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Tribological Properties of Steel/Steel, Steel/DLC and DLC/DLC Contacts in the Presence of Biodegradable Oil

Abdul MANNAN, Mohd Faizul Mohd SABRI⁎, Md. Abul KALAM, and Haji Hassan MASJUKI

Faculty of Engineering, University of Malaya, Kuala Lumpur 50603, MALAYSIA

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Tribological properties of tetrahedral diamond-like carbon (ta-C DLC) coating were investigated to assess the effects of the counter surfaces under the boundary condition in the presence of biodegradable oil. The tribological tests were performed using steel/steel, steel/DLC and DLC/DLC contacts, and found that the coefficient of friction was 0.062, 0.059 and 0.048, and the wear loss was $5.57 \times 10^{-4}$, $9.81 \times 10^{-4}$ and $6.02 \times 10^{-4}$ mm$^3$, respectively. DLC/DLC contacts showed the lowest friction coefficient and steel/steel contacts showed the lowest wear. The high wear of steel/DLC compared to the other two contacts might be due to the high affinity between iron and carbon. The high content of oleic acid may maintain the low friction in self-mated ta-C DLC contacts and tribocchemical wear was prevented by nano-scale graphitization. Therefore, material combinations are important for tribological properties and canola oil is an effective lubricant for contacts coated with ta-C DLC films.

Keywords
Friction, Wear, DLC coating, Vegetable oil, Four ball tribometer

1. Introduction

Diamond like carbon (DLC) coating is defined as the metastable form of amorphous carbon materials consisting of carbon atoms with both sp$^2$ and sp$^3$ bonding$^1$. DLC coating chemically inert and has excellent tribological and mechanical properties very suitable for bearings, gears, piston rings and pins, cutting tools, hydraulic components, etc.$^2$–$^8$. DLC coating shows good tribological properties under dry conditions$^9$, but generally lubricated with the liquid lubricants for further improvements of the tribological properties. In addition, the liquid lubricants have some other functions such as cooling the system, increasing the durability, and removing debris particles, and others$^{10}$, which cannot be achieved by the dry lubrication. In practice, friction at a contact should be as low as possible, because large amount of input energy are wasted to overcome friction such as approximately 30 % of fuel energy in an automobile engine$^{11}$. Therefore, the tribological performance of DLC coating in the presence of liquid oils is very important to establish.

Mineral-oil-based lubricants are the most widely used type of lubricants worldwide but are highly toxic and non-biodegradable$^{12}$. The waste lubricants contain toxic hydrocarbons and some heavy metals which pose persistent threats to health and the environment. Social concern is very high now about the preservation of the environment and protection of life, so lubricants with good tribological properties without harmful effects on the environment and health are highly desirable$^{13,14}$. Vegetable oils may be the best substitutes for mineral-oil-based lubricants since these lubricants are highly biodegradable and very low friction as well as low wear$^{13,15}$. The present study investigated canola oil as the biodegradable oil because of its availability.

The tribological properties under the boundary condition vary greatly with the contacts materials$^{16}$, so study of different material combinations is important. A previous study investigated canola oil with and without boric acid at the boundary condition using a pin-on-disk tribotester, with a DIN 1.2090 pin and AISI 8620 disk$^{17}$. Another study examined the tribological properties of several vegetable oils including canola using a pin-on-disk tribometer$^{15}$ where copper hemispherical pin was rubbed against aluminum disk. DLC coatings are widely used for practical applications so evaluation of the tribological performance of DLC coatings with vegetable oil is very important for the future development of industrial lubricants.

The present study investigated the tribological properties of tetrahedral diamond-like carbon coating (ta-C DLC) in steel/DLC and DLC/DLC contacts under the boundary condition, and steel/steel contact as the refer-
Boundary lubrication can be defined as lubrication by a liquid under conditions in which the solid surfaces are so close together that appreciable contact between the opposing asperities is possible. The friction and wear under boundary lubrication are determined predominantly by the interactions between the solids and between the solids and the liquid. The bulk flow properties of the liquid have little or no effect on the friction and wear behavior. Appendix A shows a typical schematic diagram to illustrate the boundary lubrication condition.

### 2. Experimental Details

#### 2.1. Preparation of ta-C DLC Coating

The substrate material was AISI 440C steel ball of 12.7 mm diameter, which widely used in the manufacturing of bearing and hydraulic components in which mechanical interactions of surfaces are very common\(^{(18,19)}\). The minor chemical components (wt%) of the steel ball were S-0.03, P-0.035, Mn-0.72, Si-0.8, C-0.96, and Cr-17.8. The base canola oil had kinematic viscosity of 35.606 mm\(^2\)/s and density of 0.9031 gm/cm\(^3\) at 40°C. ta-C DLC films of ~1 \(\mu\)m thickness were deposited on AISI 440C steel balls using the Filtered Cathodic Vacuum Arc (FCVA) technique from My Tech Seimitsu Sdn. Bhd. (Malaysia). Deposition was performed at 80°C under the chamber pressure of 1 \(\times\) 10\(^{-5}\) Torr (1 Torr = 133.322 Pa). Absence of adequate adhesion between the coating and substrate is the main reason for premature failure of PVD coatings\(^{(20)}\). In this study, a Cr based interlayer was provided on the substrate surface prior to deposition of the DLC coating to increase the coating-substrate adhesion. The physical properties of the steel ball and ta-C DLC films are given in Table 1.

#### 2.2. Measurement of Fatty Acid Composition

Fatty acids are the long hydrocarbon chains with a carboxyl group (-COOH) at one end (reactive portion of the molecule). Fatty acids can be saturated (carbon-carbon single bond) and/or unsaturated (carbon-carbon double bonds). The fatty acid composition of canola oil was measured by the gas chromatography-mass spectrometry (GC-MS) was highly unsaturated as shown in Table 2. The content of oleic acid was very high among all the unsaturated molecules, see Table 2.

#### 2.3. Fourier Transform Infrared (FTIR) Analysis

FTIR analysis of canola oil was performed with a Perkin-Elmer Spectrum 400 FTIR analyzer equipped with a mid-infrared (MIR) detector in the range of 4000 to 450 cm\(^{-1}\). The equipment was coupled with a standard KBr beam splitter and a deuterated triglycine sulfate (DTGS) detector. The spectra were recorded at 4 cm\(^{-1}\) resolution. The crystal plate was cleaned with toluene and 0.4 mL of sample was placed. The FTIR spectrum of canola oil is shown in Fig. 1. The peak at 1743 cm\(^{-1}\) was due to the ester carbonyl groups (C = O) at 1651 cm\(^{-1}\) to the unsaturated bonds (C=C)\(^{(21)}\).

#### 2.4. Friction and Wear Tests

Tribology measurements used a four ball tribometer as shown in Fig. 2. Prior to each test, all key components (i.e. pot, clamp ring and holders) of the tribometer and the ball samples were cleaned ultrasonically in an acetone bath for 10 min. The sample set-up in the four

| Common name | Lipid number | Saturated | Unsaturated | Arachidic acid |
|-------------|--------------|-----------|-------------|---------------|
|             | wt [%]       | 16 : 0    | 18 : 0      | 18 : 1        | 18 : 2        | 18 : 3        | 20 : 0        |
| Palmitic acid | 4           | 18       | 56          | 26            | 10            | 2             |
| Stearic acid  | 2           | 18       | 56          | 26            | 10            | 2             |
| Oleic acid    | 56          | 56       | 56          | 26            | 10            | 2             |
| Linoleic acid | 26          | 26       | 26          | 26            | 10            | 2             |
| Linolenic acid| 10           | 10       | 10          | 26            | 10            | 2             |
| Arachidic acid| 2            | 2        | 2           | 2             | 2             | 2             |
ball tribometer clamped three balls in the lower stationary cup and the fourth ball in the upper rotating chuck. The DLC coated balls were clamped in the lower stationary cup and the steel ball was clamped in the upper rotating chuck to test steel/DLC contacts. The parameters used in the tribology tests are given in Table 2. All tribology tests were repeated three times without changing the test parameters. The Tallian parameter “lambda (λ)” was calculated to determine the lubrication regimes using Eq. (1)\(^{22,23}\).

\[
\lambda = \frac{h_0}{\sqrt{R_{q1}^2 + R_{q2}^2}}
\]  

(1)

Where, \(h_0\) is the minimum oil-film thickness (\(\mu m\)) and \(R_{q1}, R_{q2}\) are the surface roughness of the lower stationary balls and the upper rotating ball in \(\mu m\) respectively. The calculated \(\lambda\) value for all cases was less than unity (see Table 3). Therefore, the system was working under the boundary condition.

The four ball tribometer was interfaced with a computer to measure the friction torque (\(T\)) which was converted into the friction coefficient using Eq. (2)\(^{24}\). In addition, the wear scar diameter (WSD) was measured with an optical microscope (OM) before removal from the cup, and the WSD was converted to wear volume using Eq. (3).

Friction coefficient, \(\mu = \frac{T\sqrt{6}}{3W_f}\)  

(2)

Wear volume, \(V = \frac{1}{6} \pi h [3r^2 + h^2]\)  

(3)

Where \(T\) is the friction torque in kg mm and \(W\) is the applied load in kg.

2.5. Surface Analysis

The worn surfaces were investigated with a field emission scanning electron microscope (FESEM) and an electron dispersive X-ray spectroscopy (EDX) at \(2000\times\) magnification and 5 kV accelerating voltages. In addition, the fresh and rubbed DLC surfaces were studied with Raman spectroscopy to detect any graphitization. The Raman analysis used diode laser wavelength-514 nm, output power 10 % and 50 \(\times\) objective lens. In this study, the output power was set at 10 % based on the previous study showing that this power range can isolate the peak from the outermost surfaces without influence of the bulk substrate on the measurements\(^{25}\).

3. Results

3.1. Friction Results

Figure 3 presents the coefficient of friction (COF) of all material combinations (steel/steel, steel/DLC and DLC/DLC) against time and Fig. 4 shows the average coefficient of friction. Figure 4 clearly indicates that the friction coefficient was highest for the steel/steel contact (0.062) and lowest for the DLC/DLC contact (0.048). The steel/DLC contact (0.059) showed higher COF than the DLC/DLC contact but lower than the steel/steel contact. Figure 3 shows that the friction coefficient at the beginning was very low for all three contacts, which is known as the run-in phase and results from the pre-load condition. The reasons behind the sudden increase of friction coefficient will be explained in the discussion section. Briefly, under the boundary

| Types of contact | Temperature [°C] | Load [N] | Time [s] | Velocity [m/s] | Lambda [\(\lambda\)] |
|------------------|------------------|----------|----------|----------------|---------------------|
| DLC/DLC          | 30               | 100      | 3600     | 0.46           | 0.39                |
| Steel/DLC        |                  |          |          |                | 0.32                |
| Steel/Steel      |                  |          |          |                | 0.27                |

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condition, the wear and friction mainly depend on the properties of the surface asperities and the lubricant film thickness has only minor effects on the friction and wear characteristics. For the steel/steel surface, the load was mainly carried by surface asperity contact in the elastic deformation mode so the friction coefficient was higher than for the DLC surfaces, because the asperity height was much smaller for DLC surfaces than for steel surfaces (see Table 1). DLC is hard and with low load (only 100 Nm), the DLC/DLC contact is very low. Therefore, the DLC/DLC surface is suitable for low loads at low-temperature. For the steel/DLC surface, the asperity of the steel surface might damage the DLC surface, especially under the boundary condition, because the DLC surface does not undergo elastic deformation but will be scratched by the steel surface asperity. Therefore, although the friction coefficient of steel/DLC is lower than steel/steel, wear will increase abnormally as shown in Fig. 5.

3.2 Wear Results

The wear losses of steel/steel, steel/DLC and DLC/DLC contacts lubricated with base oil were $5.57 \times 10^{-4}$, $9.81 \times 10^{-4}$ and $6.02 \times 10^{-4}$ mm$^3$, respectively (Fig. 5) measured as the average of the three stationary balls.

Wear loss in DLC/DLC contact was comparable with the wear loss for the steel/steel contact, but the wear loss for the steel/DLC contact was relatively very high at 63 % higher wear than the DLC/DLC contact. Therefore, the performance of the DLC coating significantly depends on the counterpart surface.

The wear mechanism for boundary lubrication using canola oil can be explained as follows: Section 3.1 explained that wear and friction at the steel/steel surface under the boundary lubricated condition mainly depends on the asperity properties and less on the lubricant characteristics. Wear at the DLC/DLC contact, mainly depends on the lubricant properties of canola oil, despite the minimum film thickness, as the asperity effect is negligible due to the DLC coatings.

Canola oil consists of several types of fatty acids with various structures containing only carbon, hydrogen and oxygen atoms. Canola oil is especially high in unsaturated fatty acids, including oleic acid ($C_{18}H_{34}O_2$) 56 % with a single carbon–carbon double bond, linoleic acid ($C_{18}H_{32}O_2$) 26 % with two carbon–carbon double bonds, and linolenic acid 10 % with a carbon–carbon triple bond. During the boundary lubrication conditions, linolenic acid will be decomposed into different molecules under the high pressure-temperature condition followed by linoleic acid and oleic acid due to the reactivities of the specific unsaturated bonds.

Free oxygen molecules will be quickly absorbed by the DLC surface and tend to reach the bulk materials resulting in removal of carbon from the DLC surface and later graphitization. Such graphitization and oxidation (of the oil and bulk material) products will be formed. Therefore, friction decreases during oxygen absorption at the surface but later wear increases. Detailed explanations of the wear and friction mechanisms can be found elsewhere.

3.3 FESEM-EDX Results

Figure 6 shows the FESEM micrographs of the worn surfaces of stationary balls of steel/steel, steel/DLC and DLC/DLC contacts under lubrication with base oil.
Signs of breaking of the coating were observed in the DLC/DLC contact, and the surface outside of the damaged area appeared shiny and very smooth, (Fig. 6(c)). No major signs of adhesion or formation of islands were observed. No transverse scratches were present on the DLC/DLC contact surfaces when lubricated with base oil. The steel/DLC contact surfaces were covered by many islands flakes of adhered debris particles under base oil (Fig. 6(b)). The transferred particles were fused together by the friction-induced heating (known as welding) and to form the islands flakes. The steel/DLC contact surfaces showed the highest wear, but surprisingly, no mechanical damage or transverse cracks were observed (see Fig. 6(b)). This finding results from the absence of counter asperities on the DLC surface, and plastic deformation and increased wear of the steel surface, which could later initiate vibration. In addition, signs of tribochemical reaction were found on the DLC surfaces. Wear of the steel/steel contact surfaces was lowest, but the surface was deformed plastically. In addition, grooving in the direction of motion together with signs of adhesion were found on the contact surface. Energy dispersive X-ray fluorescence spectrometer (EDX) analyses of as-deposited DLC surface and rubbed contacts were performed and data is presented in Table 4.

3.4. Raman Analysis

High working temperature and/or high friction induced heating can cause structural change of the DLC coatings which is also called graphitization, and is generally analyzed with Raman spectroscopy through oxygen absorption from the canola oil. DLC coatings show some common features in Raman spectra such as the D peak around 1360 cm⁻¹ (breathing mode of sp² atoms in rings only) and the G peak around 1560 cm⁻¹ (bond stretching of sp² atoms in both chains and rings). In this study, the Raman spectra of the DLC contact surfaces in DLC/DLC and steel/DLC contacts were compared with the Raman spectra of as-deposited surfaces in Figs. 7(a) and 7(b) respectively. The spectrum is shown within 1000-2000 cm⁻¹ because the anticipated peaks for DLC coating lie in this range. Graphitization occurs if the ratio of the D peak and G peak (I_D/I_G) of the contact surfaces is higher than that of the as-deposited surface. In this study, the change of peak ratios in both contacts was very small (Fig. 7) so that any graphitization occurred only on the nano-scale.

Table 4 Elemental Compositions Obtained from EDX Analysis

| Contact     | C     | O     | Cr    | Fe    |
|-------------|-------|-------|-------|-------|
| As-deposited| 98.78 | 1.04  | 0.06  | 0.12  |
| Steel/Steel | 10.66 | 16.10 | 2.84  | 70.40 |
| Steel/DLC  | 34.15 | 7.77  | 13.89 | 44.19 |
| DLC/DLC    | 48.80 | 10.61 | 7.06  | 33.53 |
This study investigated the effect of the counter surfaces on the tribological behaviour of ta-C DLC coating in the presence of vegetable oil, using steel/DLC and DLC/DLC contacts with steel/steel contact as the reference. The friction coefficient was lowest for the DLC/DLC contact and highest for the steel/steel contact as shown in Fig. 4. GC-MS investigation has shown that the canola oil used in this study was highly unsaturated with oleic acid as the main element (Table 2). The characteristics of the polar groups and unsaturated bonds are important for the tribological properties, especially for the non-reactive DLC surface\(^{13,14,30}\). The lowest friction coefficient observed for the DLC/DLC contacts suggests that any type of unsaturated molecules performed better with ta-C DLC than steel. Oleic acid is known to perform very well with ta-C DLC compared to steel\(^{31}\). Since canola oil contains high amounts of oleic acid, we assumed that contacts involving ta-C DLC showed reduced friction due to the presence of oleic acid in the canola oil. This effect is mainly due to the oxygen contained in canola oil which reacts by molecular absorption with the DLC surface and consequently decreases friction, but such absorption is also responsible for wear of the DLC surface.

The higher friction coefficient observed for the steel/DLC contact might be caused by the debris particles formed during operation penetrating the uncoated steel surface\(^3\). Graphitization of the sliding surfaces would reduce the friction coefficient. In this study, a small increase in peak ratios (\(I_d/I_g\)) was observed in the Raman analysis of steel/DLC and DLC/DLC contacts, which indicates the occurrence of nano-scale graphitization on the outermost DLC surfaces during the tribology tests. Graphitization (carbon oxidation) was slightly higher in the DLC/DLC contact compared to steel/DLC contact. Therefore, graphitization reduced the friction coefficient for the DLC/DLC contact compared to the steel/DLC contact. Presumably the intrinsic properties of the coating such as high hardness and low adhesion, and the surface properties such as surface roughness mainly maintained the low friction coefficient of the DLC/DLC contact compared to the steel/DLC contact\(^{14}\).

Figure 3 shows that the friction coefficient was very low at the beginning, subsequently drastically increased, and again became stable. This drastic increase in friction was due to the formation and agglomeration of wear particles as well as detachment from the sliding surface\(^{32,33}\). The continuous agglomeration of wear particles and detachment was the cause of the stability of the friction coefficient.

Figure 5 clearly shows that the wear loss was different for various combinations, lowest for the steel/steel contact and highest in steel/DLC contact, and very low for the DLC/DLC contact and comparable to the steel/steel contact. Wear loss of DLC surface was very high for the steel/DLC contact despite the hardness of DLC being several times that of steel. The wear characteristic of the steel/DLC contact can be understood from analyzing the behaviour of carbon and iron atoms of the contacting surfaces. Investigation of diamond against carbon has shown that carbon showed strong affinity for iron to form covalent bonds\(^{34,36}\). In addition, the diamond surface was graphitized due to friction-induced heating. Therefore, diffusion of carbon into the steel surface due to the thermo-chemical interaction between the DLC and steel, and the resultant graphitization accelerated the wear of the DLC surface in the steel/DLC contact. In fact, Raman analysis of the DLC surface of the steel/DLC contact observed nano-scale graphitization (Fig. 7b). Carbon atoms have strong affinity to form inter-carbon covalent bonds, but the wear of the DLC/DLC contact was noticeably lower than that of the steel/DLC contact.
nation by adsorbed oils might have prevented the formation of covalent bonds in the DLC/DLC contact\(^ {37}\).

In this study, the worn DLC surface of the steel/DLC contact was covered by many islands/ flakes of adhered wear debris (Fig. 6(b)). Presumably the applied load and speed resulted in high temperature sufficient to fuse the loose particles from both surfaces, thus forming islands/ flakes\(^ {38}\). Some of the adhered layers were de- formed plastically and adhesive wear seemed to be the leading wear mechanism for the steel/DLC contact. No major signs of adhesion were observed for the DLC/ DLC contact, as expected because the DLC films are well known for low adhesive properties (Fig. 6(c)). In addition, no transverse scratches were seen on the rubbed surface of the DLC/DLC contacts because the sliding took place between surfaces of similar hardness. Breaking of the coating indicates that high contact stresses were produced during rubbing and the smooth mirror-like surface indicates polishing caused by mechanical wear (Fig. 6(c)). Moreover, although Aero- components and unsaturated molecules improve the tribological properties of the lubricated contact, these are also the main reason for the high oxidation of vegetable oil\(^ {31}\). Iron oxide hard particles can be formed due to the high level of oxidation, and may cause the mechanical damage to the sliding surface\(^ {39}\). However, wear was lowest for the steel/steel contact, although smearing and deformation of wear particles was observed (Fig. 6(a)).

5. Conclusion

This study investigated the tribological properties of steel/steel, steel/DLC and DLC/DLC contacts under boundary conditions in the presence of canola oil. The results are summarized below.

(1) Steel/steel contact showed the lowest wear and highest friction coefficient.

(2) DLC/DLC contact showed the lowest friction coefficient and comparable wear to the steel/steel contact. Steel/DLC contact showed the highest wear but slightly lower friction coefficient than the steel/steel contact.

(3) Low friction coefficient of the DLC/DLC contact might be due to the high oleic acid content in canola oil and graphitization of the topmost DLC surfaces, because of oxygen absorption, and the intrinsic properties of DLC coating such as low adhesion, high hardness, etc.

(4) Islands were formed in the steel/DLC contact that was absent the DLC/DLC contact. Wear of the DLC surface in the steel/DLC contact was tribochemical not present in the DLC/DLC contact.

(5) Oxygen contained in the canola oil formed an inert coating on the DLC/DLC surface. However, this oxygen was quickly absorbed by the bulk materials and caused chemical reactions, resulting in increased wear.

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