, we used glass beads, as shown in Fig. 4(a), which were observed by the laser optical microscope. Each glass bead was numbered, and we measured its diameter, as shown in Fig. 4(b). The volume $V_{\text{cal}}$ was calculated geometrically. In addition, we measured the volume $V_{\text{auto}}$ of each glass bead from the laser optical microscope image using software. The two volumes differ because the software includes
the additional volume $V_{\text{add}}$ shown in Fig. 4(c). To obtain real volume of the glass beads, the additional volume was subtracted. The volume calculated from the diameter agreed with the volume calculated by the software, as shown in Table 1. Hence, we could apply the same procedure to obtain the volume of the wear particles.

![Image of glass beads and glass plate](image.png)

**Fig. 4** (a) Laser optical microscope image of glass beads (top view). (b) The diameter $D$ of a glass bead. (c) The additional volume $V_{\text{add}}$ measured by the laser optical microscope (side view). The glass beads were hypothesized to be spherical. The laser optical microscope observation was conducted from above the glass plate; therefore, volume $V_{\text{add}}$ was included because it was under the sphere. The additional volume was subtracted from the measured values to give real volume of glass beads.

| No. | Diameter $D$, $\mu$m | Calculated volume from diameter $V_{\text{cal}}$, $\mu$m$^3$ | Estimated volume by laser microscope $V_{\text{est}}$, $\mu$m$^3$ | Additional volume $V_{\text{add}}$, $\mu$m$^3$ | Real volume $V_{\text{real}}$, $\mu$m$^3$ |
|-----|---------------------|-------------------------------------------------|-----------------------------|-------------------|------------------|
| 1   | 35.74               | 23903.5                                        | 29880.1                     | 5975.9            | 23904.1          |
| 2   | 14.43               | 1573.2                                         | 1966.8                      | 393.3             | 1573.5           |

The resolution of the laser optical microscope was approximately 0.1 $\mu$m in the horizontal and vertical directions with regard to the observation direction and approximately 0.01 $\mu$m in depth (observation direction). To clarify the effect of the size and amount of wear particles, we classified them by diameter as 1–4 $\mu$m, 5–9 $\mu$m, and larger than 10 $\mu$m.

### 3. Results

The friction test between the DLC cylinder and DLC disk was carried out for 12,000 cycles, as shown in Fig. 5. Wear particles were collected every 2000 cycles. The surface roughness before the test and after 12,000 cycles was observed by atomic force microscopy, as shown in Figs. 6(a) and (b). The surface roughness values of the DLC-coated disk were 4.6 nm Ra and 22 nm Rz before the test. The surface roughness values decreased a little to 3.5 nm Ra and 20 nm Rz. The observations indicated clearly that $A$ did not significantly change. Representative SEM images of particles are shown in Figs. 7(a)–(d). After 2000 cycles, many particles with sharp and angular edges were observed, as shown in Fig. 7(a). It was assumed that these particles quickly peeled off from the topmost surface because they had sharp edges...
and were no longer part of the contact area. After 6000 cycles, few wear particles larger than 10 μm were observed. The predominant particles were of size 5–9 μm. A representative SEM picture is shown in Fig. 7(b). The edges are dull, unlike those generated at the beginning of the friction test. After 12,000 cycles, there were still wear particles of size 1–4 and 5–9 μm. It was assumed that

4. Discussion

From the friction test results and SEM and laser optical microscope images of DLC wear particles, we can consider how they were created. A schematic of the generation of wear particles is shown in Fig. 9. During the first 2000 cycles, wear particles larger than 10 μm were generated from the surfaces. Those particles dispersed into the lubricant without entering the contact area. This period was assumed to be the running-in of the DLC surfaces. After 2000 and 4000 cycles, the predominant size was 5–9 μm. It was hypothesized that these particles were in the contact area; hence, they suffered cyclic pressing and cyclic friction seizure (as shown schematically in Fig. 9). Therefore, the friction coefficient varied significantly. The tendency for these particles to re-enter the contact area continued for 6000 cycles. After 6000 cycles, there were still DLC wear particles larger than 10 μm; however, the amount decreased gradually with the number of sliding cycles. By contrast, there were still wear particles of size 1–4 and 5–9 μm after 6000 cycles. It was assumed that
wear particles larger than 10 \( \mu \)m were accumulations of small wear particles. Figure 9(d) shows a possibility for a contact point. The surface of the wear particle has a linear sharp edge, as shown in the schematic. After 6000 cycles, the amplitude of the friction coefficient oscillations dwindled because there were few wear particles in the lubricant and the predominant size was smaller than 10 \( \mu \)m.

Fig. 7 SEM images of DLC wear particles. Particles obtained after (a) 2000 cycles, (b) 6000 cycles, and (c) 12,000 cycles. (d) Enlargement of (b). The wear particles consist of small pieces of DLC debris. The small specks distributed over the glass plate in (d) are from the osmium coating. The specks have diameter of approximately 0.1 \( \mu \)m or less.

Fig. 8 (a) Total volume of wear particles versus the number of sliding cycles. (b) Distribution of wear particle size versus number of sliding cycles \( N \). At the beginning of the friction test, most wear particles were larger than 10 \( \mu \)m. The number of large particles dwindled with the number of sliding cycles. The volume of the large particles decreased by 0.2 times compared to the beginning. The number of wear particles of size 1–4 and 5–9 \( \mu \)m decreased to nearly zero after 4000–12,000 cycles.
5. Conclusions

To clarify the relation between wear particles’ size, amount of the particles and friction coefficient, we established an electric voltage applying system to collect wear particles that were smaller than 10 μm by electrophoresis effect. Laser observation was carried out to confirm the equipment was able to measure particles’ volume. After 12000 cycles friction test with replacing the lubricant in each 2000 cycles at room temperature under kerosene lubricant was conducted to obtain wear particles of DLC cylinder/DLC disk contact. Following conclusions were obtained.

1) The relation between friction coefficient and wear particles’ size, shape and amount was investigated. At the beginning of friction during 0-2000 cycles between the DLC cylinder/DLC disk contacts, friction coefficient varied largely. Wear particles were larger than 10 μm was observed as the predominant of all wear particles. Those particles had line edge. In the case of 6000-8000 cycles, the wear particles larger than 10 μm little observed, on the other hand, 5-9 μm wear particles became predominant of all particles.

2) One of the representative wear particle showed that it experienced several seizure or cyclic friction under pressing at the contact point. The wear particle had line like sharp edges that was assumed to be built by several wear particles.

References
Akagami, Y. and Umehara, N., Development of electrically controlled polishing with dispersion type ER fluid under AC electric field, Wear, Vol. 260 (2006), pp. 345–350.
Bowden, F.P. and Tabor, D., The friction and lubrication of solids, Oxford University Press (2001), p. 223.
Donnet, C. and Erdemir, A., Histrical developments and new trends in tribological and solid lubricant coatings, Surface and Coatings Technology, Vol. 180-181 (2004), pp. 76–84.
Erdemir, A., Eryilmaz, O.L., Nilufer, I.B. and Fenske, G.R., Synthesis of superlow-friction carbon films from highly hydrogenated methane plasmas, Surface and Coatings Technology, Vol. 133-134 (2000), pp. 448–454.
Fontaine, J., Loubet, J.L., Le Mogne, T. and Grill, A., Superlow friction of diamond-like carbon films: a relation to viscoplastic properties, Tribology Letters, Vol. 17, No. 4 (2004), pp. 709–714.
Doh, I. and Cho, Y.-H., A continuous cell separation chip using hydrodynamic dielectrophoresis (DEP) process, Sensors and Actuators A, Vol. 121 (2005), pp. 59-65.
Kano, M., Yasuda, Y., Okamoto, Y., Mabuchi, Y., Hamada, T., Ueno, T., Ye, J., Konishi, S., Takashima, S., Martin, J.M., De Barros Bouchet M.I. and Le Mogne T., Ultralow friction of DLC in presence of glycerol mono-oleate (GMO), Tribology Letters, Vol. 18, No. 2 (2005), pp. 245–251.
Ohara, K., Tokoroyama, T., Kousaka, H. and Umehara, N., Analysis Method of Wear Particles from DLC Sliding against

Fig. 9 The schematic images of wear particles sliding between surfaces, (a) large particles entering into contact point and those particles were seized and pressed repeatedly. Several cracks generated to wear particles, then those particles gathered. (b) The gathered wear particles seized between two surfaces, and it were aggregated. Finally, (c) seized particles distributed to the lubricant. (d) The enlargement of seized evidence on a particle from cross-sectional view point. There were line like edges.
Itself under Oil Lubrication, The Journal of Japanese Society of Tribologists, Vol. 59, No. 7 (2014), pp.429–436 (in Japanese).

Tasdemir, H.A., Wakayama, M., Tokoroyama, T., Kousaka, H., Umehara, N., Mabuchi, Y. and Higuchi, T. Ultra-low friction of tetrahedral amorphous diamond-like carbon (ta-DLC) under boundary lubrication in poly alpha-olefin (PAO) with additives, Tribology International, Vol. 65 (2013), pp.286–294.