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Spontaneously detaching self-standing diamond films

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ABSTRACT

Spontaneously detaching self-standing diamond films were successfully fabricated using saturated hydrocarbon polymers as seeding source by hot filament chemical vapor deposition (HFCVD) technique. X-ray diffraction confirmed the crystal structure of diamond. The microcrystalline morphology of the diamond films was analyzed by atomic force microscopy (AFM) and scanning electron microscopy. Raman spectra from the front and back sides of the films show the characteristic diamond band at 1332 cm^{-1} and no carbide layer. EELS spectra also show the signature features of diamond. The use of hydrocarbon polymers as seeding source will improve the cost-effectiveness of the fabrication of diamond wafers by simplifying the fabrication process.

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1. Introduction

Diamond is a great candidate for electronic devices because of its extreme properties like high thermal conductivity, high mechanical strength and optical transparency [1,2]. These properties have triggered many studies on diamond including its application in electronic devices under extreme operating conditions where other materials like Si, Ga, As are not suitable. Self-standing diamond could be used as a window material for electromagnetic waves, a heat sink substrate for high power electric devices, a substrate for microelectromechanical systems and in surface acoustic wave devices [3–6]. Developing a simple and reliable technique for the production of large diamond wafers or self-standing diamond films is significantly important for the realization of diamond electronic and optoelectronic devices.

The methods to prepare self-standing diamond film involve chemical vapor deposition (CVD) and post-deposition processing. Among the CVD methods, Yan et al. [7], Zhong et al. [8] and Lu et al. [9] reported the fabrication of self-standing films by plasma CVD, arc-jet and the dc-plasma method, respectively. Extensive research has been carried out on various aspects of self-standing diamond formation on various types of substrates [10,11] including those that form carbide materials like Si, Mo and Ta. But all of the above processes require chemical etching or mechanical peeling in order to separate the film from the substrate. Furthermore, in order to obtain a uniform diamond film the interfacial carbide layer has to be eliminated. Hence, it would be a significant accomplishment to develop a method to synthesize self-standing diamond films on a substrate that does not form a carbide interlayer. Copper seems to be a promising candidate for such a substrate, but there are only few reports of diamond growth on copper [12–14]. Part of the problem is that diamond films grown on copper usually cracks during the cooling phase after deposition, due to the large mismatch in thermal expansion coefficients of diamond and copper (diamond ~1.0×10^{-6} K^{-1}, copper ~17.6×10^{-6} K^{-1}) [15,16].

It has been reported that diamond nucleation is enhanced by using polymers like phenyl carbyne [17,18] in the hot filament chemical vapor deposition (HFCVD) method, but the process resulted in the formation of isolated micro- or nano-crystals of diamond. However, a uniformly dense self-standing diamond film has not been achieved. Recently, it was reported that high seeding densities could be achieved by coating the substrate with a thin layer of polymer polyvinyl alcohol (PVA) doped with diamond nanoparticles [19]. We hereby report the fabrication of spontaneously detaching self-standing diamond films on copper substrates by hot filament chemical vapor deposition (HFCVD) technique using a mixture of saturated hydrocarbon polymers and nano diamond powder as seeding material.

2. Experimental procedure

Nano diamond powder (Alpha Aesar, particle size<1 μm) of amount 0.05 g of was mixed with 1 g of n-tetracosane (Alpha Aesar) and 1 g of n-octacosane (Alpha Aesar) and the mixture was melted in a glass beaker on a hot plate with constant stirring for 30 min and heated to a temperature of 120 °C at a rate of 15 °C/min. A small portion of this mixture was transferred on to a 14 mm diameter copper disk substrate with the help of a glass dropper and was allowed to cool to room temperature at a cooling rate of 15 °C/min.

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The disk was then placed in the HFCVD chamber. A gas mixture consisting of 0.3% methane in hydrogen and 99.7% of hydrogen at a constant pressure of 20 Torr and having a total gas flow of 100 sccm, was activated by a rhenium filament (8 cm in length and 0.5 mm in diameter) positioned at 7.5 mm above the substrate surface. The temperature of the substrate and the filament was kept at approximately 450 and 2500 °C, respectively.

The X-ray diffraction data were collected using an X-ray powder Diffractometer (SIEMENS, model D5000) with scanning speed of 4 °C/min and Cu *Kα* radiation ($\lambda = 1.5405$ Å) configuration. The Raman scattering spectrum was obtained on a triple monochromator (ISAJ-Y Model T 64000) with around 1 cm^{-1} resolution using the 514.5 nm line of Ar laser. Scanning electron micrographs were recorded using a JEOL JSM-7500F microscope and the TEM and EELS analyses were done in a LEO 922 OMEGA/200 kV. The AFM images were obtained with a Digital Nanoscope IIIA.

3. Results and discussion

Upon substrate cooling, the diamond films spontaneously detach from the substrate. Fig. 1 shows the photographic image of a self-standing diamond film along with the copper substrate employed that has a diameter of 14 mm. The size of the films is of the order of the diameter of the substrate.

The XRD diffractogram obtained from the front surface of the self-standing diamond films is shown in Fig. 2. It contains strong crystalline diamond diffraction peaks at ~43.5, 74.0, and 95.0°, corresponding to the {111}, {220}, and {311} planes, respectively. The XRD pattern obtained from the back side (not shown here) of the diamond films shows the same diffraction peaks but less intense that can be attributed to the small particle sizes in the initial growth stage of diamond.

The surface morphology of the films was ascertained by atomic force microscopy (AFM) and scanning electron microscopy (SEM). The self-standing films show an rms surface roughness of 80 nm for a scanned surface of $5 \times 5\ \mu m^2$ at an average height of ~100 nm from the film's topography (Fig. 3). The increase in surface roughness is due to the growth kinetics of the diamond film that favors the growth of some crystallites and inhibits that of others [20]. These effects result in a higher density of grain boundaries at the back side of the film. Fig. 4(a) and (b) shows the SEM images of the self-standing diamond films revealing the presence of well-faceted diamond microcrystals. Inset of Fig. 4(b) shows the high magnification SEM image of the film showing facets of the diamond crystals with a crystallite size of the order of 0.7–1.0 μm. Fig. 5 shows the cross-sectional SEM images of the self-standing diamond films for different deposition times showing an increase in the thickness which reaches to about 3 μm in 3 h revealing the deposition rate to be around 1 μm/h. They also show that the film morphology is polycrystalline with the standard columnar growth structure typical of CVD diamond (see Fig. 5). The nucleation density of the diamond films is enhanced by increasing the amount of nanodiamond powder added to the saturated hydrocarbon polymer mixture used for seeding.

Raman spectroscopy was employed to characterize the seeding material and the types of carbon materials present in the films. Fig. 6(a) shows the Raman spectrum of the saturated hydrocarbon polymer mixture coated substrate. The characteristic fundamental vibrational modes of saturated hydrocarbon polymer used as a seeding source to grow diamond films were observed between 1000 and 2000 cm^{-1}. The spectrum shows distinctive Raman contributions at 1063 cm^{-1}, 1133 cm^{-1}, 1171 cm^{-1}, 1296 cm^{-1}, 1418 cm^{-1}, 1441 cm^{-1} and 1463 cm^{-1}. These are attributed to C–C (carbon–carbon) stretching, and the CH_2 and CH_3 deformations [21], which arise from the straight-chain hydrocarbon structure of the seeding polymer [22]. Additionally, it shows a band around 1333.8 cm^{-1}

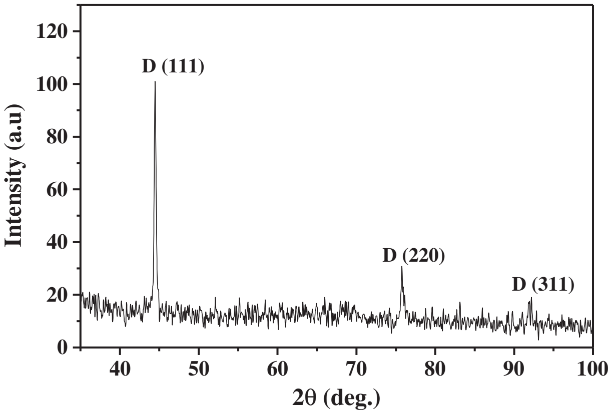

Fig. 2. XRD pattern of a self-standing diamond film.

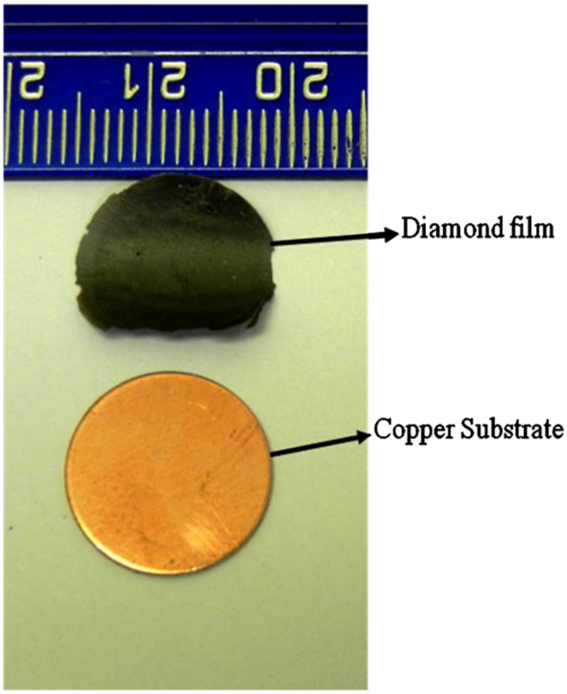

Fig. 1. Photographic image of a spontaneously detached self-standing diamond film.

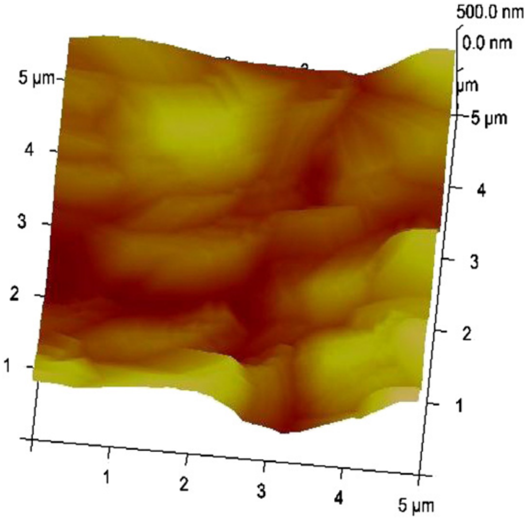

Fig. 3. AFM image of a self-standing diamond film.

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