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Atomically flat diamond (100) surface formation by anisotropic etching of solid-solution reaction of carbon into nickel

Kazuhiro Nakanishi, Hiroki Kuroshima, Tsubasa Matsumoto, Takao Inokuma, Norio Tokuda *

Graduate School of Natural Science and Technology, Kanazawa University, Kanazawa, Ishikawa 920-1192, Japan

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ABSTRACT

The morphology of diamond (100) surfaces etched by a solid-solution reaction of C into Ni was investigated. The etching was conducted at 900 °C under an Ar atmosphere. Diamond was selectively etched only under the Ni films. Atomically flat diamond (100) surfaces with step-terrace structures were formed on the etched regions. The step height was approximately 0.18 nm, which is equivalent to that of a double monatomic layer of (100) diamond. The step-edge lines were along the <110> directions. These results indicate that the diamond etching by the solid-solution reaction of C into Ni was anisotropic.

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1. Introduction

Diamond has superior physical and electronic properties, making it a wide-bandgap semiconductor material for next-generation high-power, quantum, bio/chemical electronic, and optoelectronic devices [1–11]. The fabrication of these devices requires diamond-etching processes. Typically, a plasma process such as reactive-ion etching is used to etch diamond because of its high chemical stability. However, the plasma process causes a deterioration of device performance by generating defects on the diamond surfaces and subsurfaces and by surface roughening [12–15]. Therefore, the development of non-plasma processes for etching diamond is essential. It is well known that diamond is etched by a solid-solution reaction between Ni and C because Ni has a high solid solubility of C [16]. Morofushi et al. reported that diamond under Ni films is selectively etched by the solid-solution reaction of C into Ni at 900 °C under an inert atmosphere [17]. Additionally, a catalytic etching of diamond by Ni nanoparticles and films at temperatures of around 900 °C under a hydrogen atmosphere has been reported [18–22]. However, the morphology of the selective etched diamond surfaces has not been investigated in detail.

Here, we report on the morphology of diamond (100) surfaces etched by the solid-solution reaction of C into Ni. In addition, the selective formation of atomically flat diamond (100) surfaces by an anisotropic etching of the solid-solution reaction is presented, and the mechanism is discussed.

2. Experimental

In this study, high-pressure and high-temperature (HPHT) synthetic Ib-type single-crystal diamond (100) substrates were used. The substrate surface was polished by Syntek Co., Ltd., in Japan. Each substrate was immersed in an acid mixture of H_2SO_4/HNO_3 (3:1) at 220 °C for 15 min in order to remove the surface contaminants. A Ni film was deposited onto the substrates by a vacuum evaporation method through a metal mask. The thickness of the Ni film was approximately 400 nm. Each sample was annealed using a rapid thermal annealing system at 900 °C for 5 or 15 min under an Ar atmosphere; the purity and flow rate of the Ar were 99.99995% and 1 L/min, respectively. After the Ar annealing process, graphite precipitated on the Ni film surface. Each sample was immersed in an acid mixture of H_2SO_4/HNO_3 (3:1) at 220 °C for 15 min in order to remove graphite and Ni.

The surface morphologies were observed by laser microscopy (LM; Olympus, LEXT OLS4100) using differential interference contrast (DIC) and by atomic force microscopy (AFM; Shimadzu, SPM-9700) using a contact mode.

3. Results and discussion

Fig. 1 shows LM images of a sample (a) before the annealing process and (b) after 15 min of annealing and subsequent removal of the deposited Ni film. The diamond was selectively etched under

* Corresponding author.
E-mail address: tokuda@ec.t.kanazawa-u.ac.jp (N. Tokuda).

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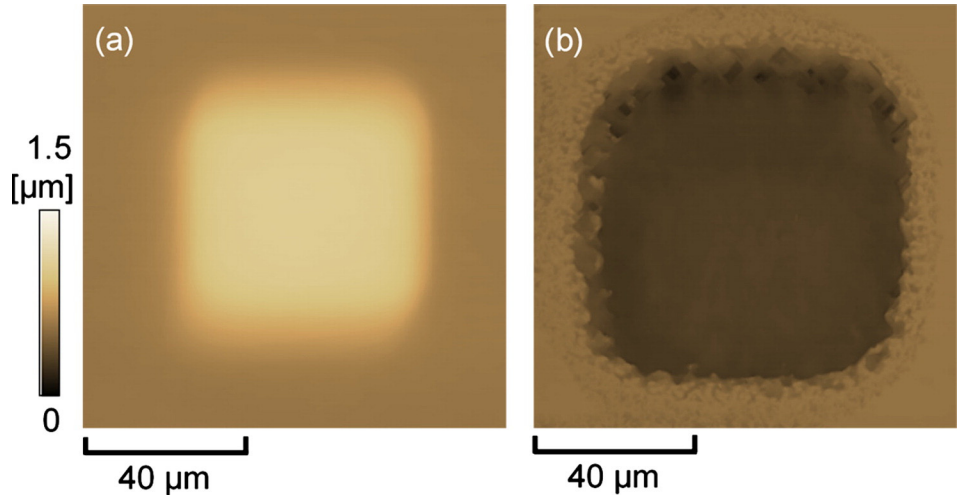

Fig. 1. LM images of a sample (a) before the annealing process and (b) after 15 min of annealing and subsequent removal of the deposited Ni film.

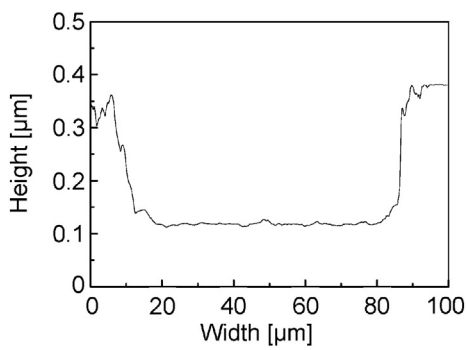

Fig. 2. A cross-sectional image of the pit shown in Fig. 1(b).

the Ni film, as shown in Fig. 1(b). On the basis of the cross-sectional image of the pit shown in Fig. 2, the etching depth of the diamond was approximately 260 nm, similar to that reported previously [4].

Fig. 3 shows LM-DIC images of the diamond (100) surfaces (a) before and (b) after annealing for 5 min and (c) 15 min. As shown in Fig. 3(b), the diamond (100) surface was roughened and formed numerous pits approximately 30 nm deep. The square shape of the pit indicates that the diamond etching by the solid-solution reaction was anisotropic. Such square pits on the diamond (100) surface are caused by anisotropic etching of the diamond by hydrogen plasma with/without O_2 addition at dislocations in diamond substrates and surface defects because of surface polishing [23–31]. In Fig. 3(b), the pit density ranges from 10^7 to 10^9 cm^{-2}, which is substantially greater than the typical dislocation pit density of HPHT diamond, i.e., approximately 10^5 cm^{-2} [32,33]. Therefore, the pits in Fig. 3(b) could be because of surface defects rather than dislocations. As shown in Fig. 3(c), the diamond (100) surface was flattened and exhibited no pits. The anisotropic diamond etching after the 15-min annealing process completely removed the surface defects from the diamond (100) surface as shown in Fig. 4.

Fig. 5 shows AFM images of (a) unetched and (b) etched diamond (100) surfaces outside and under the Ni film after the film was annealed for 15 min and after the removal of the Ni film, respectively. In Fig. 5(a), the diamond (100) surface exhibited numerous scratches and pits because of the surface mechanical polishing; this surface was similar to the diamond (100) surface before the annealing step. The RMS roughness of the diamond (100) surfaces was 0.08 nm. In contrast, the etched diamond (100) surface exhibited a step-terrace structure, as shown in Fig. 5(b). The RMS roughness within the diamond (100) terrace without steps was 0.03 nm, which is equivalent to that of step-free (111) diamond [34,35]. On

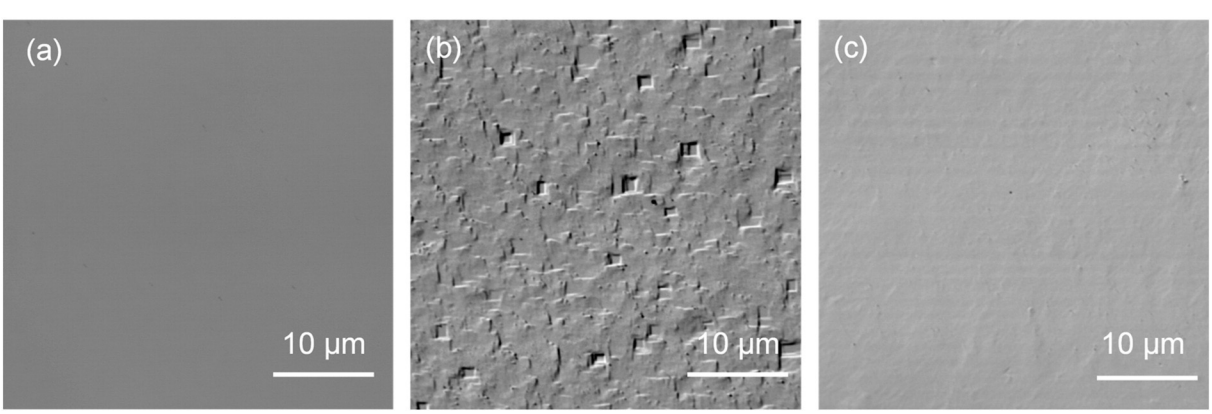

Fig. 3. LM-DIC images of the diamond (100) surfaces (a) before and (b) after annealing for 5 min and (c) 15 min.

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