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Sub-micrometer scale patterning on single-crystal diamond surface using focused ion beam and deep ultraviolet laser irradiations

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ABSTRACT

In this study, a novel method is proposed for patterning the surface of a single-crystal diamond in the sub-micrometer scale using a combination of focused ion beam (FIB) and deep ultraviolet (DUV) laser irradiations. The surface area of the diamond irradiated by the FIB was selectively machined using a low-power DUV laser, whereas the non-FIB-irradiated area was hardly machined. Hence, diamonds can be patterned at the sub-micrometer scale by selectively machining the FIB-irradiated area using lasers. To investigate the machining characteristics in this process, the effects of the FIB and DUV laser-irradiation parameters were investigated. Consequently, the shape of the DUV-irradiated area was improved by applying the gallium removal step; debris was not formed. In addition, a structure with a maximum depth of approximately 80 nm was fabricated. The damage density induced by the FIB irradiation was employed to determine the depth induced by the FIB; the depth obtained using this method is more than twice that of the heating technique in air. A concave structure was fabricated based on these results. The results indicate that the proposed method is effective for fabricating sub-micrometer-scale structures on diamond surfaces.

1. Introduction

Single-crystal diamonds are expected to be employed in various industrial equipment, such as optical devices, semiconductors, heatsinks, and cutting tools, because of their superior material properties. To fabricate diamonds for these applications, the nanometer- to micrometer-scale fabrication technique is required. However, diamonds cannot be easily fabricated using this technique because of their superior characteristics. Focused ion beam (FIB) direct machining is an effective method to fabricate these structures [1]. This method is used not only to prepare the samples for transmission electron microscope but also to fabricate various diamond-based structures such as field-emission tips [2], integrated solid immersion lenses [3], micro-cantilevers [4], and diamond tools [5,6]. However, the method is time consuming for diamond. In addition, the FIB irradiation induces non-diamond phases because the irradiated ions interact with the target atoms. The ions remain on the fabricated diamond structures and deteriorate their performance. The femtosecond laser is used for high-efficient machining [7]. However, the beam diameter is large for these fabrications.

By combining the ion irradiation technique with other methods, structures can be effectively fabricated with high efficiency and low damage. Although this fabrication method is largely applied to silicon and other materials [8–10], it can be applied to diamond. The non-diamond phase induced by the high-energy ion beams can be selectively etched using various methods including heating in an oxygen atmosphere [11], wet chemical etching [12,13], and electrochemical etching [14,15]. In previous studies, the heating technique was applied to fabricate structures with multiple depths [16] and improve diamond-cutting tools [17,18]. In these experiments, the FIB-irradiation-induced non-diamond phase was removed by heating in air. The maximum temperature until which diamond cannot be etched is 500 °C, with no change in the surface roughness. The maximum removal depth was limited to approximately 40 nm, which is determined using the damage density induced by the FIB irradiation. The threshold damage density for removal changes with respect to the temperature, and the maxi-

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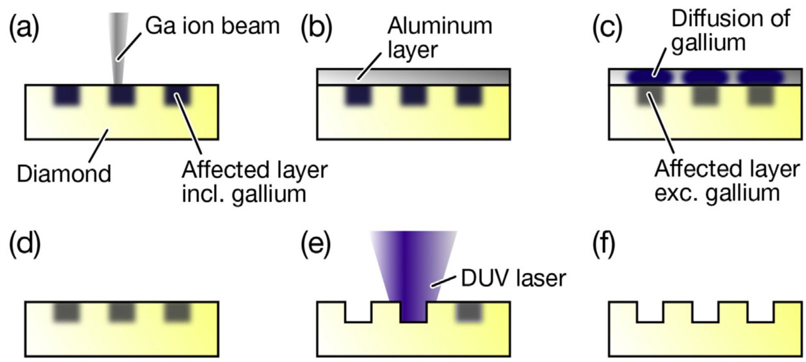

Fig. 1. Patterning method on a diamond surface using FIB and DUV irradiations. (a) Formation of the non-diamond phases using the FIB irradiation, (b) Deposition of the aluminum layer by sputtering, (c) Heating in vacuum to diffuse the gallium to aluminum, (d) Removal of the aluminum layer by etching in a solution of sodium hydroxide, (e) Irradiation of the DUV laser to remove non-diamond phase, and (f) Resulting structures on the diamond surface.

mum depth increases with the increase in the heating temperature. However, higher temperature cannot be applied because diamond devices undergo thermal damage. In addition, the fabrication technique should be more efficient if the surfaces are to be employed in various industrial fields.

In this study, a novel method is proposed for patterning the surface of a single-crystal diamond in the sub-micrometer scale using a combination of the FIB and deep ultraviolet (DUV) laser irradiations. The Raman-spectra results of the FIB-irradiated-diamond surface showed that the FIB-irradiated-diamond area was selectively machined using a low-power DUV laser, whereas the non-FIB-irradiated area was hardly machined. Hence, the surface can be patterned in the sub-micrometer scale using this method. The laser can induce photochemical reaction and high temperature locally; hence, different machining characteristics were obtained using this method compared to those of the heating-in-air technique. In this study, the fundamental machining characteristics and effectiveness of this method were investigated to apply it for sub-micrometer-scale patterning.

2. Patterning method using FIB and DUV laser irradiations

Fig. 1 shows the fabrication method of a structure using the FIB and DUV laser irradiations proposed in this study. This process comprises three steps: patterning using the FIB (FIB step, Fig. 1(a)), removal of gallium (Ga) by using heat treatment (Ga removal step, Fig. 1(b)–(d)), and pattern removal using the DUV laser (laser step, Fig. 1(e)). The surface of a single-crystal diamond is irradiated by Ga ions using FIB to induce a non-diamond phase in the diamond [Fig. 1(a)]. The surface of the diamond is covered with an aluminum (Al) layer obtained using a sputtering method [Fig. 1(b)]. It is heated in vacuum, where the Ga ions diffuse into the Al layer [Fig. 1(c)] [19]. It is subsequently cooled, and the Al layer is removed using a solution of sodium hydroxide (NaOH) [Fig. 1(d)]. The surface is irradiated using the DUV laser to machine the non-diamond phase selectively [Fig. 1(e)]. The concave patterns are fabricated along the FIB-irradiated area [Fig. 1(f)].

To investigate the machining characteristics, the effect of the machining conditions on the shape of the structure was evaluated in this study.

3. Experimental procedure

A Ib-type single-crystal diamond was used in this study. In the FIB step, the diamond was irradiated by the Ga ions on an area of $100 \times 100\,\mu m^2$ in an FIB facility (FB-2100, Hitachi High-Technologies Corporation) for studying various irradiation parameters. The chosen area is substantially wider than the spot size of the laser.

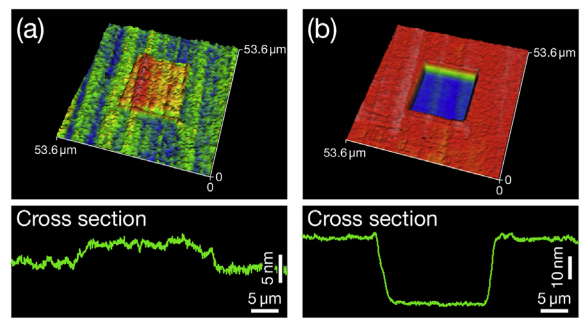

Fig. 2. Surface topography of the diamond surface irradiated by Ga^+ ions of energy 40 keV at a dose of (a) 10 mC/cm^2 and (b) 100 mC/cm^2, measured using a coherence scanning interferometer.

In the Ga removal step, the surface was covered with an Al layer of thickness > 100 nm using a sputtering method. It was heated to 500 °C in vacuum and maintained at this temperature for 1 h. The Al layer was then etched using a 10 wt.% NaOH solution.

In the laser step, the DUV with a continuous wave oscillation of wavelength 266 nm was irradiated using laser Raman spectroscopy (inVia Raman microscope, Renishaw Corporation) in a DUV laser facility (UW-1020A, Sony Corporation). The diameter of the laser spot was a few micrometers.

The crystallization of the diamond was measured using the laser Raman spectroscopy. The shape of the irradiated area was measured using an atomic force microscope (AFM: SPM-9500J2, Shimadzu Corporation) and a coherence scanning interferometer (NewView 7200, Zygo Corporation).

4. Results and discussion

4.1. Effect of radiation dose on the depth

In the method described in this paper, the non-sputtered condition is more effective for fabricating the structure because the sputtered condition is time consuming and increases the width of the structure. Hence, the effect of the dose on the depth of the structure after irradiating with the FIB was investigated.

Fig. 2(a) shows the surface topography of the FIB-irradiated diamond surface at a dose of 10 mC/cm^2 measured using a coherence scanning interferometer. The Ga^+ ions were irradiated on the surface of the diamond with an energy of 40 keV on an area of $20 \times 20\,\mu m^2$. The irradiated area was not sputtered and protruded at a height of 4 nm. This is because the non-diamond phases are

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