

# Investigating run-in behavior of ta-C friction and understating wear behavior at high temperature

Woo-Young Lee<sup>1,\*</sup>, Takayuki Tokoroyama<sup>1</sup>, Motoyuki Murashima<sup>1</sup>, Young-Jun Jang<sup>2</sup>, Jong-Kuk Kim<sup>2</sup>, Noritsugu Umehara<sup>1</sup>

<sup>1</sup>) Department of Mechanical Science and Engineering, Graduate School of Engineering, Nagoya University, Furo-cho, Chikusa-ku, Nagoya, 464-8601, Japan.

<sup>2</sup>) Surface Engineering Department, Implementation Research Division, Korea Institute of Materials Science, 797, Changwondaero, Seongsan\_gu, Changwon, Gyeongnam, South Korea.

\*Corresponding e-mail: lwy13579@naver.com

**Keywords:** ta-C; high temperature; transferred layer

**ABSTRACT** – In this study, to clarify wear mechanism and role of carbon transferred layer on wear, ta-C coatings were subjected to friction tests under 200°C in air at different sliding cycles. The steady state friction coefficient of 0.02 was obtained after finishing a running-in cycles approximately 2,000 cycles. During the steady state, wear rate of ta-C decreased with increasing number of cycles. Such the decrease of wear rate was explained to form carbon transferred layer on the mating material during the running-in. The mechanism of those tribological properties was analysed by Raman spectroscopy and non-contact type microscopy.

## 1. INTRODUCTION

Diamond-Like Carbon (DLC) coatings are mixed structure of sp<sup>2</sup> and sp<sup>3</sup> bonded carbon atoms. DLC coating have been attracting attention due to a high hardness, high resistivity and low friction coefficient. These properties are promising for a wide range of applications such as wear resistance coatings for dry machining, engine components and cutting tools. However, these tribological properties of DLC coatings deteriorate rapidly under the high temperatures with generating frictional heat during contact [1]. Among the DLC series, non-hydrogenated tetrahedral amorphous carbon (ta-C) is an ideal candidate for tribological applications owing to good thermal properties from the higher sp<sup>3</sup> bonding in its structure.

The purpose of this study is to investigate the influence of formation of transfer layer on the friction and wear behavior which is able to help in designing wear resistance coatings applied at high temperature condition. And in this paper focused on tribological behavior of ta-C coated disk against Si<sub>3</sub>N<sub>4</sub> ball experiments at high temperatures of 200°C.

## 2. EXPERIMENTAL DETAILS

### 2.1 Preparation of DLC coating

The ta-C coatings investigated in this study were fabricated on Inconel disk of 20 mm diameter by using a Filtered Cathodic Vacuum Arc (FCVA) method. A chromium layer was deposited on the substrate as an interfacial layer between ta-C and substrate to promote adhesion properties. Pure graphite (99.999 %) was used as the cathode and the negative substrate bias of -100 V in a chamber was set to enhance the sp<sup>3</sup> ratio in its

structure, then the mechanical properties became high due to its high sp<sup>3</sup> ratio.

### 2.2 Coating characterization

The friction coefficient and wear rate of ta-C coatings were confirmed by using a high-temperature ball-on-disk tribo-meter between a ta-C coating and a Si<sub>3</sub>N<sub>4</sub> ball in a high temperature condition. These disks were heated to 200 °C with the infrared lamp, and the temperature of the specimens were maintained at a set value during the friction tests. After the friction test, the wear scar on the Si<sub>3</sub>N<sub>4</sub> ball was observed by optical microscopy and analyzed by using Raman spectroscopy (Jasco NRS-1000), with 532 nm laser to confirm the presence of carbon transferred layer. Quantitative wear rates were determined by calculating the volume of the wear tracks from the non-contact type surface profiler (ZYGO).

## 3. RESULTS AND DISCUSSION

Understanding the role of the running-in behaviors and the mechanism of transfer layer formation on the mating material are important to optimize the design of ta-C coating and assure the thermal stability of ta-C coating. Figure 1 showed friction coefficient of the ta-C coatings fabricated with a substrate bias voltage of -100 V at sliding cycles of 12,000 under temperature of 200°C. In this experiment, ta-C coating exhibited a running-in cycles and then the steady state ( $\mu_s=0.02$ ) was reached after 2,000 cycles. The other result with a different number of cycles of 4,000, 8,000 and 12,000 showed similar behavior that the running-in cycles was maintained up to 2,000 cycles and steady state was obtained as a result of 12,000 cycles.

The two-dimensional cross section image of wear track of ta-C coating for 2,000, 4,000, 8,000 and 12,000 cycles are shown in Figure 2. At 2,000 cycles, wear track of ta-C was considerable with apparent damage surface. Up to the 2,000 cycles, a fluctuation of COF was maintained with a higher wear. However, the depth of wear track became slightly deeper with an increase of cycles. A Specific wear rates can be calculated using the cross-sectional wear area with length of wear track and applied load. And the rate of the 4,000, 8,000 and 12,000 cycles was calculated except to the wear occurred in 2,000 cycles to confirm the effect of the transferred layer on the ball. The ta-C film exhibited a

higher wear rate of  $9.6 \times 10^{-6} \text{ mm}^3/\text{Nm}$  at 2,000 cycles. However, wear rate started to be decreased to  $6.0 \times 10^{-7} \text{ mm}^3/\text{Nm}$  at 4,000 cycles and then reached to the  $2.5 \times 10^{-7} \text{ mm}^3/\text{Nm}$  at 12,000 cycles.

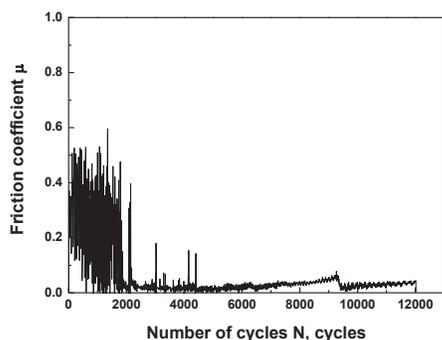


Figure 1 Friction coefficient of ta-C deposited at -100 V against the  $\text{Si}_3\text{N}_4$  ball at  $200^\circ\text{C}$ . Tests were run for 12,000 cycles using 1 N applied load at 200 rpm.

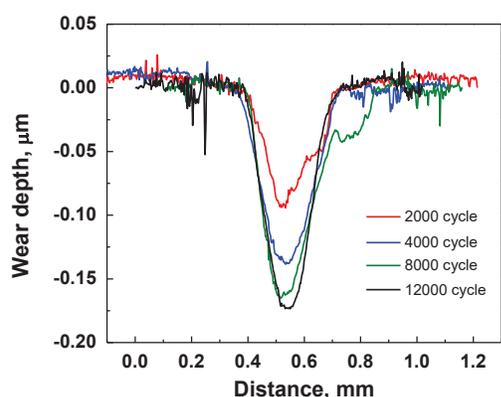


Figure 2 Cross-sectional image of wear track as a function of number of cycles.

After the friction test, there were clearly noticeable wear scar on surface of the  $\text{Si}_3\text{N}_4$  ball. Figure 3(a) shows the optical image of wear scar on the counterpart material in the contact area at 4,000 cycles. It is obviously seen that wear scars, partially covered by colourful layer, formed on the  $\text{Si}_3\text{N}_4$  ball. And there was an accumulation of carbon debris in front of wear scar.

The Raman spectra of black powder accumulated in front of the wear scar, colourful layer formed on the wear scar and polished surface was investigated by Visible Raman spectrometer, shown like Figure 3(b). The Raman spectra showed typical a characteristic of carbonaceous transferred layer in the black powder and colourful area. The appearance of D-band at  $1350 \text{ cm}^{-1}$  in the Raman spectra on the black powder and colourful layer indicated an increase of  $\text{sp}^2$  bonds with a change in the bonding structure of the surface of ta-C and attributed to the sliding induced graphitization. However, polished surface beside the transfer layer only indicated the  $\text{Si}_3\text{N}_4$  peak. Therefore, it is obviously seen that wear scars, partially covered by tribo-induced transfer films.

Under the high temperature, hydrogen and water molecules covered with the contact surface on the ta-C

film and  $\text{Si}_3\text{N}_4$  ball was released. Covalent interactions on the unsaturated surface between the ta-C and  $\text{Si}_3\text{N}_4$  ball result in run in cycles and higher wear rate until the 2,000 cycles as a process of formations of carbonaceous transfer film [2].

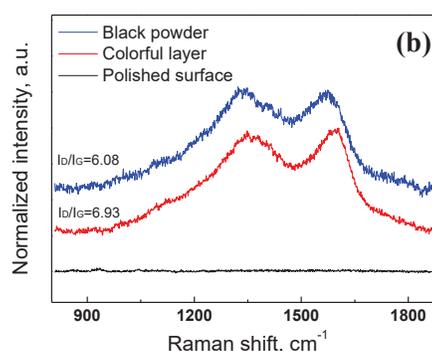


Figure 3 (a) Wear scar on the ball, (b) Raman spectrum on the wear scar after testing up to 4,000 cycles.

From the optical image of wear scar region and result of Raman spectrum, these are clearly noticeable formation of transferred layer corresponding to the low friction coefficient (0.02 shown in Figure 1) and lower specific wear rate after 2,000 cycles. Due to the amount of transferred layer formed on the wear scar on the ball, it is enough to passivate dangling bond generated at high temperature condition with a decreasing a shear strength and a protection of both ta-C film and counterpart material [3].

#### 4. CONCLUSION

The study presented the tribological behaviors of ta-C layers versus  $\text{Si}_3\text{N}_4$  ball at high temperature as a function of sliding cycles. A conclusion is that tribological behaviors at high temperature is affected by dangling bond passivation and transfer film formation. As a result, running-in cycles was finished after 2,000 cycles and wear rate was sharply decreased during the steady state region. Low friction coefficient of 0.02 and corresponding to lower wear rates of  $2.5 \times 10^{-7} \text{ mm}^3/\text{Nm}$  were observed at 12,000 cycles. Such a steady state friction coefficient and decrease of wear rate was attributed by the formation of transferred layer on the wear scar of the  $\text{Si}_3\text{N}_4$  ball. This carbonaceous transferred film can significantly decrease the covalent

interactions at the surface with protecting both the ta-C surface and Si<sub>3</sub>N<sub>4</sub> surface. The results demonstrate that transfer layer formation influence tribological behavior decreasing the interaction between ta-C films and wear scar on the Si<sub>3</sub>N<sub>4</sub> sphere. Furthermore, higher wear rate was correlated with run-in cycles as a process of formations of carbonaceous transferred film.

#### REFERENCES

- [1] Deng, X., Kousaka, H., Tokoroyama, T., & Umehara, N. (2014). Tribological behavior of tetrahedral amorphous carbon (ta-C) coatings at elevated temperatures. *Tribology International*, 75, 98–103.
- [2] Marino, M. J., Hsiao, E., Chen, Y., Eryilmaz, O. L., Erdemir, A., & Kim, S. H. (2011). Understanding run-in behavior of diamond-like carbon friction and preventing diamond-like carbon wear in humid air. *Langmuir*, 27(20), 12702-12708.
- [3] Konca, E., Cheng, Y. T., Weiner, A. M., Dasch, J. M., & Alpas, A. T. (2006). Elevated temperature tribological behavior of non-hydrogenated diamond-like carbon coatings against 319 aluminum alloy. *Surface and Coatings Technology*, 200(12-13), 3996-4005.