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## Study on the property of low friction complex graphite-like coating containing tantalum

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### Introduction

Lubricity refers to the ability to reduce frictional forces in a boundary lubrication state [1]. The Lubricity of ceramic coatings is poor in dry environment in particular [2,3]. For instance, traditional ceramics, like nitrides, carbides and nitrides exhibit high hardness, while their lubricity is poor, leading to the poor friction reduction effect. Liu et al reported that TiN film possessed high hardness, but showed poor lubricating property, and its frictional coefficient was far greater than that of diamond-like film [4]. However, graphite coatings such as graphite-like coating (GLC) and diamond-like coating (DLC) exhibit good lubricity, thus their wear resistance is good. Fujisawa et al in their research showed that graphite-like coatings had more effective lubricity property in wet environment, resulting in good wear resistance [5]. Wang et al reported that the deposition of graphite-like carbon film on Si<sub>3</sub>N<sub>4</sub>, SiC and WC films significantly reduced their wear rate and improved the wear resistance [6]. However, the wear resistance of the GLC and DLC under high pressure condition cannot meet the requirements [7,8]. In the previous research, doped GLC coatings were prepared using magnetron sputtering by separately embedded Ce, Y and Ta into the carbon target and chrome target.

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In order to enhance equipment lifetime under low oil or even dry conditions, tantalum was introduced into the graphite-like coating (GLC) by sputtering mosaic targets. The results showed that the introduction of Ta obviously reduced the friction coefficient and hardness of the GLC, while improved the wearability. When the atomic percentage of Ta was larger than 3%, the steady friction coefficient was lower than 0.01, suggesting the coating exhibited super lubricity. When the content of Ta was about 5.0%, the average friction coefficient was 0.02 by a sliding friction test under load of 20 N in unlubricated condition. Its average friction coefficient reduced by 75%, compared with that of control GLC (0.0825). © 2017 Published by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (http:// creativecommons.org/licenses/by-nc-nd/4.0/).

> It was found that the doping of Ta improved the wear resistance of the GLC coating and reduced its frictional coefficient [9,10]. However, the effect of Ta doping content on the wear resistance is still unclear, limiting the application of Ta in GLC coatings.

> In this paper, Ta was incorporated with the carbon target and chromium target by insert process during magnetron sputtering to prepare GLC coatings doping with Ta. The effects of Ta doping amount on the friction coefficient, hardness and wear loss were studied, and the cause for the better friction property was analyzed. The results can provide a foundation for preparing new wearability and low friction coefficient coatings.

### Experimental

### Preparation of the coatings

The doping element was introduced into GLC by magnetron sputtering. The sputtering targets were composed of two Cr targets (Chengdu Ultra Pure Applied Materials CO., LTD) and two carbon targets (Beijing Zhongjinyan New Materials Science and Technology Ltd.), in which there has 6 through holes with the diameter of 10 mm in the Cr target's etching area, and 8 through holes with the diameter of 6 mm in the C target's etching area, as shown in Fig. 1. Then, A certain number of tantalum inserts (Chengdu Ultra Pure Applied Materials CO., LTD) were prepared and plugged into the holes of C targets. C inserts or Cr inserts were placed into the







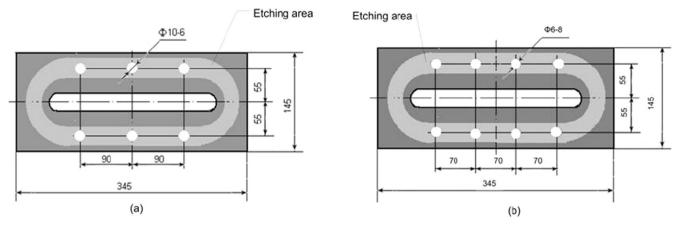


Fig. 1. Holes distribution allow etching area for (a) chromium target and (b) carbon target.

rest holes to form an integrated target. The effects of Ta on the properties of the complex GLC were studied by adjusting the inserted materials and numbers.

The number (n) of the inserts was calculated according to Eq. (1) [10]:

$$\mathbf{n} = k\mathbf{s}/\alpha\beta\omega\mathbf{S}_1\tag{1}$$

where k is atomic content of Ta element in the GLC coating (wt%); S is total target area of sputtering (mm<sup>2</sup>);  $\alpha$  means inserts to target sputtering coefficient ratio;  $\beta$  means inserts to target density ratio;  $\omega$  is weight fraction of inserts (wt%); S<sub>1</sub> is cross-section area of one insert (mm<sup>2</sup>).

Carburized AISI 3310 steel with a quenching hardness of HRC60 ± 2 was used as substrate. The ground surface was further polished to a roughness of Ra =  $0.02 \mu m$ . Subsequently, the substrates were ultrasonic washed with acetone for 15 min and dried in hot air to remove the residual solvent. The substrates were then put on a sample holder in sputtering equipment for coating. The complex GLC were prepared using an industrial CFUBMSIP system (UDP 650/4) with two mosaic carbon targets and two mosaic chromium targets. The dimensions of vacuum chamber are  $\Phi650 \text{ mm} \times 650$ mm, and the length and width of the sputter targets were 380 mm and 175 mm, respectively. The purity of carbon targets and chromium targets were 99.5% and 99.95%, respectively. Pulsed bias power is controlled by an Advance Energy Pinnacle Pulse 10 KW and DC magnetron powers are controlled by two Advance Energy Pinnacle  $6 \times 6$  power units which both provided by Advance Energy, Ltd. The rotation speed of the one rotation axis substrate holder was 4rpm. As the vacuum degree of the chamber was 3.0  $imes 10^{-5}$  Torr, the substrates were first precleaned through Ar plasma by using pulsed DC bias (-400 V). Then, 0.5  $\mu$ m Cr interlayer was deposited on the substrates by DC magnetron sputtering with a high power of 6.0 A and a pulse DC bias voltage of -100 V. Subsequently, the Cr targets power was decreased from 6.0 A to 0.3 A and the carbon targets power was increased from zero to 7.0 A, followed by the deposition of a C/Cr/Ta multilayer of graded composition (approx 0.2 µm). After that, with the Cr target power of 0.3 A and the C target power of 7.0 A, the main carbon layer contented an amount of Cr and Ta (2  $\sim$  3  $\mu$ m) was deposited. The samples keep on rotating to ensure the uniformity of each step of deposition. The substrate temperature was kept at 300 °C during the deposition [11].

### Characterizations

Adhesion was measured using a Teer Coatings ST3001 scratch tester. The radius of Rockwell diamond indenter was 0.2 mm. Addi-

tional testing parameters were: loading rate of  $100 \text{ N} \cdot \text{min}^{-1}$  and sliding speed of  $10 \text{ mm} \cdot \text{min}^{-1}$  [12].

Optical microscopy was used to examine the coatings. The thickness of coatings was assessed using the ball crater tapersection technique.

Plastic microhardness was measured using a Fischerscope H100 ultra microhardness tester with a load of 50 mN. Under an indentation depth being more than 10% of the coating thickness, a composite hardness value was obtained.

The microstructure of the complex GLC was analyzed using JSM-6700F scanning electron microscope (SEM). The composition and elements combining state of the complex GLC coating were analyzed by energy dispersive spectrometer (EDS) and X-ray photoelectron spectroscopy (XPS).

Pin-on-disc sliding wear tests were performed on the coated samples using a  $\Phi$ 5 mm chrome steel (AISI52100) ball as the counterface. The test was carried out in air at room temperature and without lubrication. The counterface ball was fixed and coated samples slid beneath with a sliding distance of 360 m and a friction time of 0.5 h. A sliding speed of 0.2 m·s<sup>-1</sup> was set for all the tests. Specific wear rates were determined from the abrasion loss [12]. Each type of samples was tested at normal applied forces of 20 N, 40 N and 80 N, giving an actual contact pressure of about 0.8  $\sim$  3.2 GPa. The schematic diagram for the test setup is shown in Fig. 2.

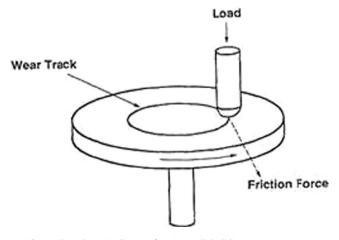


Fig. 2. The schematic diagram for pin-on-disk sliding wear test setup.

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