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# **EVALUATION OF TRANSFORMED LAYER HARDNESS OF** DLC COATING AFTER FRICTION TEST

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## **Graphical abstract**



### Abstract

Diamond-like carbon (DLC) provide very excellence performance in term of friction coefficient and wear resistance under boundary lubrication. the nano characteristic of the transformed layer has not been studied in terms of its hardness which is believed to have a significant effect in the tribological performance. This study presented the scratch test of the DLC transformed layer was obtained from the AFM scratch test that governs the friction behavior of DLC. As a result, the hardness of the DLC transformed layer depends on the oil temperature, where the sliding interface of DLC softened during the friction test due to graphitization process.

Keywords: DLC, scratch test, graphitization, transformed layer hardness.

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# **1.0 INTRODUCTION**

Diamond-like carbon (DLC) provide very excellence performance in term of friction coefficient and wear resistance under boundary lubrication in mineralbased oil with or without additives [1, 2, 3, 4]. DLC became more interesting as a coating material in tribological applications especially in the automotive industry in order to reduce the fuel consumption [5], because of their significant tribological properties such as low friction, high hardness, and high wear resistance. Improvements in coating technology have made the DLC coating suitable for various machine components that operate under severe conditions i.e under boundary lubrication. The low friction mechanism in the DLC coating under friction tests in either dry or boundary lubricated conditions is due to the formation of a graphite-like layer in the nonlubricated [6, 7, 8] or transformed layer [8, 9] which provides low shear strength at the contact interface. The transition phase of the as-deposited DLC to graphite-like structure at the topmost sliding interface is due to graphitization by conversion of sp<sup>3</sup> to sp<sup>2</sup>. This transition phase has been reported in other studies as a friction-induced transformation of the topmost sliding interface of the DLC coating [10, 11, and 12]. The high pressure during impact test also could be caused a graphitization process of DLC [13].

However, the nano characteristic of the transformed layer has not been studied in terms of its hardness which is believed to have a significant effect in the tribological performance, such as a low friction coefficient under boundary lubrication conditions. The most important finding in this study is the scratch hardness of the transformed layer at the topmost of the sliding interface of the DLC from the AFM scratch test under different oil temperatures.

## 2.0 METHODOLOGY

#### 2.1 Friction Test

Table 1 shows the mechanical properties of the as-deposited DLC coated sliding pin (\$55C) and the \$55C disk. The specimens were obtained from outside sources. Figure 1 shows the schematic diagram of the pin-on-disk apparatus used to conduct the friction test. The information on the coating method is reported elsewhere [14].

The friction tests were conducted using a pinon-disk apparatus (Figure 1), where the DLCcoated cylindrical pin specimen was in a sliding line contact on the disk specimen. The constant normal load of 10 N was applied to the pin. The disk specimen was rotated with a constant velocity of 65 mms<sup>-1</sup>, and the pin was kept stationary.

 $\ensuremath{\mbox{Table1}}\xspace$  Hardness and deposition method of DLC coating and disk

Friction tests were conducted for 100 m sliding distance at different oil temperatures of 25, 80 and 160

Name	Hardness (GPa)	Deposition Method
DLC 1	47	PVD
DLC 2	12	CVD
DLC 3	6	CVD

°C. During the friction test, the sliding contact between the specimens occurred in boundary lubrication and while immersed in additive-free mineral based oil.



Figure 1 Schematic diagram of pin-on-disk friction test apparatus.

#### 2.2 Scratch Test

A scratch hardness test was performed to find the hardness of the transformed layer that is believed to have been formed at the topmost worn surface during the friction test. The scratch test was carried out using a commercial AFM (SII, SPA-400) in ambient temperature and humidity of 25°C and 40 %, respectively. The polycrystal diamond (PCD) tip cantilever from the Nano World AG, which has a tip radius of 150 nm and a spring constant of 42 Nm<sup>-1</sup>, was used to perform the scratch test. Figure 2 shows the scanning and the scratch area when the scanning process was done before and after the scratch hardness test in order to obtain different surface profiles of the worn area. The load was applied to the cantilever for the scanning and hardness scratch of 50 and 1000 nN, respectively. The AFM stage speed of 1 µms<sup>-1</sup> was set for both the scanning and scratch hardness test. The scratch movement of the PDC tip was programmed to be the same in all the tests. As shown in Figure 2, the initial position of the scratch hardness was at the bottom-left corner of the scratch area and then moved in a straight line by 500 nm along the y axis with a constant normal load and scratch speed. Then the PCD tip detached from the surface, and moved to the bottom position and was translated by 4 nm of x axis. These movements were repeated for 125 times for 1 cycle in order to create a scratch area of 500×500 nm<sup>2</sup>. The advantage of the scratch method compared to the nano-indentation in terms of the determination of the thin layer hardness is that the influence of the substrate hardness can be minimized [16].



Figure 2 Schematic diagram of scan and nanoscratch test area at worn surface of DLC.

#### **3.0 RESULTS AND DISCUSSION**

#### 3.1 Effect of Oil Test Temperature On Scratch Depth

A scratch test was carried out to investigate the hardness properties of transformed layer of all DLCs after the friction test in different oil test temperatures from 25 to 160 °C. Figure 3 shows the cross-sectional profile of the worn area. All types of DLCs showed the same behavior in that the scratch depth increased with increases in oil temperature. The different depth of worn area shows the development of transformed layer duirng friction test.

The deepest scratch depth for all the DLCs shows at a high oil test temperature with the value of 5.5, 12.7 and 29.2 nm for DLC1, DLC2 and DLC3, respectively.

In Figure 4, the scratch depth values of all the DLCs have been plotted as a function of the oil temperature. The increase in the scratch depth by increasing the oil temperature showed that the DLC coating became softer during the friction test and was influenced by the oil temperature [8]. At the high test temperature, the transform rate of sp<sup>3</sup> to sp<sup>2</sup> is higher comparing to lower test temperature. Higher sp<sup>2</sup> content means that the topmost surface becomes softer. This can be observed that the depth of the scar from scratch test is much bigger at high test temperature comparing to low test temperature.



Displacement x, nm





Figure 4 Influence of oil temperature on scratch depth

The structure of the coating at the sliding contact interfaces has transformed the DLC into a graphite-like layer due to the friction-induced graphitization process [12]. The graphite-like transformed layer has a more sp<sup>2</sup> structure compare to the as-deposited DLC. The friction-induced graphitization process softened the contact interface during sliding [9, 14]. Graphitization occured more at high temperature which contribute to low hardness of transformed layer. The grahite like transformed layer has a significant influence to friction performance[7].

# 3.2 Effect Of Oil Test Temperature On Friction Coefficient

Figure 5 shows the friction coefficient as a function of oil temperature. The friction coefficient of DLC1 decreased from 0.05 to 0.03 with an increase in oil temperature. However, the friction coefficient of DLC1 increased from 0.03 to 0.04 when the oil temperature increased from 120 to 160 °C. DLC 2 and DLC3 showed the opposite behavior, where the friction coefficient increased with elevated oil temperatures, and the value was higher than DLC1. Referring to Figure 4 and Figure 5, the lowest friction coefficient occurred at test oil temperature of 80 and 120 °C for DLC1. At this test parameter, the hardness of DLC1 after friction test shows the medium hardness. The medium hardness of transformed layer acting as separator to avoid the contact of hard surface. The transform layer controls the friction coefficient at this condition. The Low friction value is considered due to graphite layer (transformed layer) that produced low shear strength at sliding contact [6]. However, the increment of friction at high temperature because the transformed layer of DLC is too soft. In this situation, the transformed layer is not capable to act as separating layer between two hard sliding surfaces. There is more contact between two hard surface s of pin and disc, which increase the friction coefficient as occur in boundary lubrication.



Figure 5 Friction coefficient as a function of oil temperature

### 4.0 CONCLUSION

DLC is well known as a coating material that has essential properties for tribological applications due to its low friction coefficient, high hardness and wear resistance. The low friction coefficient that achieved during the friction test was due to the phase transformation of the hard DLC coating into a soft graphite-like transformed layer. This study presented the scratch test of the transformed layer was obtained from the AFM scratch test that governs the friction behavior of DLC. The hardness of the transformed layer depends on the oil temperature, where the sliding interface of DLC softened during the friction test due to graphitization.

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