



Influence of diamond surface chemical states on the adhesion strength between Y_2O_3 film and diamond substrate



Chenyi Hua, Jianchao Guo, Jinlong Liu, Xiongbo Yan, Yun Zhao, Liangxian Chen, Junjun Wei, Lifu Hei, Chengming Li*

Institute for Advanced Materials and Technology, University of Science and Technology Beijing, Beijing 100083, China

ARTICLE INFO

Article history:

Received 29 January 2016

Received in revised form 4 May 2016

Accepted 9 May 2016

Available online 10 May 2016

Keywords:

CVD freestanding diamond

Chemical oxidation

Y_2O_3 antireflection film

Adhesion strength

ABSTRACT

The Y_2O_3 film deposited on chemical oxidation diamond substrate exhibited high adhesion strength and survived thermal shock at 800 °C. Oxygenated functionalization of the diamond surface had been successfully achieved via wet chemical oxidation with the increase in O/C atomic ratio from 0.04 to 0.13. The surface energy of the diamond substrate was significantly improved from 35.7 mJ/m² to 61.4 mJ/m². After depositing Y_2O_3 film on the diamond substrate, the C—O—Y bonds were observed at the interface. In addition, the quantity of C—O—Y bonds significantly increased with the chemisorbed oxygen concentration increasing. The critical scratch load of Y_2O_3 film deposited on chemical oxidation diamond substrate increased from 22.5 N to 34.1 N. Furthermore, the Y_2O_3 film deposited on chemical oxidation diamond substrate can withstand harsh thermal change and protect the diamond substrate from elevated temperature oxidation in atmospheric air without showing degradation of infrared optical performance.

© 2016 Elsevier Ltd. All rights reserved.

1. Introduction

Chemical vapor deposition (CVD) diamond is a type of ideal long-wave infrared (LWIR) (8–12 μm) windows and dome material for high-speed aircraft because of its excellent properties, such as high transmittance in the infrared (IR) wave band, low absorption coefficient, and high resistance to thermal shock and friction [1–3]. However, CVD diamond has been observed to oxidize in air above 700 °C; moreover, the IR transmittance of CVD diamond is considerably degraded, thereby restricting the use for IR windows in high-temperature oxidation environments [3]. To find a suitable protective coating for diamond, which may provide better oxidation resistance and increase the transparency by reducing the reflectivity is desirable.

Yttrium trioxide (Y_2O_3) possesses attractive properties, such as good thermal and chemical stability, high mechanical strength and hardness, low free carrier concentration, as well as high transmittance in infrared and ultra-violet wavebands [3,4]. Yttrium oxide is an ideal material as protective film to improve the antireflection and oxidation resistant properties of diamonds [5–8]. However, the protective film frequently suffers from damage or delamination from substrate when functioning in severe conditions, which are due to considerable thermal stress caused by thermal expansion coefficient differences between the film and substrate [9,10]. To obtain good adhesion to the diamond substrate, researchers have exerted considerable efforts to improve adhesion

strength between the Y_2O_3 film and diamond surface using aluminum nitride films [4] or amorphous hydrogenated silicon [5–7] as an interlayer. However, aluminum nitride film exhibits poor optical performance beyond 8 μm [6,7], and the silicon yttria bilayer film displays an unacceptable degree of optical loss at approximately 11 μm [6]. The disadvantages impede the intended broad spectral performance of the bilayer coating.

In this study, the surface of diamond substrates were functionalized via wet chemical oxidation. The chemical states, surface morphology, and surface energy of diamond substrates were systematically investigated. The adhesion strength and the thermal shock resistance of Y_2O_3 antireflection film deposited on diamond substrate were studied. The mechanism of enhanced adhesion between Y_2O_3 film and diamond substrate was discussed.

2. Experimental

2.1. Sample preparation

The diamond substrates in the experiment were CVD freestanding polycrystalline diamond films prepared by DC arc jet plasma method. The double sides of the deposited diamond film were polished. The thickness of the diamond film was about 500 μm and the surface roughness (R_a) was below 1 nm. The diamond films were ultrasonically cleaned in deionized (DI) water, acetone, ethanol, and DI water to remove surface contamination. The chemical oxidation of diamond films was prepared by boiling in a strong acid via mixing H_2SO_4 and HNO_3

* Corresponding author.

E-mail addresses: hua07de@163.com (C. Hua), chengmli@mater.ustb.edu.cn (C. Li).

solution at a volume ratio of 9:1 for 30 min. After acid boiling, samples were cleaned using an ultrasonic machine in DI water.

Y₂O₃ films were deposited onto diamond substrates by radio-frequency magnetron sputtering. Yttrium (Y) metal target of 2 in. diameter and 99.95% purity was employed for reactive sputtering. Distance between substrate and the target is 12 cm in accordance with process parameter optimization. The base pressure of the deposition chamber was 1.2×10^{-3} Pa. The substrate temperature was controlled at 400 °C by resistive heaters with feedback control. Before deposition, no pre-deposition sputter cleaning stage was used on these diamond substrates to avoid graphitization and reduction of the content of oxygen-related chemical groups on the diamond surface. The target was pre-sputtered in an argon atmosphere for 10 min to remove the surface oxide. During deposition, the flow rate of oxygen and argon was 0.9 sccm and 40 sccm, respectively. The sputtering power was 180 W and the application of negative bias to the substrates was –100 V. In accordance with the deposition rate of 3.10 nm/min, two kinds of thickness of the Y₂O₃ films were deposited on diamond substrates, as follows: one was an ultrathin film of ~3 nm, which was used to study the chemical states of Y₂O₃ at the interface. [11] The other one was an antireflection Y₂O₃ film of 1.71 μm. Based on the antireflection design, which was the quarter-wave layer that gave minimum reflection at a design wavelength of 10.6 μm, the designed thickness of antireflection Y₂O₃ film was 1.58 μm. [12,13] Therefore, during the adhesion tests and thermal shock tests, the 1.71 μm-thick Y₂O₃ antireflection films were used; the adopted thickness was slightly higher than the design value of 1.58 μm.

2.2. Sample characterization

Atomic force microscopy (AFM) images of the diamond substrates were recorded by a scanning probe microscope (SPM-9600, Shimadzu) in contact mode, with a scanned area of $5 \times 5 \mu\text{m}^2$ and resolution of 512 pixels/line. The surface chemical states of diamond substrates and the chemical states of Y₂O₃ at the interface between the films and diamond substrates were investigated by X-ray photoelectron spectroscopy (XPS) (PHI Quantera, Ulvac-Phi) with a monochromated Al Kα X-ray source. Surface morphologies of Y₂O₃ films after thermal shock test were observed by a scanning electron microscope (FEI Quanta FEG 450). IR transmittance analysis was performed using Fourier transform infrared spectroscopy (Excalibur 3100, Varian). All transmission spectra were recorded at 8 cm^{-1} resolution.

2.3. Surface energy measurement

The surface free energy was determined by a contact angle test using the sessile drop method on a contact angle measurement system (JC2000C4, Powereach) at ambient humidity and temperature. Doubly distilled water and glycerin were used to determine the hydrophilicity, as well as the surface energy of the samples. For each specimen, average values of five measurements at different positions were adopted to obtain good statistics. The surface energy of the films was calculated using the Zimans and Good method [14] by combining the Young equation and the Van Oss equation of the work of adhesion [15,16], as follows:

$$W_a = 2(\gamma_l^p \gamma_s^p)^{1/2} + 2(\gamma_l^d \gamma_s^d)^{1/2} = \gamma_l(1 + \cos\theta) \quad (1)$$

where W_a is the work of adhesion, θ is the contact angle, γ_l , γ_l^d , and γ_l^p are the surface tensions and their dispersive and polar components of the liquid phases, respectively, and γ_s , γ_s^d , and γ_s^p are the dispersive and polar components of the solid phases, respectively.

2.4. Scratch test

The adhesion of the Y₂O₃ film on diamond substrate was evaluated on a micro-scratch tester with a diamond stylus of 0.2 mm radius

(WS-2005, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences). The load increased from 0 N up to 50 N at a loading rate of 50 N/min. The scratch distance for each test was set at 5 mm. Scratch tests conducted on each sample included five repeated scratch tests. The scratched track was examined by a light microscope (Olympus BX51, Olympus).

2.5. Thermal shock test

The thermal shock tests were carried out in a muffle furnace in atmospheric air. When the temperature of the furnace reached up to 800 °C, the specimens were pushed into the furnace. The specimens were held for about 60 s in the furnace, and subsequently cooled down in air (Beijing winter at average of 5 °C).

3. Results and discussion

3.1. Surface composition and bonding states of diamond

XPS measurements were performed to identify and quantify the functional groups introduced at the surface of the diamond by chemical oxidation. Both wide and high resolution XPS spectra were recorded to investigate the surface composition of diamond substrates. Fig. 1 shows the survey spectra of diamond substrates before and after chemical oxidation treatment. The absence of peaks other than the C 1s peak (284.9 eV) and the O 1s peak (532.0 eV) in wide scan XPS spectra of typical diamond substrates demonstrated that the diamond substrates were free from any other detectable contamination after chemical oxidation treatment. A sharp C 1s peak together with a peak of O 1s were observed for the as-received diamond. After the chemical oxidation treatment, a strong O 1s peak and an O KLL Auger peak were clearly observed. The calculated O/C ratio was increased from 0.04 to 0.13, which indicated that the amount of chemisorbed oxygen greatly increased after chemical oxidation treatment.

Deconvolution of the C 1s core level allows the identification of the different bonding states of the surface carbon atoms. As shown in Fig. 2, for both as-received and chemical oxidation diamond surfaces, high intensity peaks were identified at 284.8 eV, attributed to sp³ bonded carbon in bulk [17]. The components at 285.9 and 287.0 eV may be attributed to C—O—C ether bonds and to C=O carboxyl radicals, respectively [17,18]. The intensity of the C—O—C associated peak of the chemical oxidation diamond was significantly higher than that of the untreated specimen. The as-received diamond substrate exhibits a peak at 283.7 eV, assigned to sp² bonded carbon [17], which was not

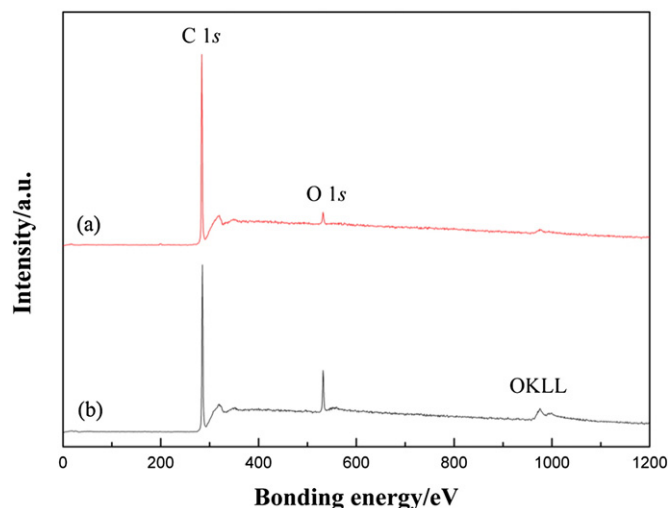


Fig. 1. XPS survey spectra of diamond substrates: (a) as-received substrate; (b) chemical oxidation substrate.

Download English Version:

<https://daneshyari.com/en/article/827818>

Download Persian Version:

<https://daneshyari.com/article/827818>

[Daneshyari.com](https://daneshyari.com)