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Researches on uniformity of diamond-like carbon films deposited on inner surface of long and slender quartz glass tube by enhanced glow discharge plasma immersion ion implantation and deposition

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ABSTRACT

Enhanced glow discharge plasma immersion ion implantation and deposition (EGD-PIII&D) has been successfully used for depositing diamond-like carbon (DLC) on the inner surface of the quartz glass tube with an inner diameter as small as 0.9 mm and aspect ratios of over 100. And the uniformity is a key problem for the DLC films deposited on the inner surface. By changing the C_2H_2 flow rate and negative voltage, the effects of the gas flow rate and voltage on the uniformity of the DLC films deposited on the inner surface of the quartz glass tube are experimentally examined. The electron-neutral collision frequency, ν_e, is used to qualitatively analyze the mechanism of uniformizing DLC films. The results reveal that uniform DLC films may be obtained by balancing both the gas flow rate and the negative voltage.

1. Introduction

Interest in diamond-like carbon (DLC) films is motivated by their unique combinations of physical hardness, high electric resistivity, high thermal conductivity, chemical inertness and optical transparency [1–4]. Studies have been recently accelerated by its prospects of commercial applications such as bearings [5], dies [6], artificial joints [7], cutting tools [8] and tubes [9]. Among these applications, DLC films deposited on inner surface have a wide range of application as protective coating in areas such as oil pipeline [10], high aspect dies [11], and vascular graft [12,13]. Therefore, a large number of techniques have been studied to fabricate DLC films on the inner surface, such as radio-frequency plasma [13], electron spin resonance [14], magnetron sputtering [15], and arc ion plating [16]. In spite of the recent progress, it is still difficult to deposit DLC films on the inner surface of insulating tube with a high aspect ratio, and a convenient and simple inner surface plasma deposition technique is required for numerous biomedical and industrial applications of DLC [12,13].

In our previous works, a hybrid deposition method, enhanced glow discharge plasma immersion ion implantation and deposition (EGD-PIII&D), is developed to deposit DLC films on the inner surface of the quartz glass tubes with different aspect ratios [17]. Compared with other techniques, EGD-PIII&D system is an easily installed and low-cost device which can be used to deposit the DLC films on the inner surface of the quartz glass tube with an inner diameter as small as 0.9 mm and length-to-diameter ratios of over 100. Meanwhile, in EGD-PIII&D process, the DLC film deposition rate is as high as 1.3 μm/min accompanied by minor changes in the bulk properties. However, in our previous studies [17], the thickness of the DLC films is not uniform along the inner surface of the quartz glass tube in EGD-PIII&D process, whereas the thickness of DLC on the outlet decreases to a quarter of that on the inlet. Consequently, the issue of DLC film deposition uniformity is considered as the most important problem to be solved.

In this article, by changing the C_2H_2 flow rate and the negative voltage during deposition, the mechanism of uniformizing DLC films is investigated. The effects of the C_2H_2 flow rate and negative voltage on the uniformity of the DLC films deposited on the inner surface of the quartz glass tube are systematically examined and theoretically analyzed. As a result, suitable processing parameters are identified to improve the uniformity.

2. Experimental details

The schematic diagram of the EGD-PIII&D system with a base pressure of 2×10^{-3} Pa was depicted in Fig. 1. The EGD-PIII&D apparatus used in this paper was described in detail elsewhere [17]. The glass chamber had a 170 mm inner diameter with 270 mm height. 200 mm long quartz tube with inner diameter of 4 mm was pre-cleaned in deionized water bath ultrasonically, rinsed in an ethanol bath, and then loaded into the EGD-PIII&D chamber. Prior to DLC film deposition, the inner surface of the quartz glass tube underwent Ar^+ sputter cleaning

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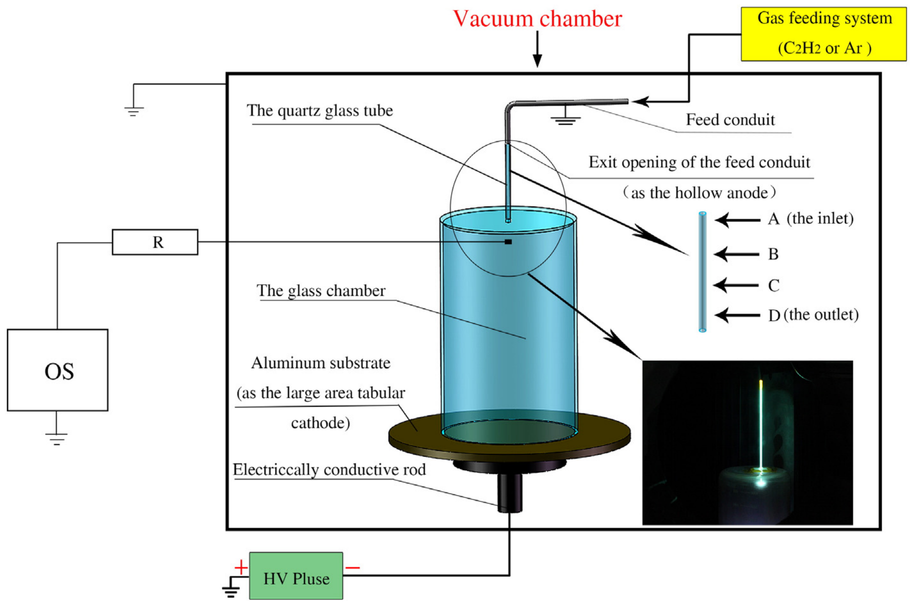

Fig. 1. Schematic of the EGD-PIII&D system for inner surface treatment of quartz tubes.

for 5 min by using a deposition pulse voltage to remove surface contaminants and surface oxide. Subsequently, acetylene (C_2H_2) was bled into the chamber through the quartz glass tube. In order to investigate the mechanism of uniformizing DLC films, two sets of contrast experiments were designed: the first one kept the negative voltage fixed while the C_2H_2 flow rate was varied (the detailed parameters of film deposition were summarized in Table 1, samples 1#, 2#, 3#, 4#); the other one kept the C_2H_2 flow rate fixed while the negative voltage was varied (samples 1#, 5#, 6#, 7#). In the process, the pulse width and frequency of the voltage pulse were 100 µs and 50 Hz, respectively.

Via the finite element method, the acetylene pressure between the glass chamber and the quartz glass tube was calculated. The pressure at the bottom of glass chamber (see Fig. 1) was set as the exit pressure of the model and was measured by ionization vacuum gauge. The pressure at the bottom of glass chamber with different C_2H_2 flow rates was listed in Table 2. The acetylene density and coefficient of dynamic viscosity at standard temperature and pressure were 1.17 kg/m^3 and 10.5×10^{-6} Pa·s, respectively [18].

In our experiments, as shown in Fig. 1, a Langmuir probe made of copper wires with a diameter of 2 mm was employed to investigate the plasma potential (by considering that in all cases of the present experiment, the plasma potential was typically a few volts more positive than the floating potential, then floating potential was used as the plasma potential instead for simplification in this paper) at the outlet of the tube [19–21]. The tip was positioned at the center axis and the distance between the tip and the outlet was 5 mm. The probe was connected to ground via a large resistor (R = 10 MΩ). The plasma potential of the plasma bulk was monitored by an oscilloscope via a 10× Tektronix probe with a hooked probe tip and the resulting traces were saved in the oscilloscope. Experiments were performed by the parameters which were listed in Table 1.

To simplify the operation, DLC films deposited on the inner surface at four locations (A, B, C and D, see Fig. 1), which were evenly distributed on the inner surface of the tube, were used for the estimation of thickness homogeneity. DLC films on the four locations were supposed to be able to reflect the deposition rate variation along the inner surface. Then quartz glass tube was sectioned at the four locations and the DLC films' thickness was determined by scanning electron microscopy (SEM, JEOL, and JSM6010).

3. Results and discussion

The deposition rate variations of DLC films on the inner surface deposited by varying gas flow rate and by varying negative voltage are shown in Figs. 2 and 3, respectively. In Fig. 2, the films are respectively deposited with different gas flow rates of 10 SCCM, 20 SCCM, 30 SCCM and 40 SCCM under the same negative voltage of 10 kV. With a C_2H_2 gas flow rate of 10 SCCM, the film thickness at the inlet (0.43 µm) is thinner than that at the outlet (0.77 µm). And the film thickness increases linearly from the inlet to the outlet of the tube with a slope of 0.0017. Although at the 20 SCCM C_2H_2 gas flow rate, the film deposition rate (0.677 µm/min) at the inlet is higher than that (0.215 µm/min) for the 10 SCCM one, the film thickness increase shows nearly the same positive slope from the inlet to the outlet. However, a contrary tendency appears when the C_2H_2 flow rate reaches to 30 SCCM and 40 SCCM, that is, the inlet film deposition rate is higher than that at the outlet. The negative slope of the film thickness variation from inlet to outlet is nearly the same for the 30 SCCM one and the 40 SCCM one. At the inlet, with

Table 1
Preparation technique with different deposition parameters.

Sample	Negative voltage/kV	Flow rate/SCCM	Deposition time/min
1#	−10	10	2
2#	−10	20	2
3#	−10	30	2
4#	−10	40	2
5#	−12	10	2
6#	−14	10	2
7#	−16	10	2

Table 2
The pressure at the bottom of the glass chamber and the pressure in the vacuum chamber with different flow rates.

C_2H_2 flow rate/SCCM	Pressure (bottom of the glass chamber)/Pa	Pressure (vacuum chamber)/Pa
0	0.002	0.002
10	0.2	0.04
20	0.4	0.065
30	0.6	0.094
40	0.8	0.13
50	1.1	0.15