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### Single-crystal diamond tools formed using a focused ion beam: Tool life enhancement via heat treatment



DIAMOND RELATED MATERIALS

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#### 1. Introduction

Mechanical machining is an effective method of fabricating highprecision parts, high-precision molds, and micron- or nanometer-scale structures, with applications including lenses, molds, and biomedical system parts [1–3]. Mechanical machining can be used to fabricate high-spatial-resolution structures with high throughput, and a variety of workpiece materials can be used, whereas the shape and materials of the part are limited when using conventional photolithographic techniques [4]. Single-crystal diamond tools are generally used in ultra-precision machining because of their superior wear resistance, sharp cutting edge, and low affinity for machined materials. In mechanical machining, structures are typically fabricated by transcribing the tool shape, and therefore the shape accuracy of the tool itself, as well as that of the motion of the tool, are important factors in the fabrication process.

The shape of diamond tools is generally formed by mechanical polishing; however, fabrication of complex and minute tool shapes is difficult because of the hardness of the diamond. The use of a focused ion beam (FIB) is an effective means of fabricating micrometer and nanometer-scale tool shapes [5]. FIB machining of diamond at a

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#### ABSTRACT

We describe a technique to improve diamond cutting tools used in nanometer- and micrometer-scale machining and formed via focused-ion-beam (FIB) micromachining. Although FIB irradiation is an effective means of fabricating arbitrary miniature shapes in diamond cutting tools, FIB irradiation induces a non-diamond phase, as well as Ga ion implantation, in the irradiated area. This adversely affects the performance of the ultra-precision machining process, especially in terms of tool life and the quality of the machined surface. To eliminate the affected layer, we applied heat-treatment techniques and investigated the optimum thermal profiles. A temperature of 500 °C applied to the cutting tool provided optimal machining of nickel phosphorus. The tool life was significantly improved, and a tool life similar to that of a non-irradiated diamond tool was obtained. The quality of the machined surface was also improved markedly owing to superior tool wear and adhesion resistance.

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nanometer scale can be carried out using irradiation with gallium (Ga) ions via sputtering. However, this method leads to ion implantation, defects, and non-diamond phases in the tool, which adversely affect its machining performance. Therefore, further developments to FIB machining methods are desirable.

We have previously proposed an extraction method for implanted Ga ions in diamond tools via aluminum deposition and subsequent heat treatment in a vacuum [6]. The adhesion and roughness of the machined surface were improved using this method; however, the wear resistance of the tool was significantly lower than that of a nonirradiated diamond tool, due to the residual non-diamond phase (although it was improved compared with an FIB-irradiated tool). In this study, we applied heat treatment in air to remove the nondiamond phases from the surface of the diamond cutting tool. We investigated the optimum thermal profile for the diamond cutting tool and the machining performance of the tool.

#### 2. Experimental details

#### 2.1. Diamond etching experiment

Diamond etching was used to form sub-micron-scale and micronscale fabrications via non-diamond-phase formation, and subsequent

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etching of those non-diamond regions. The non-diamond phases included amorphous and graphitic carbon phases, and were formed in diamond via irradiation with high-energy ions. An area can be selectively etched using a number of methods, including heating in an oxygen atmosphere [7], wet chemical etching [8,9], and electrochemical etching [10,11]. Wet chemical etching and electrochemical etching may etch the shank and brazing materials in addition to the diamond surface because the FIB micromachining is carried out on the diamond material brazed on the tool shank. Heating in oxygen was therefore applied to form features on the diamond tool.

Several attempts have been made to etch ion-irradiated diamond by heating it in air to fabricate a thin diamond wafer [7]. Non-diamond phases were first formed using MeV ion irradiation, and these regions were then selectively etched (i.e., they were oxidized to form CO and  $CO_2$ ). The temperatures during these processes were over 550 °C, which is in excess of the thermal budget of the brazing and shank materials. The brazing melts at temperatures in excess of 550 °C, and when the diamond itself is etched at temperatures in excess of 600 °C, the tool shape accuracy and roughness may be compromised. For these reasons, the maximum process temperature was 500 °C.

A type lb (100)-oriented single-crystal diamond was rinsed in ethanol, and then irradiated using an FB-2100 FIB facility (Hitachi High-Technologies Corporation). Following irradiation, it was mounted in an electrical furnace (KM-160, Advantec Toyo Kaisha, Ltd.). The temperature inside the furnace, where the atmosphere was air, was increased to the preset temperature and maintained there for 4 h, followed by slow cooling. The diamond tool was then rinsed again. The shape of the irradiated area was measured using a white-light interferometer (NewView 7300, Zygo Corporation).

#### 2.2. Machining experiment

The machining experiment was carried out using an ultra-precision cutting machine (ROBONANO  $\alpha$ -0*i*B, FANUC Corporation) with 1-nm resolution of the translation axes. A high-speed shaping method was employed using a shuttle unit model B [12], which can machine a surface with a maximum reciprocating speed of 5.4 Hz, a stroke width of 200 mm, and a mean cutting speed of 130 m/min.

Table 1 shows the machining conditions. Single-crystal diamond tools with rounded corner radii of 0.5 mm were used for the experiments. The rake angle was 0° and the clearance angle was 10°. The workpiece material was an electroless nickel phosphorus (NiP) layer, which is commonly used for fabricating precision molds, deposited on stainless steel. Machining was carried out at a mean cutting speed of 130 m/min, a depth of cut of 3 µm, and a feed rate of 10 µm per stroke, under wet conditions at 23 °C. The cutting fluid was Metal Work HS (JX Nippon Oil and Energy Corporation). The wear of the tool rake face was measured using a white-light interferometer. The shape of the cutting edge was transcribed to oxygen-free copper following machining, and the transcribed tool shape was measured.

#### 3. Results and discussion

#### 3.1. Diamond etching with heat treatment

Fig. 1 shows the change in the depth of the irradiated area following 4 h of heating, plotted as a function of the process temperature. The diamonds were irradiated using 40-keV Ga ions at doses of  $7.9 \times 10^{16}$  cm<sup>-2</sup>,  $7.9 \times 10^{17}$  cm<sup>-2</sup>, and  $3.0 \times 10^{19}$  cm<sup>-2</sup>, with a beam current of 21.5 nA over an area of 20 µm × 20 µm. Prior to irradiation, a carbon layer that was approximately 10-nm-thick was deposited on the diamond surface to avoid electrostatic charging during irradiation. Following irradiation, the irradiated area protruded by 1 nm at a dose of  $7.9 \times 10^{16}$  cm<sup>-2</sup>. The irradiated areas were then sputtered at doses of  $7.9 \times 10^{17}$  cm<sup>-2</sup> and  $3.0 \times 10^{19}$  cm<sup>-2</sup>; the sputtered depths were 51 nm and 2499 nm, respectively.

Table 1 Machining conditions.

Tool	Single-crystal diamond
Work materials	NiP
Mean cutting speed (m/min)	130
Depth of cut (µm)	3
Feed per stroke (µm)	10
Rake angle (°)	0
Clearance angle (°)	10
Lubrication method	wet

The increase in depth following heat treatment depended on the ion dose. The depth was measured before heating,  $d_b$ , and after heating,  $d_a$ , as shown in Fig. 1, and therefore was determined not only by the etch depth of the irradiated area, but also by the etch depth around the irradiated area. The increase in depth was very small at low temperatures, and negative values were obtained. The irradiated area was not deeply etched at 400 °C at low doses, and only the carbon layer around the irradiated area was etched. A large amount of re-deposited material was observed around the irradiated area at high doses, where sputtering occurred. The re-deposited material increased the apparent depth after irradiation, and etched following heating even at 400 °C. This indicates that the etch rates of the carbon and re-deposited material was etched area, and led to a large decrease in depth values.

The change in depth following heat treatment increased with temperature. The changes at 500 °C and 550 °C were similar, and were in the range of 30–40 nm for low-doses. However, at 600 °C, the change in depth was greater than 60 nm. At 600 °C, not only the non-diamond phase but also the crystalline diamond phase were etched; the large depth difference resulted from the multiple etching effects of these phases. Although the change in depth was small at a dose of  $3.0 \times 10^{19}$  cm<sup>-2</sup>, the depth of the irradiated area increased with heating. Therefore, the irradiated area was etched both with and without sputtering. These results indicate that a process temperature of 500 °C is optimal for diamond cutting tools in terms of the etch rate and damage to the diamond tools. No further change in depth occurred for heating times longer than 4 h at 500 °C, indicating that the non-diamond phase was completely etched within 4 h.

To evaluate the machining performance, the heating technique was applied to a diamond cutting tool. Fig. 2 shows scanning electron microscope (SEM) images and the surface topography of diamond cutting tools irradiated using an FIB. The tool rake and flank faces were irradiated with 40-keV Ga ions at a dose of  $6.2 \times 10^{16}$  cm<sup>-2</sup>. To avoid adverse effects due to carbon deposition, Ga ions were irradiated at lower beam current of 4.7 nA without carbon deposition so that electrostatic charging did not occur. The irradiated area is shown in Fig. 2(a), where the appearance changed due to the presence of Ga ions and the



**Fig. 1.** Changes in the depth of the irradiated area as a function of the process temperature (i.e., the difference in depth before and after heating).

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