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Synthesis and structural characterization of highly ⟨1 0 0⟩-oriented {1 0 0}-faceted nanocrystalline diamond films by microwave plasma chemical vapor deposition

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ABSTRACT

We report the synthesis and characterization of high-quality highly ⟨1 0 0⟩ oriented nanocrystalline diamond (NCD) films consisting of {1 0 0} nano-facets with a high growth rate of 2.6 μm/h. The NCD samples were grown on large (1 0 0) silicon wafers of 5.08 cm in diameter by employing $CH_4/H_2/O_2/N_2$ chemistries without the aid of bias for orientation, microwave power 3 kW and the substrate temperature about 700 °C using a 5 kW-type high-power microwave plasma chemical vapor deposition (CVD) system. The strong ⟨1 0 0⟩ preferred orientation is unambiguously demonstrated by a detailed crystallographic texture analysis and the conventional X-ray diffraction. Moreover, a detailed morphological characterization by the high-resolution scanning electron microscopy (SEM) and the atomic force microscopy (AFM), reveal that the growth surface consists of square (1 0 0) facets with an average size of about 60 nm and has a cylindrical microstructure. We demonstrate that the root-mean-square surface roughness as low as ~15 nm, measured by AFM on 1 μm^2 scan areas, can be obtained even for considerably thick (76 μm) films. The high quality of these films is confirmed by the Raman and Fourier-transformer infrared spectra. The high-quality smooth ⟨1 0 0⟩-oriented {1 0 0}-faceted NCD films may have high potential in mechanical, tribological and micro-electromechanical system (MEMS) applications.

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1. Introduction

It is well known that single-crystal diamond is difficult and costly to produce and polycrystalline diamond is generally rough. The current world-wide interest in nanomaterials and nanotechnology strongly stimulates the development of various nanostructured diamonds [1–15]. Among them, nanocrystalline diamond (NCD) thin films [12–14] are useful for micro- and nano-electromechanical systems (MEMS/NEMS) applications [5,6,12,16], because they posses smooth surface.

The morphology, texture or orientation, and microstructure of CVD diamond films are very important and have significant influences on applications concerning CVD diamonds, because diamond posses anisotropic physical and chemical properties along [1 0 0] and [1 1 1] directions [17]. High-quality NCD thin films were synthesized through a new nucleation process to obtain extreme high initial nucleation density, however, their grain size and surface roughness are dependent on the deposition time and film thickness due to the columnar growth [12,13]. Ballas or cauliflower-type NCD films have a rounded appearance and exhibit no faceting or evidence of columnar growth, even at thicknesses of several microns [18]. Wild et al. studied the dependence of the film morphology and texture on the methane concentration and deposition temperature [19]. They showed that only non-textured or ⟨1 1 0⟩ textured fine-grained diamond films were produced since for alpha, the ratio of the growth rates on {1 0 0} and {1 1 1} faces, approaching 3 the {1 0 0} facets vanish. The ⟨1 1 0⟩ preferred orientation or texture was commonly observed not only for large-grained polycrystalline CVD diamond

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films [19], but also for NCD [20], and for UNCD thin films [21]. Jiang et al. studied the effect of post-growth ion bombardment on diamond orientation and obtained [0 0 1]-oriented diamond crystallites with a lateral size of about 0.5 μm grown on a pre-deposited large-grained polycrystalline diamond film by employing negative bias [22]. For CVD diamond films, usually square {1 0 0}-faceted surface contains much fewer structural defects than the triangular {1 1 1}-faceted surface [19]. Hence, among various textured CVD diamond films, ⟨1 0 0⟩-orientated {1 0 0}-faceted diamond films are most desirable materials and they posses intrinsic smoothness. Nevertheless, neither NCD films of ⟨1 0 0⟩ texture (or orientation) nor NCD films consisting of {1 0 0} facets have been developed to date.

In this work, we report the successful synthesis of highly [1 0 0]-oriented smooth thick NCD films composed by {1 0 0} nanofacets and with columnar microstructure grown on large silicon wafers of 5.08 cm in diameter by using novel $CH_4/H_2/O_2/N_2$ chemistries without bias assistance for orientation and high-power microwave plasma CVD (MPCVD). The representative ⟨1 0 0⟩-textured {1 0 0}-faceted thick NCD film has high quality and low surface roughness. This is a promising alternative for mechanical, tribological and MEMS/NEMS applications. Moreover, the ability to grow ⟨1 0 0⟩-oriented {1 0 0}-faceted NCD film sheds light on possible growth mechanisms of CVD diamond films.

2. Experimental details

The [1 0 0]-oriented NCD films were grown using a 5 kW-type ASTeX PDS-18 MPCVD reactor on large (1 0 0) silicon wafers of 5.08 cm in diameter and 3 mm in thickness, which were pre-scratched with diamond power of 0–0.5 μm in size for nucleation enhancement. The growth parameters were microwave power of 3000 W, pressure 105 Torr, 4% CH_4 diluted in H_2 with 0.5% O_2 and 0.5% N_2 addition and substrate temperature about 750 °C.

SEM analysis was performed with a Hitachi S-4100 and a high-resolution SU70 SEM operated at 25 kV. AFM measurements were performed in tapping mode on a commercial setup Multimode, NanoScope IIIA, DI. Commercial SuperSharpSilicon^{TM} Nanosensors tips tip-cantilever system SSS-NCH with spring constant of k=42 N/m and tip apex radius less than ~2 nm have been used. AFM images were collected using various scan sizes; $5.0\times5.0\,\mu m^2$, $2.0\times2.0\,\mu m^2$, $1.0\times1.0\,\mu m^2$, $0.5\times0.5\,\mu m^2$ with a scan speeds <10 nm/s.

Micro-Raman spectra of the samples were taken using a Jobin Yvon T64000 Raman spectrometer excited by a 514.5 nm Ar^+ laser and by a 325 nm He–Cd UV laser. XRD characterization was performed in a high-resolution 4-circle difractometer X'Pert PRO MRD (PANalytical, The Netherlands) using Cu radiation. Symmetric diffraction patterns (conventional θ–2θ scans) were recorded in reflection geometry and using high intensity X-ray optics. In such configuration, the incident parallel X-ray direct beam, out of a primary Göbel mirror, encompasses both Cu $K_{\alpha1}$ and Cu $K_{\alpha2}$ components. In order to avoid the very intense diffraction peak from the Si substrate (4 0 0) peak (2θ=69.16°) we have deliberately introduced a small offset of ~1° relatively to the sample surface normal when collecting the θ–2θ patterns. Specifically for the determination of crystallite size in our NCD films, we have collected the diffraction peaks in high-resolution mode using a 4-crystal asymmetrical (2 2 0) Ge monochromator on the primary beam. In this configuration only the Cu $K_{\alpha1}$ radiation is used and the instrumental broadening is negligible. For the crystalline orientation study or in the pole-figure measurement, the diffracted beam intensity was measured for a χ range from −80° to 80° and a rotational angle, φ, range from 0° to 360° in 5° steps, the position of the detector is fixed at a specific reflection angle, 2θ, corresponding to diffraction from the diamond film (e.g., 75.3° for the {2 2 0} plane). By varying the angles χ and φ the sample can be rotated in any direction relative to the scattering wave vector. Pole densities are plotted in stereographic projection with polar and azimuthal angle χ and φ, respectively. The plane of the stereographic projection was chosen to be parallel to the sample surface.

3. Results and discussion

The ⟨1 0 0⟩-textured thick NCD films were grown on large (1 0 0) silicon wafers of 5.08 cm in diameter by a new growth process employing $CH_4/H_2/O_2/N_2$ gas mixtures and 3 kW high-power MPCVD. The NCD samples produced are dark grey, their top growth surfaces are flat, and the substrate sides or nucleation side (exposed after etching away the silicon substrates) are very smooth and mirror-like. These NCD samples are uniform in terms of large-scale morphology as observed by using routine optical microscope.

In order to characterize the crystalline quality and optical properties of the representative NCD film which will be discussed in detail here, the micro-Raman spectra are shown in Fig. 1. The appearance of a strong and sharp diamond peak at around 1333 cm^{-1} in the spectra evidences the diamond nature of the film. The full-width at half-maximum (FWHM) of the diamond Raman

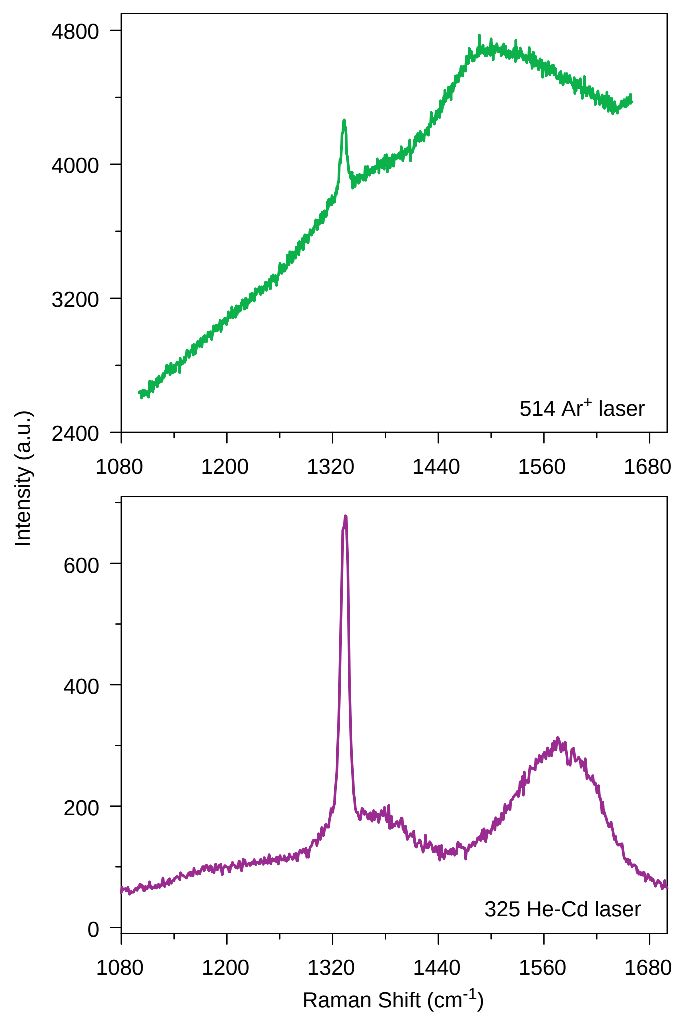

Fig. 1. Raman spectra excited by 514.5 nm Ar^+ green laser and 325 nm He–Cd UV laser of a representative NCD film.

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