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Effect of atomic hydrogen bombardment on the surface conductivity of polycrystalline diamond films



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ABSTRACT

The surface conductivity of polished polycrystalline diamond films after atomic hydrogen bombardment for different time was compared and the carrier transport characteristic beneath the H-terminated diamond film surface was investigated correspondingly. It is found that, as the bombardment time increases, the surface roughness of diamond films first decreases due to disappearance of scratches, and then increases because of appearance of protrusions produced by plasma preferentially etching. Meanwhile the total C—H bonding concentration on the diamond surface increases gradually until it is saturated when the bombardment time is up to 30 min. The almost invariable carrier density is obtained for all samples treated for different time, which indicates the monohydride (CH) mode responsible for surface conductivity forms when hydrogen treatment starts. While for the carrier mobility, it shows the inverse trend with change of the surface roughness. A model based on the surface roughness. Combining with the carrier mobility and density, the lowest square resistance of 13.85 kΩ is obtained for polycrystalline diamond film when it is treated for 30 min. It is speculated that the surface conductivity can be further enhanced by reducing the surface roughness.

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1. Introduction

H-terminated diamond film has been proven to be a potential material alternative for high-frequency and high-power electronic devices due to the interesting surface conductivity when exposure to some absorbates [1–3]. Generally it is accepted that the surface conductivity is attributed to the sub-surface holes accumulation layer induced by electron transfer from the valence band of hydrogen terminated diamond to the absorbates on the surface [4,5]. It means that formation of H-termination and exposure to the absorbates are indispensable for the surface conductivity. As for the latter, much work has been done to enhance the surface conductivity by choosing different atmospheres [6,7] and neutral solid absorbates [8,9]. While for the former, H-termination on the diamond surface is usually obtained by chemically adsorbing atomic hydrogen activated by micro wave (MW) or hot filament (HF). The surface conductivity of H-terminated diamond films is directly influenced by the hydrogen treatment condition when the energetic atomic hydrogen interacts with the diamond surface and grain boundaries. Until now, although many plasma treatment conditions have been adopted to form H-termination by different groups such as sample temperature (500–800 °C), duration (a few seconds to 1 h) and various microwave power density and chamber pressure [10–12], there is no conclusion on which treatment condition is optimal. Thus more work should be done on the effect of atomic hydrogen bombardment under different conditions on the carrier transport characteristic below the polycrystalline diamond film surface, especially when the comparable square resistance and carrier density for different quality polycrystalline diamond films are found.

In this study, the self-standing polycrystalline diamond films were prepared by DC arc jet CVD. After atomic hydrogen bombardment in plasma for different time, the diamond surface properties were investigated. Combining with the variations in C–H bonding concentration and the surface roughness, the surface conductivity of diamond films after atomic hydrogen bombardment was analyzed. In particular, the surface roughness scattering mechanism was proposed to show the carrier transport characteristic of H-terminated polycrystalline diamond film.

2. Experimental

Self-standing polycrystalline diamond films were prepared by DC arc jet CVD in circulating gas and the deposition parameters



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| Table 1 |
|--|
| Deposition parameters of polycrystalline diamond films by DC arc jet CVD |

| Temperature/°C | Power/kW | Pc/kPa | Pr/kPa | Feed gas flow | | |
|----------------|----------|--------|--------|-----------------|------------------------|---------------------------------------|
| | | | | $H_2/Lmin^{-1}$ | Ar/L min ⁻¹ | CH ₄ /mL min ⁻¹ |
| 1050-1070 | 15–17 | 2.8-3 | 13–14 | 7 | 1.5 | 50 |

Note: Pc is chamber pressure and Pr is recycling gas pressure.

Table 2

| nyurogen plasma treatment parameters by microwave CVD. | | | | | | | |
|--|-----------|--------------|----------|--|--|--|--|
| Temperature/°C | Power/W | Pressure/kPa | Time/min | | | | |
| 800 | 1200-1300 | 5 | 5-60 | | | | |

are listed in Table 1. Then they were roughly polished by using commercial diamond grits with varying grit sizes, and fine polishing was done with fast rotating diamond grinding wheel. Before hydrogen plasma treatment, the diamond films were laser cut into $15 \text{ mm} \times 15 \text{ mm}$ pieces and the samples were boiled in the solution of H₂SO₄/HNO₃ (1:1) for 1 h to remove the amorphous carbon and other contaminants and to form O-termination on the surface of diamond film. After that, they were rinsed by deionized water, acetone, ethanol and deionized water in order. Hydrogen plasma treatment started when limiting vacuum of microwave CVD system reached 10⁻³ Pa. The polished growth surfaces of samples were bombarded by atomic hydrogen under the condition listed in Table 2. The treatment time varied from 5 min to 60 min. Hydrogen plasma was turned off after temperatures of samples decreased below 300 °C and H₂ flow was persistent until samples temperatures reached room temperature.

The growth surface of as-deposited diamond film was observed by SEM. After polishing, the crystal orientation of the diamond film was characterized by XRD. The morphology of polished and hydrogenated diamond surfaces was compared by AFM. In order to determine the C-H bonding on the diamond surface, analogical experiments were conducted on the diamond powders with size of 500 nm and the hydrogenated diamond powders were characterized by diffuse reflectance Fourier transform infrared spectroscopy (DR-FTIR). Meanwhile semi quantitative calculation based on the peak intensity of C-H bonding was used to demonstrate the change of the C-H bonding concentration on the diamond powder surface with bombardment time increase [13]. Diffuse reflectance spectra at 2 cm⁻¹ resolution were recorded with a Thermo Electron Nicolet 8700 infrared spectroscopy equipped with a DTGS detector. KBr standard powder was used as the reference spectrum for the diffuse reflectance method and 256 scans were accumulated for each



Fig. 1. Optical microscope image of the Van der Pauw-Hall pattern.



Fig. 2. Morphology of the as-deposited polycrystalline diamond film prepared by DC arc jet CVD.

diffuse reflectance spectrum. Diffuse reflectance was converted into Kubelka–Munk units.

The surface conductivity of H-terminated diamond films was characterized by Van der Pauw-Hall test using Accent HL5500 Hall System. The Van der Pauw-Hall pattern, as shown in Fig. 1, was fabricated by using Au-mask photolithography technology, which could avoid the damage of H-termination through evaporating Au film as the stopping mask of oxygen plasma contamination. Au ohmic contact was evaluated by transmission line method (TLM) and the as-deposited Au contact on H-terminated diamond surface showed low specific contact resistance of $10^{-5} \Omega \text{ cm}^2$ without



Fig. 3. X-ray diffraction pattern of the growth surface of polycrystalline diamond film.

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