stle ATLANTA
May 21-25, 2017

Effects of Load and Humidity on Friction Fade-out at DLC Films Slid by ZrO_2 Pins in H_2-N_2 Gas Mixture Environment

TRACK OR CATEGORY

Materials Tribology III

AUTHORS AND INSTITUTIONS

Kato, Takahisa[1]; Nosaka, Masataka[1]; Murase, Atsushi[2]; Tohyama, Mamoru[2]; Suzuki, Masahiro[3]; Kawai, Shigekazu[3]

1. The University of Tokyo, Tokyo, Japan.
2. Toyota Central R&D Labs., Inc., Nagoya, Japan.
3. JTEKT CORPORATION, Osaka, Japan.

INTRODUCTION

Friction tests using fully hydrogenated DLC film against ZrO_2 pins under H_2 gas containing small amount of ethanol vapor were reported in authors' previous reports [1,2]. It was shown that the friction coefficient dropped to the friction tester noise level as low as 0.0001, which we termed friction fade-out (FFO). Since H_2 gas is hazardous by nature because of the risk of explosion and a wide range of flammability limits in air, i.e. from 4.1% to 74%. We attempted to reduce the H_2 content in the H_2–N_2 gas mixture by changing the main flow from dry H_2 gas to dry N_2 gas, and FFO under H_2–N_2 gas mixture is reported in this report.

For more information of FFO, the tribofilm made on ZrO_2 surface was investigated using FT-IR and TOF-SIMS (time-of-flight secondary ion mass spectrometry) analyses, where deuterated ethanol was used. It was found that a large amount of D- ions were included in the tribofilm, suggesting that the ethanol was dehydrated by a catalytic action of ZrO_2 and remained C_2H_4 contributed to form the tribofilm on ZrO_2. It will be shown that FFO depended a great deal on a humidity of environmental gas.

FRICTION TEST

An ion vapor deposition system was used in the present study to produce DLC films (NPS-330S, Nanotec Corp.). The source gas was introduced into the vacuum chamber and was then ionized by thermal electrons emitted from an anode. Carbon or hydrocarbon ions were attracted by the pulsed negative-bias voltage applied to the electrode, on which sample substrates were fixed and ions were deposited onto the substrates. Si (100) wafers (R_a: 0.12 nm, thickness: 525 μm) were prepared for use as sample substrates and were ultrasonically cleaned in ethanol, followed by acetone. After placing the Si substrate on the bias electrode in the chamber, the chamber was pumped down to a base pressure of 3×10^{-3} Pa and Ar^+ ion bombardment was performed for 30 min. For test samples, a 1200-nm-thick DLC layer was deposited using a C_7H_8–H_2 gas mixture with a bias voltage of −4.0 kV. The temperature of the heater near the sample was maintained at 230 °C throughout the deposition. H content of sample DLC, measured by Elastic Recoil Detection Analyzer (ERDA, KOBELCO, HRBS-1000), was larger value, e.g. 60 %, at the surface, but decreased down to 20 % at the depth of 10 nm by Ar ion etching and kept constant at deeper layer.

Friction tests were conducted using a pin-on-disk friction tester (FPR-2000, RHESCA INC.) in the reciprocating mode. A ZrO_2 pin was placed on a DLC sample fixed on the disk. The disk reciprocated at a constant speed of 7.2 mm/s, with a single sliding corresponding to 4.3 mm, and at a maximum applied load of 63.7 N corresponding to the maximum Hertzian pressure of 2.6 GPa. The test area including the disk and pin was covered with a double plastic casing, but without seals, namely the test area was kept at the pressure of the atmosphere. The friction tester and supplied gas system is shown in Figure 1.

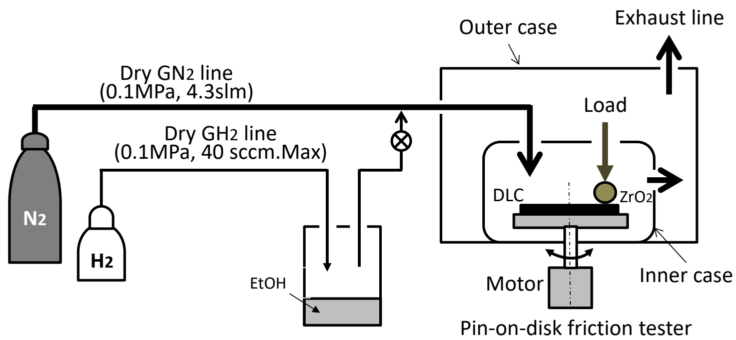

Figure1. Friction tester and supplied gas system

TEST RESULTS

Friction test results obtained in an H_2-N_2 gas mixture environment is shown in Figure 2. During run-in process in this test, an applied load was increased in a stepwise from 19.8 N to 63.7 N under a gas mixture of dry N_2 and ethanol-vapored H_2 gas. Their flow rates were 4.3 slm and 40 sccm, respectively. Then, the ethanol-vapored H_2 gas flow was reduced. When the flow rate of ethanol-vapored H_2 gas reached 5 sccm, the friction coefficient sharply dropped down to 0.0001, and remained constant for over 7 hours.

With a reduction in ethanol-vapored H_2 gas, a relative humidity at the section was also reduced. It will be shown that the steep reduction in friction coefficient from 0.01 to 0.0001 as shown in Figure 2 appears when the relative humidity reduces to around 1%. This relative humidity of 1 % when FFO appears is almost identical in almost all cases.

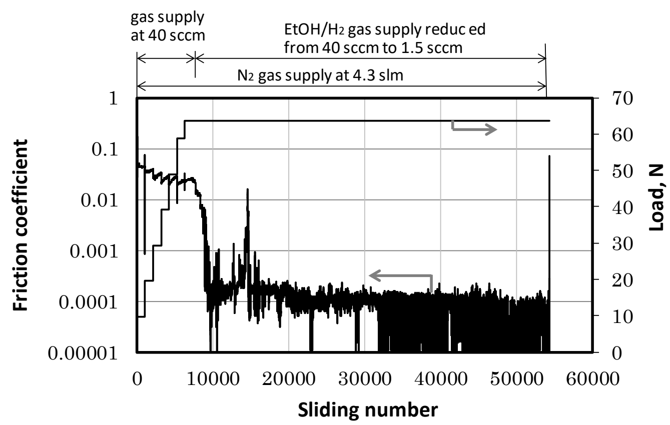

Figure 2. FFO under H_2-N_2 gas mixture environment

ANALYISIS OF TRIBOFILM

By many observations using an optical microscope and SEM, it was found the contact area of ZrO_2 was covered with tribofilm during FFO sliding. We also found the formation of tribofilm is important for the occurrence of FFO, since the tribofilm was found to be peeled off at the observation after FFO disappearance. We analyzed the tribofilm before, during and after FFO, by SEM, FTIR, TOF-SIMS and nano-indenter.

Figure 3 shows an example of FT-IR spectrum of tribofilm at ZrO_2 surface after test, which indicates -CH peaks (2900~3000 cm^{-1}) originated from aliphatic hydrocarbon and -C=O peak (1720cm^{-1}) originated from carbonyl groups. We also analyzed the tribofilm by TOF-SIMS using deuterated-ethanol-vapored H_2 gas in order to clarify the role of ethanol molecules added to H_2 gas. Figure 4 shows example of secondary ion images on ZrO_2 and DLC surfaces those occurring FFO analyzed by TOF-SIMS, and found that ethanol is dehydrated and remained -CH (alkyl group) is included into the tribofilm during run-in process. More information obtained by analysis and effects of applied load and humidity will be reported at the presentation.

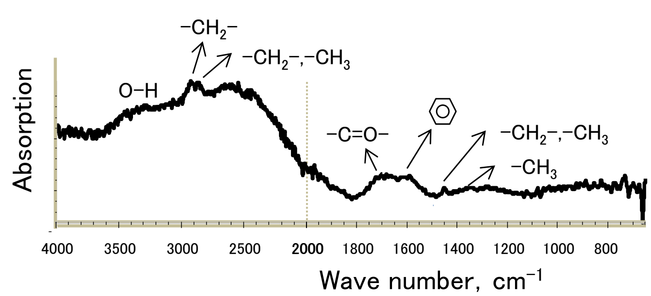

Figure 3 FT-IR spectrum of tribofilm

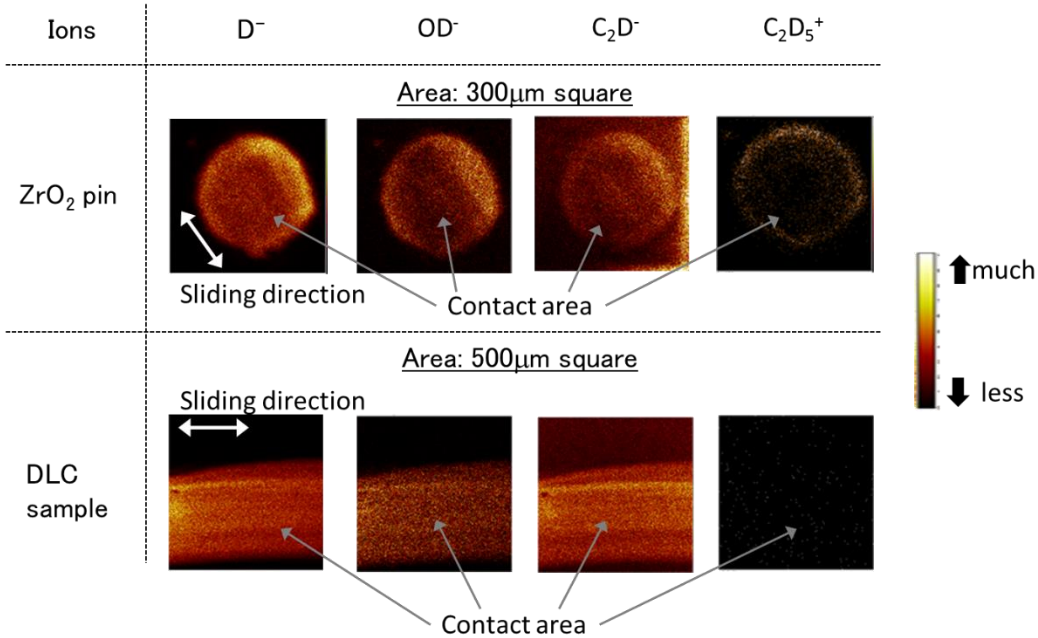

Figure 4 Secondary ion images of tribofilms analyzed by TOF SIMS

SUMMARY

ZrO_2 pin was slid against hydrated-DLC film under ethanol-vapored H_2-N_2 gas mixture, and a sharp friction reduction down to 0.0001 (FFO) was reported. FFO appeared by a stepwise increase in load and followed decrease in the supply

Tribofilm formed on the ZrO_2 surface plays important role for the occurrence of FFO, and tribofilm is analyzed by many method. An effect of relative humidity on friction reduction will be also shown in detail at the presentation.

REFERENCES

[1] Nosaka, M., Mifune, A., Kawaguchi, M., Shiiba, T. and Kato, T., 2015 "Friction fade-out at polymer-like carbon films slid by ZrO_2 pins under hydrogen environment," *Proc IMechE, Part J; J Engineering Tribology*, 229, 2015, pp.1030-1038.

[2] Nosaka M, Kusaba R, Morisaki Y, Kawaguchi M, Morisaki Y and Kato T. "Stability of friction fade-out at polymer-like carbon films slid by ZrO2 pins under alcohol-vapored hydrogen gas environment" *Online J of Proc IMechE, Part J; J Engineering Tribology,* 2016, 0(0)1-9. DOI: 10.1177/1350650116636797.

KEYWORDS

Carbon, Graphite, Ceramics