



# Submicron-scale diamond selective-area growth by hot-filament chemical vapor deposition



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## ABSTRACT

Selective-area growth, which can be an alternative to ion implantation, is an important technology for processing diamond power devices. In conventional chemical vapor deposition (CVD), a portion of the metal mask (non-growing region) peels off and is unintentionally incorporated into the film; therefore, creating high-quality fine patterns is a great challenge. In this study, we developed a technique to fabricate fine structures on a submicron scale by employing hot-filament (HF) CVD. The mask pattern was kept intact during growth, and submicron-scale selective-area growth was realized. As a result, contamination-free metallic p<sup>+</sup> diamond films possessing a smooth surface morphology were successfully selectively grown.

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## 1. Introduction

Diamond semiconductors are promising candidates for high-power, low-loss, and high-temperature power devices because they possess superlative physical properties such as a high carrier mobility (4500 and 3800 cm<sup>2</sup>/V s for electrons and holes, respectively), low dielectric constant (5.7), high breakdown strength (10 MV/cm), and high thermal conductivity (22 W/cm/K) [1]. Diamond-based Schottky rectifiers [2], metal-oxide-semiconductor (MOS) field effect transistors (FETs) using hydrogen-terminated surfaces [3], metal-semiconductor (MES) FETs [4], and junction FETs [5] have been developed and their excellent switching capabilities have been demonstrated. As for diamond device processing, selective-area growth is believed to be the most effective technique for fabricating device architectures because impurity doping by ion implantation in diamond has not been established; the implantation damage partially transforms diamond carbon bonds (sp<sup>3</sup>) to energetically favorable sp<sup>2</sup> bonds, leading to graphitization [6]. Thus far, there have been several reports concerning selective-area growth utilizing metal masks. Patterned diamond films have been obtained using masks such as Ti/Au [7], Mo/Au [8], Ru/Au [8], and yttria-stabilized zirconia (YSZ) [9]. However, it has also been pointed out that mask patterns partially peel off when employing the conventional diamond growth method, e.g., microwave plasma-enhanced (MP) CVD [8,10]. Therefore, micron- to submicron-scale selective-area growth is a great challenge, especially for homoepitaxial growth. Dry etching processes of diamond have been proposed, but these methods typically cause surface roughing.

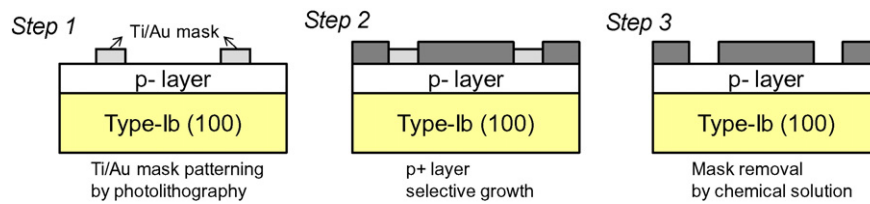
There are two important strategies to overcoming such shortcomings. One is the development of high-temperature stable masks and the other is suppression of the plasma-induced damage. In terms of the second strategy, we propose selective-area growth employing hot-filament CVD (HFCVD). Because HFCVD is simply based on the thermal decomposition of the growth species by the resistive heating of metal wires, the plasma damage to metal masks is expected to be neglected [11]. Our recent studies have also demonstrated that high-quality low-resistivity film growth (on the order of 1 mΩ cm) is possible by heavy B doping [12]. In this letter, by employing HFCVD and heavy doping techniques, selective-area growth of submicron-scale diamond film was performed. Using these methods, we obtained high-quality, contamination-free, fine-structure patterning.

## 2. Experimental

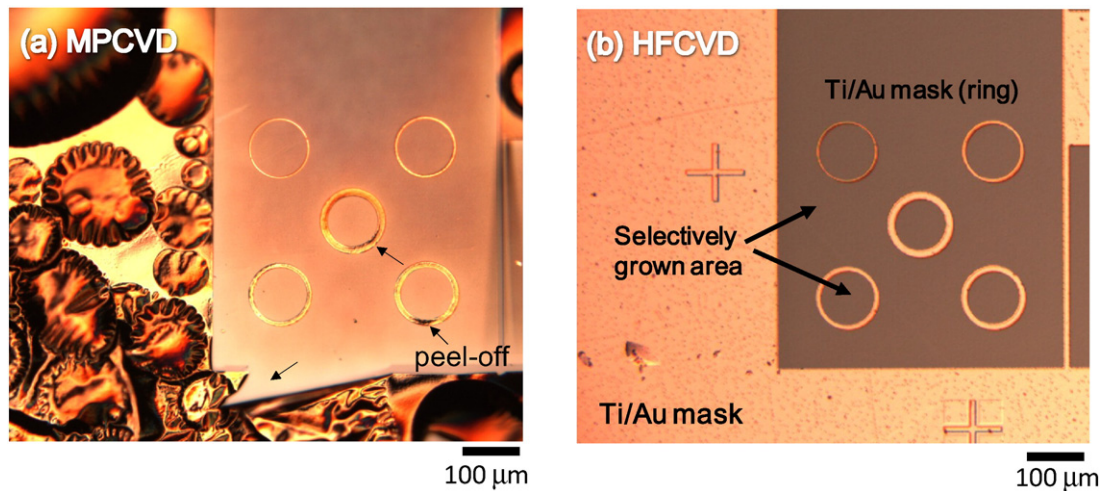
High-pressure high-temperature (HPHT)-grown type-Ib diamond crystals (100) with dimensions of 3 × 3 mm<sup>2</sup> were used as substrates. The surface roughness (Ra) was less than 0.1 nm after scribe polishing treatment. The misorientation angle from a vicinal (100) plane was approximately 2.5°. Prior to the diamond growth, substrates were chemically cleaned by a mixed acid solution of H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub> at 250 °C. Experimental procedures are schematically shown in Fig. 1. First, lightly boron-doped (p<sup>-</sup>) films with a thickness of approximately 3 μm were homoepitaxially grown by MPCVD (Step 1). After that, metal mask patterns were developed by standard photolithography using the lift-off process. Ti (30 nm thick)/Au (300 nm thick) bilayer metals were deposited by RF magnetron sputtering. Note that, we select the metal which

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**Fig. 1.** Experimental procedures of selective-area growth of diamond film using Ti/Au masks. (a) Ti/Au mask deposition by standard photolithography with a lift-off process, (b) selective-area p<sup>+</sup> growth where the Ti/Au mask was not deposited, and (c) mask removal by chemical solution.



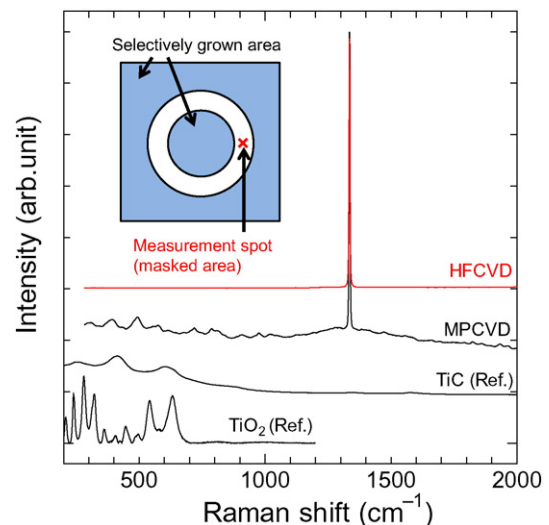
**Fig. 2.** Mask damage introduced during (a) MPCVD and (b) HFCVD film growth. These images were taken before use of the chemical solution to remove the masks (Step 2).

does not react with hydrogen in high-temperature ambient. In addition, to check the applicability for submicron-width selective-area growth, Ti/Au mask patterns were also developed by electron-beam (EB) lithography (JSM 6500F, JEOL Ltd.). Next, in Step 2, p<sup>+</sup> diamond films were selectively grown by HFCVD where the metal masks were not deposited. The detailed experimental conditions regarding to the HFCVD setup (filament configurations, substrate-filament distance, etc.) were explained in our previous paper [11]. The methane/hydrogen ratio (CH<sub>4</sub>/H<sub>2</sub>), boron/carbon atomic ratio (B/C), and total pressure were 3%, 0.3%, and 10 Torr, respectively. Total growth duration was 3 h. The 2% trimethyl boron (TMB: B(CH<sub>3</sub>)<sub>3</sub>) diluted with hydrogen gas was used for boron source. The filament temperature measured by pyrometer was ~2100 °C. Typical growth rate of these condition is ~0.2 μm/h. For comparison purposes, selective-area growth was also performed by MPCVD using the same mask patterns. The CH<sub>4</sub>/H<sub>2</sub>, input microwave power, substrate temperature, and growth time were 3%, 1000 W, 800 °C, and 2 h, respectively. Finally, patterned diamond films were developed after chemical solutions to remove metal masks (Step 3). In this step, diamond samples were carefully cleaned in an appropriate manner as following chemical treatments; the aqua regia, HCl + H<sub>2</sub>O<sub>2</sub> mixed solution, and H<sub>2</sub>SO<sub>4</sub> + HNO<sub>3</sub> mixed acid solution. The crystalline structures and quality were investigated by scanning electron microscopy (SEM), atomic force microscopy (AFM), secondary-ion mass spectroscopy (SIMS), Fourier transform infrared (FTIR) spectroscopy, Raman spectroscopy, and energy dispersive X-ray analysis (EDX).

### 3. Results and discussion

Diamond films were selectively grown by both MPCVD and HFCVD, respectively. Ti/Au metals successfully act as non-growing region for both growth techniques. Diamond nucleation was not occurred on metal surface, which might be due to the smooth roughness nature of deposited metal. However, the structural deformation of metal mask was frequently seen in the case of MPCVD. Fig. 2 shows optical

microscopy images taken immediately after Step 2 before application of the chemical solution. For MPCVD [Fig. 2(a)], the Ti/Au masks were entirely damaged and partially peeled from the substrates, as indicated with arrows. Faceted polycrystalline diamond grains were often observed where the mask patterns deteriorated; this probably comes from mask contamination. We also attempted growth at a much lower substrate temperature (~700 °C), but the mask patterns similarly degraded. We considered that the mask deformation may have been caused by an abnormal temperature increase due to microwave reflection or electron bombardment effects. In contrast, as for the growth by



**Fig. 3.** Raman spectra of diamond surface underneath Ti/Au masks. The measurements were performed after chemical solution to remove masks. The inset shows a schematic illustration of the measurement spot. The spectroscopy of TiO<sub>2</sub> was referred from the database R050591 of RRUFF project (<https://rruff-2.geo.arizona.edu/>).

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