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Bias enhanced diamond nucleation on Mo and CrN coated stainless steel substrates in a HFCVD reactor ☆

H. Li [a], M. Gowri [a], J.J. Schermer [a], W.J.P. van Enckevort [a], T. Kacsich [b], J.J. ter Meulen [a,*]

[a] *Institute for Molecules and Materials, Radboud University Nijmegen, The Netherlands*
[b] *IonBond Netherlands BV, Venlo, The Netherlands*

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Abstract

Bias enhanced nucleation was explored in order to obtain a high diamond nucleation density on Mo and CrN coated stainless steel substrates in a hot-filament chemical vapor deposition (HFCVD) reactor. Several bias geometries were tested on their nucleation enhancement for diamond on molybdenum. It was found that the nucleation properties observed for Mo were not much different for CrN coated stainless steel. The application of a bias between the filament and a cathode above the substrate at floating potential resulted in locally high diamond nucleation densities of up to $3 \cdot 10^9$ cm^{-2}.

Keywords: Diamond film; Hot filament CVD; Nucleation; Steel

1. Introduction

Because of its excellent mechanical and electrical properties, there has been a growing interest in depositing diamond onto various materials for many applications. The nucleation process is an important step in diamond deposition. A high nucleation density is essential to obtain a uniform and smooth diamond film. Several methods can be used for diamond nucleation enhancement [1]. In contrast to the commonly used scratch and powder pretreatment, bias pretreatment does not cause mechanical damage to the substrate but can induce a higher nucleation density, independent of the hardness of the substrate. Another advantage of bias enhanced nucleation (BEN) is its better suitability for application to more complex 3-D substrates.

Application of BEN was first reported for silicon [2–4] and later also for other substrate materials [5–21]. Although the concept of bias enhanced nucleation is simple, its actual application is complicated because it is sensitive to the substrate material used. In this work, the results of a study on bias pretreatment using different electrode configurations in a HFCVD reactor are described. Molybdenum was used as a test material. Good results of bias enhanced nucleation on Mo in a HFCVD reactor were already reported by Malcher et al. [22] and Wang et al. [23]. The present results, obtained with a different bias setup, show a nucleation density of a similar order of magnitude. Subsequently, we have applied this bias setup for the deposition of a nanocrystalline diamond film on CrN coated stainless steel. It was demonstrated before that a CrN film is a successful interlayer for diamond deposition onto iron-based materials [24]. Using BEN local nucleation densities up to $3 \cdot 10^9$ cm^{-2} were obtained, which is sufficient for the growth of a continuous diamond layer. As far as we know, no bias enhanced nucleation has been reported before for CrN coated stainless steel.

2. Experimental

The experiments were carried out in a HFCVD reactor, details of which can be found elsewhere [25]. Unless stated otherwise the substrate temperature (T_S) was kept at 800 °C. The temperature of the filament after carburizing was maintained at about 2150 °C, as measured by a pyrometer. Unless stated otherwise, a gas mixture of 2% CH_4 in H_2 (CH_4/H_2) was used, while the reactor pressure (P_r) was kept at about 30 mbar. The distance between the filament and the substrate was about 8 mm. The dimensions of the substrate were $10 \times 10 \times 1$ mm^3. Both Mo

☆ Bias enhanced nucleation on CrN coated stainless steel is reported for the first time in this paper.

* Corresponding author.
E-mail address: h.termeulen@science.ru.nl (J.J. ter Meulen).

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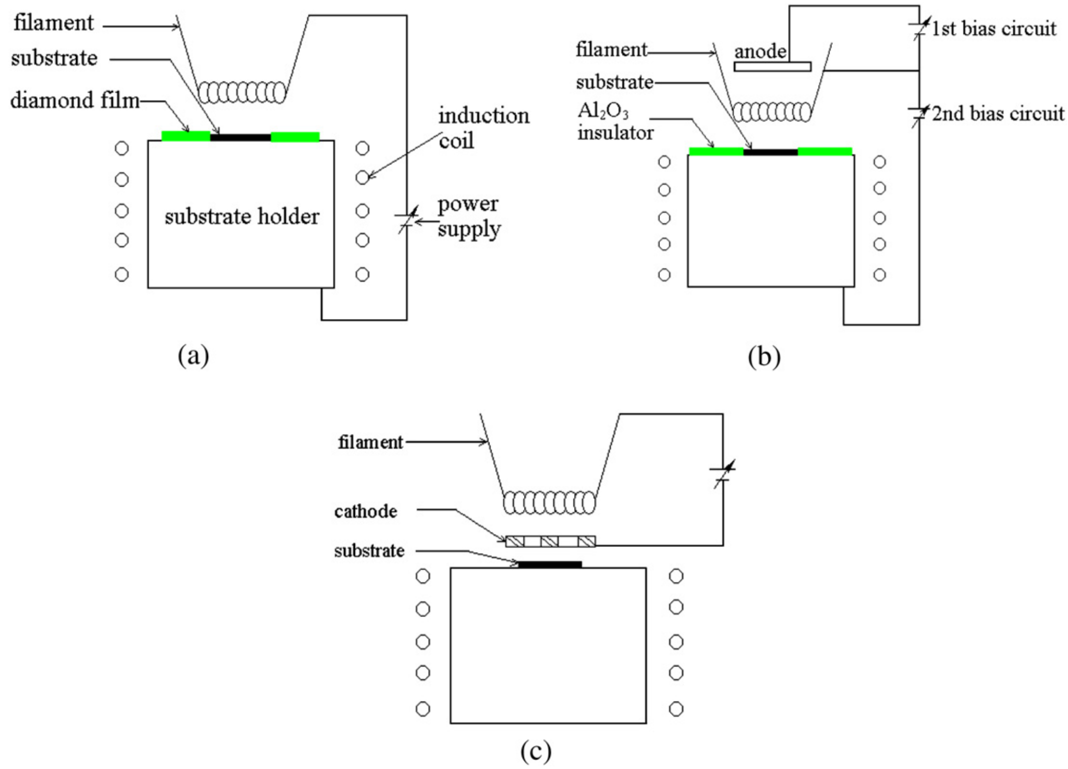

Fig. 1. Schematic representation of the electrode configurations applied to obtain bias enhanced nucleation: (a) Single-bias circuit; (b) double-bias circuit; (c) cathode above the substrate.

and CrN coated AISI type 316 stainless steel substrates were used. In the latter case the CrN film with a thickness of about 3 μm was deposited by IonBond Netherlands BV utilizing arc ion plating. Prior to bias pretreatment the substrates were sequentially cleaned in an acetone, a 2-propanol and a deionized water ultrasonic bath.

As shown in Fig. 1, three different electrode configurations for bias pretreatment were used, as will be described in the following sections. The power supply for the bias circuit was used in a current control mode. In the experiments the bias pretreatment time (t_b) as well as the bias current (I_b) and voltage (V_b) were varied in order to determine the conditions that yield the highest diamond nucleation density.

The substrate was taken out from the reactor after the nucleation stage. Scanning electron microscopy (SEM, JEOL JSM 6330 F) was employed to observe the diamond nucleation. Raman spectra were obtained with a Micro Raman Spectrometer (Renishaw system 1000). In this setup an Ar ion laser ($\lambda = 514.5$ nm) was used as the excitation source.

3. Results and discussion

3.1. Negatively biased substrate

The simplest configuration to apply a negative bias voltage to the substrate is a single-bias circuit, as shown in Fig. 1a. Diamond coated Mo plates are placed around the substrate in order to act as an electron emission source, promoting the discharge. SEM analysis indicates that using this electrode configuration a diamond nucleation density in the order of 10^9 cm^{-2} can be obtained on the whole area of a silicon substrate after a 20-minute bias pretreatment with a current of 175 mA and a voltage of 190 V. In the case of a Mo substrate bias enhanced nucleation occurred preferably at the edge of the substrate with a nucleation density of $7 \cdot 10^6$ cm^{-2} (Fig. 2), which is clearly less than obtainable on Si [26]. At the center of the Mo substrate the nucleation density was $2 \cdot 10^5$ cm^{-2}, similar to that what is obtained without bias pretreatment. It can be expected that the nucleation density at the edge of the substrate

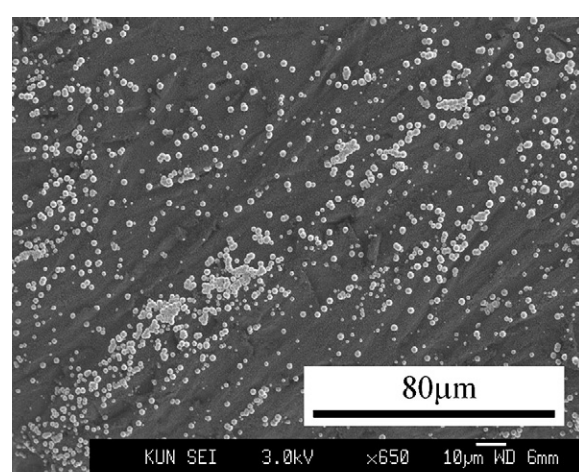

Fig. 2. SEM picture showing diamond nucleation near the edge of a Mo substrate ($CH_4/H_2 = 1\%$, $t_b = 50$ min, $I_b = 300$ mA, $V_b = 175$ V).