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# Friction and wear property of a- $CN_x$ coatings sliding against $Si_3N_4$ balls in water

Short communication

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#### Abstract

Amorphous carbon nitride coatings (a- $CN_x$ ) were deposited on Si(100) wafers and Si<sub>3</sub>N<sub>4</sub> disks using ion beam assisted deposition (IBAD), and their composition and chemical bonding were determined by X-ray photoelectron spectroscopy (XPS). The a- $CN_x$  coatings' hardness was measured by nano-indentation and the friction and wear behavior of a- $CN_x$  coating sliding against a Si<sub>3</sub>N<sub>4</sub> ball in water was investigated. The results indicated that the a- $CN_x$  coatings contained 12 at.% nitrogen and the major chemical bonding was sp<sup>2</sup> C=N and sp<sup>3</sup> C–N. The nano-hardness of the a- $CN_x$  coatings was 29 GPa. At a sliding velocity of 0.16 m/s and after running-in, the mean steady-state friction coefficient varied around 0.02 when the normal load was lower than 3.5 N, and then decreased abruptly from 0.018 to 0.007 at 5 N. For self-mated Si<sub>3</sub>N<sub>4</sub>, the specific wear rate of a Si<sub>3</sub>N<sub>4</sub> ball was a little higher than that of a Si<sub>3</sub>N<sub>4</sub> disk, while for a- $CN_x/Si_3N_4$ , the specific wear rate of a Si<sub>3</sub>N<sub>4</sub> ball was slightly smaller than that of a- $CN_x$  coating. Furthermore, the specific wear rate of Si<sub>3</sub>N<sub>4</sub> ball sliding against a- $CN_x$  coating was reduced by a factor up to 35 in comparison to that against Si<sub>3</sub>N<sub>4</sub> in water. This indicated that the wear mechanism of a- $CN_x$  coating/Si<sub>3</sub>N<sub>4</sub> ball was the formation of a carbonaceous transfer film on the a- $CN_x$  coatings via a tribochemical reaction between a- $CN_x$  coatings and water induced by friction, while that of self-mated Si<sub>3</sub>N<sub>4</sub> ceramics was the formation of silica gel on the contact zone via the reaction of silicon nitride and water. © 2007 Elsevier B.V. All rights reserved.

Keywords: Carbon nitride coatings; Si<sub>3</sub>N<sub>4</sub> ceramics; Friction; Wear; Water lubrication

### 1. Introduction

Since Liu and Cohen [1] in 1989 predicted theoretically that the existence of a metastable covalent carbon nitride compound  $(\beta$ -C<sub>3</sub>N<sub>4</sub>) with an analogous structure to  $\beta$ -Si<sub>3</sub>N<sub>4</sub>, and this carbon nitride compound, with a high bulk modulus, might have higher hardness than diamond, many attempts at developing new processing methods to obtain carbon nitride films have been performed. But until now, nearly all CN<sub>x</sub> films grown at room temperature are amorphous mixtures of carbon and carbon nitride with x ranging from 0.1 to 0.5 [2,3]. Nitrogen incorporation in the carbon coatings decreases the fraction of sp<sup>3</sup> carbon bonds by the formation of C–N, C=N and C≡N bonds. Recently,

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0043-1648/\$ - see front matter © 2007 Elsevier B.V. All rights reserved. doi:10.1016/j.wear.2006.11.048 Zhou et al. reported that the a- $CN_x$  coatings could enhance the wear resistance of a SiC ball and shorten the running-in period as sliding against a SiC ball in water at sliding velocities in the range of 0.019–0.16 m/s [4,5] and the wear-mechanism map of the a-CN<sub>x</sub>/SiC tribo-pair in water was developed [6]. Furthermore, the friction and wear properties of the  $a-CN_x/SiC$  tribo-couple in water have already been found to be better than those of the a-C/SiC tribo-pair under the same experimental conditions [7,8]. At the normal load of 5 N and the sliding velocity of 0.16 m/s in water, the friction coefficients of 0.01-0.02 were obtained as the a- $CN_x$  coatings slid against SiC and Si<sub>3</sub>N<sub>4</sub> balls, while larger friction coefficients of 0.07-0.10 were acquired as the a-CN<sub>x</sub> coatings slid against Al<sub>2</sub>O<sub>3</sub>, SUS440C and SUJ2 balls [9]. It was indicated that the amorphous carbon nitride coatings sliding against Si-based non-oxide ceramics such as SiC and Si<sub>3</sub>N<sub>4</sub> exhibited the lowest friction coefficient and lower wear rate. Currently, the friction, lubrication and wear mechanisms of the a- $CN_x/SiC$  tribo-pair have already been reviewed

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in detail [10], however, the friction and wear property of the a- $CN_x/Si_3N_4$  tribo-pair in water has not yet been studied in detail. The aim of this paper is to investigate the friction and wear properties of the a- $CN_x/Si_3N_4$  tribo-pair in water and compare their wear characteristics between the a- $CN_x/Si_3N_4$  tribo-pair and  $Si_3N_4/Si_3N_4$  tribo-pair by using ball-on-disk tribo-meters at room temperature. The influences of testing conditions on the tribological behaviors of a- $CN_x/Si_3N_4$  tribo-pair in water were analyzed.

#### 2. Experimental procedures

The IBAD machine (Hitachi Ltd., Japan) consists of a cryogenically pumped chamber, a sputter deposition source, an electron beam evaporator, two ion guns for sputtering and mixing, respectively, and a substrate holder (Ref. [5]). The diameter of the ion beam irradiation area is about 80 mm. The substrate holder consists of a water-cooled copper plate and can be rotated at a speed of 4 rpm during deposition. Prior to the IBAD process, Si<sub>3</sub>N<sub>4</sub> disks (Ø  $30 \text{ mm} \times t 8 \text{ mm}$ ) and Si(100) wafers were ultrasonically cleaned in acetone for 30 min. Then, a high purity carbon target was put into an electron beam evaporator and a substrate jig containing a Si<sub>3</sub>N<sub>4</sub> disk was fixed on the substrate holder. After that, the deposition chamber doors were closed and the vacuum chamber was subsequently evacuated to lower than  $2.0 \times 10^{-4}$  Pa. For further cleaning, the disk surface was bombarded using nitrogen ions for 5 min. After that, the a- $CN_x$  coatings were synthesized by mixing carbon vapor and energetic N ions. Energetic N ions were produced under the acceleration voltage of 1.5 kV with the acceleration current density of 90 µA/cm<sup>2</sup>. Carbon vapor was formed through heating a graphite target with an electron beam evaporator. The deposition rate was 20 Å/s, which was controlled by adjusting the emission current of carbon vapor. The coating thickness was 0.5 µm. The deposition parameters are listed in Ref. [5].

The composition and chemical bonding of the a-CN<sub>x</sub> coatings were determined by a scanning ESCA microprobe (Quantum 2000, Physical Electronics Inc., USA). The coatings' surface roughness was measured by a Surfcom-1500DX profilometer, and their hardness and Young's modulus were evaluated using a Nano Indenter ELIONIX ENT-1100A.

The diameter of all Si<sub>3</sub>N<sub>4</sub> balls was 8 mm and the balls' roughness was determined by a Surfcom-1500DX profilometer and its mechanical properties were obtained from the ball manufacturer. The data are listed in Table 1. Prior to each wear test, all samples were ultrasonically cleaned in acetone and ethanol for 30 min. The experiments were performed on the ball-on-disk apparatus consisting of a rotating disk sliding on a stationary ball at 0.16 m/s and 1.5–5 N. The rubbing surfaces were submerged in purified water. The contact point was designed at an eccentricity of 7.5 mm from the center of the rotary motion, which created a wear track of 15 mm in diameter on the a-CN<sub>x</sub> coated Si<sub>3</sub>N<sub>4</sub> disks' surface. In order to know the influence of the a-CN<sub>x</sub> coatings on the wear behavior of silicon nitride ceramic, the wear behavior of self-mated Si<sub>3</sub>N<sub>4</sub> ceramic tribo-pairs was also studied under the same conditions. The total friction cycles

were 49,200 cycles (equal to a sliding distance of 2304 m). The friction forces were detected by a LMA-A-10 N load cell (Kyowa Co. Ltd., Japan). The load cell voltage was measured by a DPM-700B strain amplifier (Kyowa Co. Ltd., Japan) and recorded by NR-110/150 data collection system (Keyence Co. Ltd., Japan) with a compatible personal computer. The diameter of the wear scar on the SiC ball under each condition was measured using a Keyence VH-8000 optical microscope (Keyence Co. Ltd., Japan). The cross-section area of the wear track on disk, A, was determined using a Tencor P-10 surface profilometer (Kurashiki Kako Co. Ltd., Japan). Thus, the specific wear rates for balls and disks were determined using the same equations in Refs. [5–9]. To know the wear mechanism of the  $a-CN_x/Si_3N_4$ and self-mated Si<sub>3</sub>N<sub>4</sub> tribo-pairs in water, the wear scars on the balls and the wear tracks on disks were observed by optical microscopy.

### 3. Result

### 3.1. Surface roughness and mechanical properties of a- $CN_x$ coatings

As seen in Table 1, the arithmetic mean roughness  $R_a$  of the a-CN<sub>x</sub> coating was a little smaller than that of the Si<sub>3</sub>N<sub>4</sub> substrate. This indicated that the energetic particle bombardment enhanced the mobility of carbon atoms on the growing surface and induced the smooth surface. Fig. 1 displays nano-indentation load versus indentation depth curves for a-CN<sub>x</sub> coatings. Based on the standard Oliver and Pharr approach [11], the mean values of the

Table 1

Surface roughness and mechanical properties of  $Si_3N_4$  ball, a- $CN_x$  coatings and  $Si_3N_4$  disk

| Name                                | $R_{\rm a}~(\mu{\rm m})$ | H (GPa)           | E (GPa)          |
|-------------------------------------|--------------------------|-------------------|------------------|
| Si <sub>3</sub> N <sub>4</sub> ball | 0.0552                   | 15.3 <sup>a</sup> | 308 <sup>a</sup> |
| $a-CN_x$                            | 0.0251                   | $29 \pm 2$        | $330 \pm 20$     |
| $Si_3N_4$ disk                      | 0.0280                   | 16 <sup>a</sup>   | 290 <sup>a</sup> |

<sup>a</sup> The data are from the sample manufacturer.



Fig. 1. Nano-indentation load vs. indentation displacement curves for  $a-CN_x$  coatings.

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