The Origin of the Tribofilm Formed in DLC/MAC Lubrication Using $^{13}$C-DLC

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1. Introduction

Applying diamond-like carbon (DLC) coating on the contact surface improves vacuum boundary lubrication properties of multiply-alkylated cyclopentane (MAC) oil [1]. In this DLC/MAC combination, carbon-rich tribofilm forms on the counterpart surface, which plays an important role to achieve friction and wear reduction. However, it is not still unknown whether it comes from DLC or MAC because both are carbon-based materials. In this study, a DLC coating made from carbon isotope $^{12}$C was examined. After friction test, time-of-flight secondary ion mass spectrometry (ToF-SIMS) were conducted in order to clarify the origin of the tribofilm.

2. Experimental

A DLC coating on a stainless steel substrate was produced from $^{13}$C methane ($^{13}$C-DLC) using plasma-enhanced chemical vapor deposition (PECVD). Prior to the topcoat formation, chromium-containing gradient DLC was deposited as interlayer to improve adhesion to the substrate. A droplet of MAC oil was applied on the DLC surface and spread uniformly using a centrifuge machine. The final weight of applied MAC was adjusted to 1 mg. Pin-on-disk friction test was conducted in high vacuum condition (1 × 10$^{-3}$ Pa) with a high load of 50 N (maximum Hertzian contact pressure 2.25 GPa) to obtain boundary lubrication regime. The test was finished at 10$^5$ reciprocating friction cycles. Surface analysis of the disk and the pin by ToF-SIMS was conducted after the test.

3. Results and Discussion

Figure 1 shows the Raman spectra of $^{13}$C-DLC and $^{12}$C-DLC (DLC made from normal methane). Both DLC coatings have Raman shape typical for amorphous carbon. The Raman shift frequency for $^{13}$C-DLC was $\sqrt{12/13}$ times smaller than that of $^{12}$C-DLC by isotope effect as theoretically predicted. This fact indicates that a DLC coating of $^{13}$C was well produced. Figure 2 shows the friction coefficient of $^{13}$C-DLC/MAC combination in vacuum. Friction coefficient was around 0.03 in steady-state which is similar to $^{12}$C-DLC/MAC in previous studies. Figure 3 shows ToF-SIMS images of the disk and pin. Cesium sputtering was conducted prior to the analysis in order to remove contamination. Mainly $^{13}$C was detected on the disk, contrary to the pin wear scar in which mainly $^{12}$C was detected. This indicates that the origin of the tribofilm is not DLC coating but MAC oil.

![Figure 1. Raman spectra of $^{13}$C-DLC and $^{12}$C-DLC.](image1)

![Figure 2. Friction coefficient of $^{13}$C-DLC/MAC combination in vacuum.](image2)

![Figure 3. ToF-SIMS images of the disk and pin.](image3)

4. Summary

A DLC coating made of $^{13}$C was produced and tribologically examined in vacuum together with MAC. The ToF-SIMS analysis revealed that the formed tribofilm on the pin is composed exclusively of $^{12}$C, indicating the origin of the tribofilm is MAC oil.

5. References