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Tribological study of nanoporous amorphous boron carbide film prepared by pulsed plasma CVD

S. Liza[1,2,*], N. Ohtake[1], H. Akasaka[1], J.M. Munoz-Guijosa[3], H.H. Masjuki[2]

[1)] Department of Mechanical Sciences and Engineering, 2-12-1 O-Okayama, Meguro-ku, Tokyo, 152-8552, Japan.
[2)] Department of Mechanical Engineering, Faculty of Engineering, University of Malaya,
50603 Kuala Lumpur, Malaysia.
[3)] Mechanical Engineering Department, Universidad Politécnica de Madrid,
C/José Gutiérrez Abascal, 2, 28006 Madrid, Spain.

[*]Corresponding e-mail: shahira@um.edu.my

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ABSTRACT – In this work, the tribological behavior of nano porous *a*-BC:H films are studied and compared with those in conventional DLC films. *a*-BC:H films were deposited by pulsed plasma chemical vapor deposition using $B(CH_3)_3$ gas as boron source. A DLC interlayer was used to prevent the *a*-BC:H film delamination produced by oxidation. Tribological test results indicate that the *a*-BC:H films show an excellent boundary oil lubricated behavior, with lower friction coefficient and qualitatively comparable wear rate than those on the DLC film. The formation of micropores from the original nanopores during boundary regimes explains this better performance. Results show that porous *a*-BC:H films may be an alternative for segmented DLC films in applications where severe tribological conditions and complex shapes exist, so surface patterning is unfeasible.

1. INTRODUCTION

This work will focus on the use of nanoporous hydrogenated amorphous boron carbide (*a*-BC:H) films as an alternative way to overcome some of the stated drawbacks of segmented diamond like carbon (DLC) films, also offering some advantages and improvements. DLC performance can be improved by alloying it with other elements such as boron, giving rise to a range of attractive physical and mechanical properties as a wear-resistant coating for mechanical systems [1-2]. We focus our attention on understanding the pore formation and tribological performance of *a*-BC:H films with a DLC thin interlayer to increase humidity permeation resistance.

2. METHODOLOGY

2.1 Film Deposition

A pulsed plasma CVD system was used for the films deposition on (100) silicon substrates. Silicon substrates were ultrasonically cleaned with distilled water, methanol and acetone for 40, 20 and 20 min, respectively. Subsequently, substrates were placed on the sample holder and loaded into the deposition chamber. Vacuum chamber was evacuated to a background pressure below 4.0 x 10^{-7} Pa using a turbo molecular pump. A 14 kHz monopolar pulsed power supply was used for plasma generation. Prior to deposition, substrate surfaces were sputter-cleaned by Ar plasma for 1 hour at a voltage of -3 kV with a gas flow rate of 30 cm^3/min. Table 1 shows the deposition conditions for the DLC and *a*-BC:H films.

Table 1 Deposition conditions for the DLC and *a*-BC:H films.

Deposition parameter	**DLC**	***a*-BC:H**	**DLC interlayer**
C_2H_2 gas flow rate (cm^3/min)	20	-	20
$B(CH_3)_3$ gas flow rate (cm^3/min)	-	15	-
Pressure (Pa)	3	5	3
Bias voltage (kV)	-3	-3	-3
Deposition time (h)	3	3	2

2.2 Film Characterization

The surface and thickness of the deposited films was examined by atomic force microscopy (AFM) (SPA300, SII Seiko Instruments Co., Ltd) and surface profilometer (Surftest SV-600, Mitutoyo Co., Ltd), respectively. Film chemical composition was determined by glow discharge optical emission spectroscopy (GDOES JY 5000RF, HORIBA). A conventional (S-DLC1) ball on disk tribometer was employed at oil boundary lubricated conditions. SAE 10W-30 engine oil and SYTOX® Green nucleic acid stain fluorescent dye fluid were used as lubricants. One fluid drop was added directly onto the film surface before initiating the tests. A 5 mm diameter stainless steel ball was pressed against the film with a normal load of 1 N. Tests were performed at a sliding speed of 0.209 ms^{-1} at ambient laboratory conditions of 25°C and 32–56% relative humidity. The duration of each sliding test was fixed at 100 000 sliding cycles, corresponding to 250 min. Finally, the porosity on the worn track was observed by fluorescence microscopy (Eclipse 80*i*, NIKON).

3. RESULTS AND DISCUSSION

In this study, the *a*-BC:H films have an average diameter of pores was approximately 189.05 ± 45.13 nm and average depth of pores was approximately 1.51 ±

0.72 nm. GDOES analysis indicated that *a*-BC:H films consist of 25.8 at.% of boron and 60.2 at.% of carbon, while DLC films consist of 84.2 at.% of carbon. Film thickness for DLC and *a*-BC:H films was 1.1 µm and 1.1 µm with 0.7 µm of DLC interlayer, respectively.

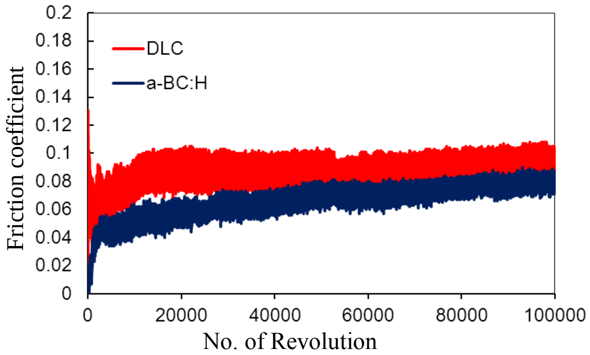

Figure 1 Friction coefficient in oil boundary lubricated.

Figure 1 shows after 100 000 sliding cycles in oil lubricated sliding, the friction coefficient of *a*-BC:H has a lower value (0.06) than that of the DLC film (0.08). This results demonstrate that porous *a*-BC:H multilayer films possess an excellent behavior under boundary lubricated regime. The wear rates for the *a*-BC:H and DLC films are not calculated because the wear scar cross section cannot be easily measured due to oil presence in the contact surface, important conclusions can be extracted by analyzing the film's wear track characteristics and correlating this information with the measured friction coefficients.

Based on this observation, we believe that pores have an important role in reducing friction under oil boundary lubricated condition, as Shum et al. [3] demonstrated for textured DLC coatings. A fluorescent dye was applied to the surface of the *a*-BC:H and DLC films in order to detect pores on the wear track during the ball on disc test

Figure 2 shows fluorescence microscopy images of the pores on the worn surface of the *a*-BC:H and DLC films. Observation on the wear track of DLC and *a*-BC:H film by fluorescence microscopy revealed very few pores on the wear track of the DLC film (Figure 2a). However, many pores were observed on the worn surface at the *a*-BC:H film (Figure 2b). In addition, the number of pores is considerably bigger than before the test (direct comparison with pore distribution out of the track area). These findings suggest that pores on the worn surface, can act as oil reservoirs, which prevent fluid film breaking by maintaining the lubricant supply to the tribo-contact during the sliding, resulting in less friction. However, more investigation is needed in order to study the influence of porosity, as there are multiple variables concurrently involved, as porosity size, shape and distribution, which affect wear and friction synergistically.

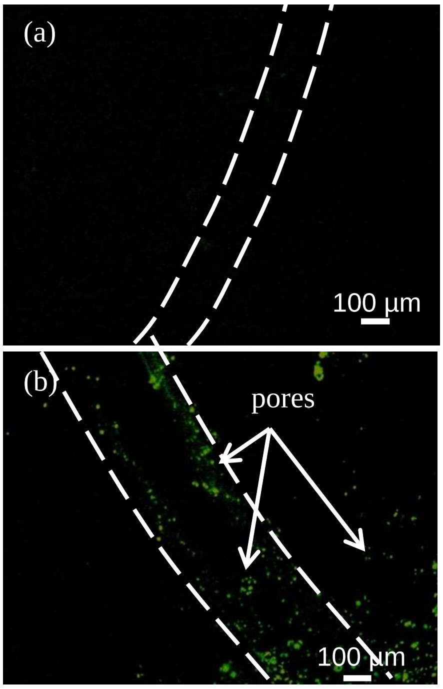

Figure 2 Fluorescence microscopy images of (a) DLC and (b) *a*-BC:H worn films after fluorescent boundary lubricated sliding test (5000 sliding cycles).

4. CONCLUSIONS

In order to investigate a reliable alternative for patterned DLC films in complex surfaces and severe conditions, 1.1 µm thick, nano porous *a*-BC:H films were prepared by using trimethylboron gas, $B(CH_3)_3$ by pulsed plasma CVD over a (100) silicon substrate, and the tribological behaviors were studied and compared to those at DLC films. Under oil boundary lubrication regime, the friction coefficient of the *a*-BC:H multilayer film was lower than that observed in the DLC film up to a higher number of cycles (100 000). Pore growth phenomenon observed on the worn surface, is beneficial in this case, as pores act as oil reservoirs which prevent fluid film breakage.

5. ACKNOWLEDGMENT

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