

EVALUATION OF TRIBOLOGICAL PERFORMANCE OF HYDROGENATED DLC BY SURFACE TEXTURING IN THE PRESENCE OF PALM BASED TMP ESTER AT DIFFERENT TEMPERATURES

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Keywords

TMP Ester, Surface textured; a-C:H DLC coating; graphitization, wear resistance; friction

Abstract

Surface textured and DLC coated (hydrogenated amorphous carbon) samples were assessed for their coating performance at 40 °C, 80 °C and 125 °C. As a result, textured *a-C:H* DLC demonstrated higher but stable coefficient of friction (COF) at high temperatures as compared to un-textured DLC samples. However, textured DLC samples showed higher wear resistance compared to un-textured DLC coating. The enhancement can be elucidated by the lower graphitization of textured DLC samples.

1. Introduction

In the past, surface texturing has demonstrated reduction in friction [1] and wear [2]. Applications such as piston/cylinder [3], mechanical seals [4] and cutting tools [4] have been textured to enhance their tribological properties. Micro/ nano textures improve the tribological properties by increasing the load carrying capacity through a hydrodynamic effect, minimizing third body abrasion by entrapping wear debris and reducing the increase in temperature at contact by keeping the lubricant at the interface [4].

Lately, surface textured diamond-like carbon (DLC) coated surfaces have been experimented in oil-lubricated [5] and water lubricated [6] sliding conditions. DLC coatings are commonly being utilized to enhance the tribological properties of engineering components such as bearings, gears, seals and metal cutting tools, because of their hardness and preferred tribological performance. Thus, the usage of DLC by automotive industry is also growing. However, the performance of DLC coating is heavily influenced by temperature and ambient condition such as relative humidity. As a result, DLC is doped with hydrogen, various metals and nitrides to sustain the tribological properties of DLC at harsh environment [7]. At the same time, surface texturing has also been considered as an alternative method to improve the tribological performance of DLC coating. Ding et al. [6] showed a decrease in friction and wear in textured *a-C* coating under water lubrication. Song et al. [8] demonstrated rise in the wear life span of *a-C:H* DLC coating in a vacuum environment. Most of the research done in this area are conducted at room temperature and the thermal stability of DLC coatings at higher temperature has yet to be investigated thoroughly. Therefore, the present study aims to examine the effect of textured *a-C:H* DLC coating at 40 °C, 80 °C and 125 °C. Moreover, the effect of textured *a-C:H* DLC coating in palm oil based trimethylolpropane (TMP) ester was also evaluated as an alternative to the conventional base oil. This investigation will then help validate the suitability of textured DLC coating at high temperature applications and in environmentally friendly base oil applications.

2. Methods

Hardened Steel was used as a substrate material with an average hardness of ~58 HRC. The material was cut into 15 mm by 15 mm pieces. It was then polished to reduce the average surface roughness to 35 nm. The counter ball material was 100Cr6 with a diameter of 6.35 mm and average surface roughness of 40 nm. Micro surface textures with approximate dimensions of dimple depth, diameter and density of ~8 μm , ~100 μm and ~20% respectively, were produced on the substrate using a Picosecond laser. Once textured, the micro bulges around the dimples were cleaned by light polishing with a diamond suspension of 0.5 μm particle size. Then, the *a-C:H* DLC coating was deposited by hybrid magnetron sputtering.

To determine the *a-C:H* DLC coating performance at different temperatures, a ball-on-flat-type tribo-tester in a reciprocating motion was employed. Palm oil based trimethylolpropane (TMP) ester was used as an environmentally friendly lubricant. The procedure for the production of palm oil based TMP ester can be found in Zulkifli et al. [17].

The load, speed and temperatures selected to simulate the cam/ tappet interaction are as follows: load = 100 N; temperatures selected is 40 °C, 80 °C and 125 °C; frequency = 5 Hz; test duration was 2 hours and stroke length was 2.5 mm. The applied load corresponds to the maximum Hertzian contact pressure of 2.36 GPa, which is much higher than the typical contact pressure of cam lobe and valve tappet interface at 0.6 GPa. Therefore, the present condition was selected to enable shorter laboratory testing procedure and accelerate the measurable wear. The symbolic representations used in the present study are as follow:

Table 1. Symbolic representations used in the present study.

| Sample and tested condition | Symbolic representations |
|--|--------------------------|
| DLC coated un-textured sample tested at 40 °C in TMP | TMP-C40 |
| Textured and DLC coated sample tested at 40 °C in TMP | TMP-T40 |
| DLC coated un-textured sample tested at 80 °C in TMP | TMP-C80 |
| Textured and DLC coated sample tested at 80 °C in TMP | TMP-T80 |
| DLC coated un-textured sample tested at 125 °C in TMP | TMP-C125 |
| Textured and DLC coated sample tested at 125 °C in TMP | TMP-T125 |

Each samples were cleaned with acetone in an ultrasonic bath before each test. After tests, the samples were cleaned with *n*-heptane before wear track characterization. After the removal of the tribolayers (if any), the wear rate was measured by calculating the wear volume using the weight loss method. An average of three measurements was taken to calculate the wear volume. Finally, the wear rate was determined with Archard equation of $V = k F s$ whereby, 'V' is the wear volume, 'k' is the specific wear rate coefficient, 'F' is the normal load and 's' is the total distance travelled.

Nanoindentation hardness tests (Hysitron) was used to determine the mechanical properties of DLC coating. An atomic-force microscopy (AFM) (Ambios technology) and a scanning electron microscopy (SEM)/ energy dispersive X-ray spectroscopy (EDS) (Hitachi) were used to analyze the wear track surface morphology. EDS was also utilized to investigate the elemental composition of the wear track. Besides that, the change in I_D/I_G ratio after the sliding test was determined by Raman spectroscopy (Renishaw). Deconvolution of the Raman spectra was conducted using data analysis and graphics software (OriginLab).

3. Results and Discussion

With nanoindentation hardness tests, DLC coating showed hardness was around 18.4 ± 2.2 GPa. Figures 1a-1c present the coefficient of friction (COF) difference with time for textured and un-textured DLC samples. From Figure 1, textured DLC samples showed comparatively steady COF with time compared to un-textured DLC

samples. With the increase in temperature, the COF for un-textured DLC decreases, while it increases for textured DLC. TMP-T40 shows lower COF compared to TMP-C40 during the friction test. Figure 1b shows that COF for TMP-C80 was lower than TMP-T80.

At 125°C (Figure 1c), COF for TMP-C125 show an abrupt increase at the start. After that, COF reduced for TMP-C125 and increased until the completion of the test. For TMP-T125, after the initial instability, COF steadily increased to until the completion of the test. The reason for the decrease in COF of un-textured DLC at 80 °C and 125 °C may be due to higher graphitization transformation [12]. As a result, the coating layer becomes soft and the COF reduces but wear increases [13]. This may be the reason for the higher wear rate in the case of un-textured DLC, as shown in Figure 2. Furthermore, graphitization could be confirmed by Raman spectroscopy. Table 2 presents the Raman spectra shift for specimens at 125 °C before and after test. The shift in the G peak and increase in the I_D/I_G ratio show an increase in the sp^2 fraction of carbon in the DLC film [14], which confirms graphitization has occurred. The results also indicate that even at higher temperatures, a lower graphitization transformation can be observed with textured coating.

TMP-C125 exhibited coating delamination and scratches as shown in Figure 3. As can be seen in Table 3, specimen TMP-C125 exhibited a decrease of carbon (C) and higher increase of iron (Fe) in comparison to as-deposited amorphous hydrogenated carbon coating.

For textured DLC, COF increases when temperature increases, as shown in Figure 1c. This may be due to the reduction in graphitization by the aid of textures. The reduction in graphitization can be explained by the lower wear rate of textured DLC as compared to un-textured DLC, as shown in Figures 2. Besides that, Table 2 confirms the lower graphitization for TMP-T125 by slight change in G peak shift and I_D/I_G ratio.

It was observed that wear coefficient values nearly followed the COF trend in the textured DLC case. However, in the case of un-textured DLC, it was the opposite trend that was observed. This can be explained by the fact that higher graphitization helps in reduction of COF but increment in wear rate. As a result, textured DLC shows higher wear resistance compared to un-textured DLC throughout the different temperature tests.

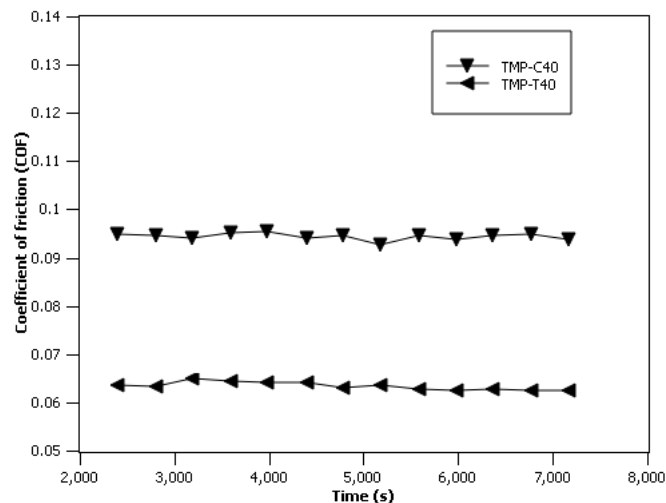


Figure 1a. Coefficient of friction (COF) at 40 °C

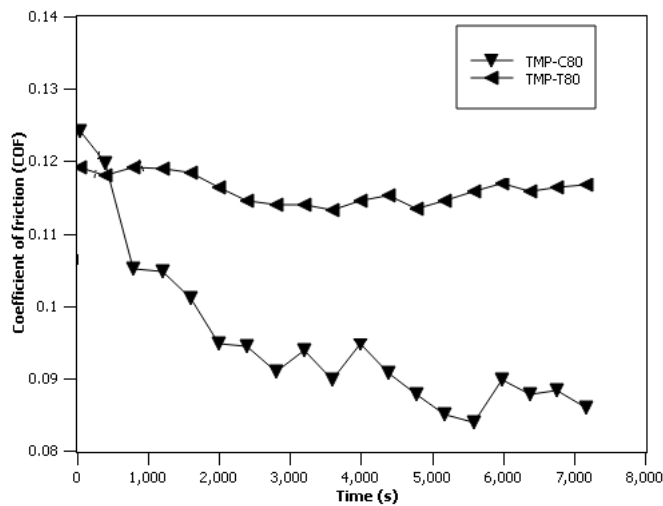


Figure 1b. Coefficient of friction (COF) at 80 °C

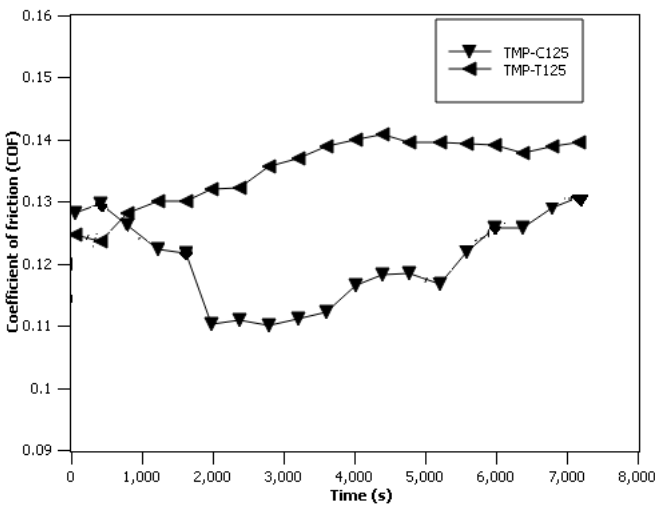


Figure 1c. Coefficient of friction (COF) at 125 °C

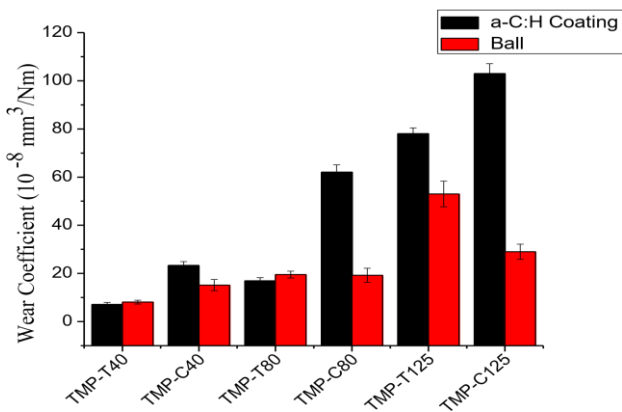


Figure 2. Wear coefficient of textured and un-textured DLC at various temperatures in TMP

Table 2. Raman spectra shift for specimens at 125 °C

| Specimen | Before test | | | After test | | |
|----------|----------------------------|----------------------------|--------------------------------|----------------------------|----------------------------|--------------------------------|
| | D-Peak (cm ⁻¹) | G-Peak (cm ⁻¹) | I _D /I _G | D-Peak (cm ⁻¹) | G-Peak (cm ⁻¹) | I _D /I _G |

| | | | | | | |
|----------|---------|---------|------|---------|---------|------|
| TMP-C125 | 1351.08 | 1533.07 | 1.17 | 1376.03 | 1559.74 | 1.57 |
| TMP-T125 | 1351.08 | 1533.07 | 1.17 | 1355.11 | 1535.00 | 1.20 |

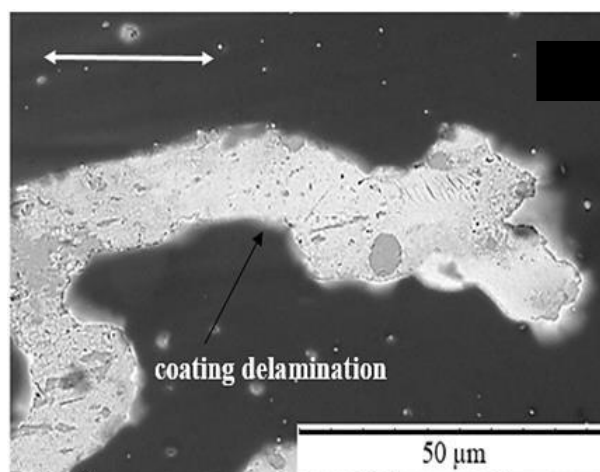


Figure 3. SEM image of un-textured DLC of TMP-C125

Table 3. EDS analysis of *a-C:H* coating at various temperatures

| Specimen | Elements (Atomic %) | | | | |
|------------------|---------------------|--------|----------|---------|---------|
| | Carbon | Oxygen | Chromium | Ferrous | Silicon |
| As-deposited DLC | 96.27 | 0.56 | 2.83 | 0.34 | - |
| TMP-C125 | 34.23 | 3.45 | 21.22 | 41.10 | - |

4. Conclusions

The tribological performance of textured *a-C:H* DLC coating has been presented and compared with un-textured *a-C:H* DLC coating. At a lower temperature (40 °C), textured DLC showed lower friction and wear compared to un-textured DLC. At higher temperature (80 °C and 125 °C), textured DLC showed higher friction compared to un-textured DLC. However, textured DLC showed higher wear resistance throughout the different temperature tests. In addition, TMP ester showed a strong potential to replace fossil fuel based lubricant. This line of study will be very useful to promote and optimize further, textured DLC coating as a wear resistant coating for automotive components.

ACKNOWLEDGMENTS

The authors are grateful to Ministry of Higher Education (MOHE) of Malaysia for FRGS FP007-2014A support. Thanks to Professor Dr. Robiah Binti Yunus (Universiti Putra Malaysia) for providing Palm oil based TMP ester.

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