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Influence of annealing temperature on thermal stabilities of hydrogenated amorphous carbon on silicon nitride balls

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ABSTRACT

Hydrogenated amorphous carbon films were synthesized on silicon, silicon nitride (Si_3N_4) ceramic balls and SUS304 by using linear ion beam deposition technology. To investigate the annealing effect on the thermal stabilities of the films on Si_3N_4 balls, Raman spectroscopy, Fourier transformation infrared (FTIR) spectroscopy, X-ray photoelectron spectroscopy (XPS), nanoindentation, stylus profiler and ball-on-disk friction test were adopted. The results showed that the annealing temperatures had no significant influence on the structure, mechanical and tribological properties of the films until 200 °C. When the temperature was above 200 °C, the film structure changes as effusion of hydrogen and graphitization leading to poor mechanical and tribological properties. Roles of the tribochemical reactions occurred on Si_3N_4 ball surface during the friction in the tribological properties of hydrogenated amorphous carbon films were discussed.

1. Introduction

With the rapid development of modern science and technology, especially aerospace technology, the wear problems of major equipment rotating friction pair under extreme working environment are attracting extensive attention [1–3]. As a basic and key component in aero-engine, rolling bearing which is widely used to enable rotation and support significant load concerns not only the reliability but the life expectancy of aero-engine. Due to the increasing rotational speed and elevated temperature occurring in the contact area, bearing balls of rolling bearing are facing various failure modes like micro spalling and micro slippage as a consequence of enhanced friction and degenerative lubrication [4]. Silicon nitride (Si_3N_4) ceramic, on account of its low thermal expansion coefficient, high strength and excellent oxidation resistance, has become the preferred material for rolling bearing balls in high-speed, vacuum, lean oil, etc. [5–7].

Lubrication is another significant factor that influences the service life of rolling bearing [8,9]. In recent years, scholars have found that solid lubricant can overcome the shortcomings of fluid lubricant and better serve in more extensive occasions, such as ultra-high vacuum, wide operating temperature range, zero gravity and intensely ionizing radiation [10,11]. Diamond-like carbon (DLC) is a metastable form of amorphous carbon containing diamond structure of sp^3-C and graphite structure of sp^2-C [12]. Due to its excellent properties in terms of high hardness, high wear resistance and chemical inertness, DLC has been widely used in space applications as solid lubricant coating [13]. Therefore, by combining each of the advantages of Si_3N_4 and DLC film, Si_3N_4 bearing balls coated with DLC is efficient to improve the wear resistance and prolong the service life of rolling bearings. However, considering the elevated temperature which can reach to 300 °C during the friction process [14], the understanding of thermal stabilities of DLC films have become important and imperative.

The H-containing form of amorphous carbon (a-C:H) is one of the major DLC families. Unfortunately, previous studies have demonstrated that high temperatures can lead to the graphitization of a-C:H film due to its unsatisfying thermal stability [15,16]. It is also well known that the film develops an intrinsic property of high compressive stress which influences the adhesion to the substrate [17]. Post annealing treatment can effectively release the internal stress and improve the adhesion to the substrate, but at the same time the structure of the film will change which can significantly affect the tribological properties [18]. It was shown that annealing under certain temperatures, a-C:H films can retain their excellent properties and even present a decrease in friction coefficient, but lose these qualities while elevating the temperatures [19]. However, the critical transition temperatures which are related to the

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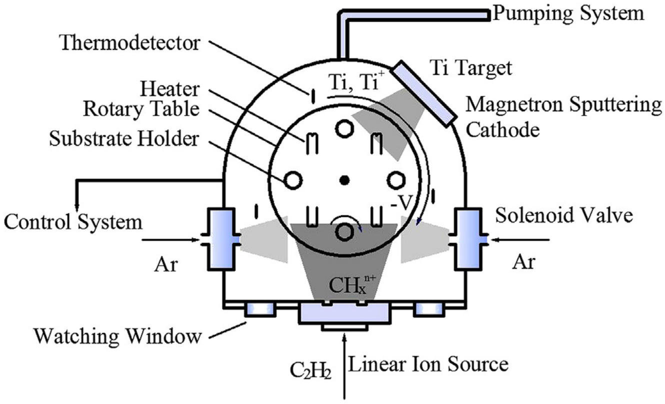

Fig. 1. Schematic diagram of a-C:H films deposition device.

preparation methods are not identical [20,21]. Besides, numerous researches have been focused on flat DLC film and how it behaves with different counterparts or in different environments, when it comes to curved or spherical surface, considering the different thickness uniformity, adhesive strength and action area size, the characteristics of DLC films may exhibit differently. Thus, the investigation of thermal stabilities of a-C:H films on Si_3N_4 balls should be important and utility for the application of the films.

In this present paper, a-C:H films were prepared by linear ion beam deposition technology (PVD). The thermal stabilities of the films on Si_3N_4 balls were studied by annealing in vacuum. The effect of annealing temperatures on the structure, mechanical and tribological properties was investigated.

2. Experimental details

2.1. Films deposition

The a-C:H films about 1.4 μm thick were synthesized on n-silicon (100) which is used for Fourier transformation infrared (FTIR) spectroscopy and mechanical test, Si_3N_4 balls and SUS304. Details of the deposition device are shown as Fig. 1.

This vacuum device contains a heating and measuring system to ensure the instantaneity of the chamber temperature and preserve the heat, a 99.99% purity titanium (size 300 × 75 mm^2) target installed as the magnetron sputtering cathode, and a linear ion source used for cleaning and a-C:H deposition. Pulsed bias voltage is superposed onto the rotary table with rated voltage ranging from 0 to −2000 V.

Prior to the deposition, the samples were cleaned ultrasonically for 15 min in absolute ethyl alcohol and then assembled in the substrate holder. The chamber was evacuated to 5×10^{-3} Pa followed by plasma sputtering with Ar^+ ions for 15 min under pulsed substrate bias voltage of −1000 V with duty cycle of 60%. Acetylene was introduced as carbon source for the formation of a-C:H films with flow rate of 80 sccm. For the purpose of enhancing the adhesive strength of the film to the substrate, a 0.2-μm sputtering titanium transition layer was prepared before the a-C:H deposition. Details of the film synthesis conditions are shown in Table 1.

Table 1
Summarizes the synthesis conditions for a-C:H films.

Item	Parameter
Ion current	1.5 A
Pulsed bias voltage	1400 V, 80%
C_2H_2 gas flow rate	80 sccm
Deposition pressure	0.8 Pa
Deposition time	40 min
Temperature	25 °C

Key: sccm: standard centimeter cube per minute.

2.2. Annealing treatment

The post annealing treatment was performed in a high-temperature box resistance furnace in vacuum at 100 °C, 200 °C, 300 °C and 400 °C for 1 h with heating rate of 10 °C/min followed by cooling inside to room temperature.

2.3. Mechanical properties

The hardness of a-C:H films was measured through TTX-NHT2 nanoindentation tester equipped with a diamond Berkovich tip and determined using continuous stiffness option. The maximum indentation depth was kept at 100 nm to minimize the substrate contribution.

The internal compressive stress was measured by Dektak XT

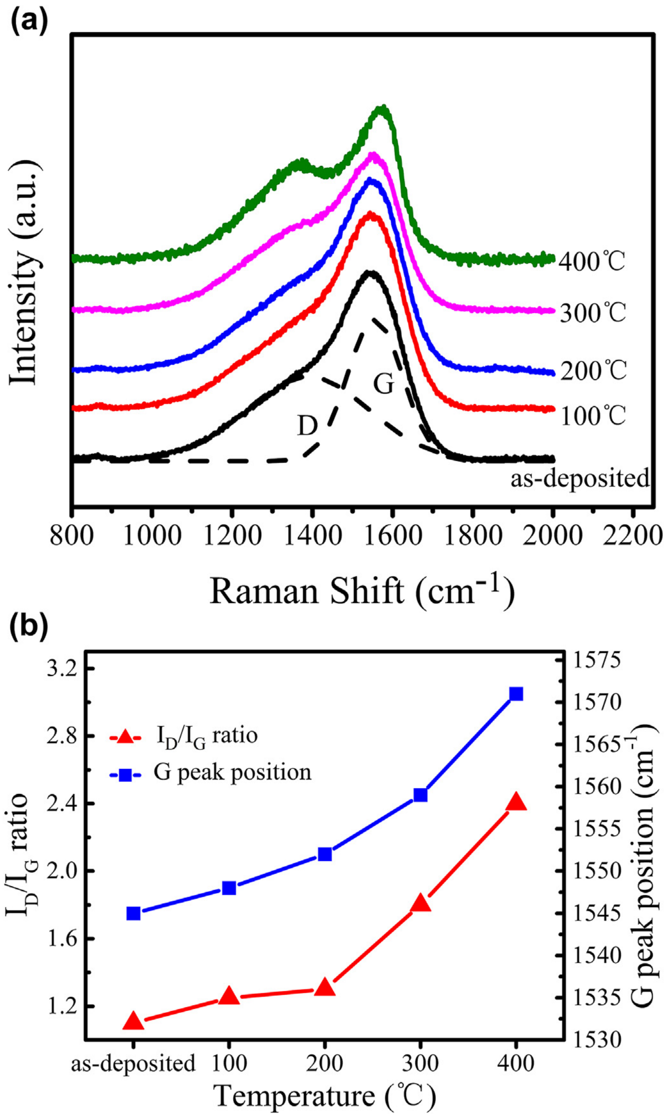

Fig. 2. (a) Raman spectra of a-C:H films with various annealing temperatures. (b) Gaussian fitting results for Raman spectra.