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Two-step polishing technique for single crystal diamond (100) substrate utilizing a chemical reaction with iron plate

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ABSTRACT

A polishing technique for diamond substrates is described. It is a two-step process, consisting of (1) a rough processing step using carbon reaction with an iron plate at elevated friction temperatures, and (2) a finish processing step using hydroxyl (OH) radicals generated by a Fenton reaction between an iron plate and hydrogen peroxide solution. We analyzed the processing characteristics using optical interferometric microscopy, atomic force microscopy and transmission electron microscopy. Experimental results show that the surface roughness of a diamond substrate is markedly improved to an atomic-scale smoothness, and that a damage-free diamond surface can be fabricated with 5 h of polishing. These results provide useful information for obtaining atomically smooth diamond substrates.

1. Introduction

Owing to its outstanding material properties, such as high hardness, wide band gap, good thermal conductivity, good carrier mobility, a low coefficient of friction, and high chemical stability, diamond is now attracting attention as a cutting tool [1] and as a power semiconductor device material [2–4]. Especially, in order to fabricate an ideal diamond device, atomically smooth and damage-free diamond substrates are essential. However, such substrates are relatively difficult to prepare because of its high mechanical hardness and chemical inertness.

To satisfy the demand for diamond substrates in device fabrication, we proposed a surface finishing technique for diamond substrate utilizing an iron plate in a hydrogen peroxide (H_2O_2) solution [5]. This technique uses catalytically generated hydroxyl radicals (OH radicals), which are generated by the decomposition of H_2O_2 on the iron surface. This technique yields an atomically smooth diamond surface. As compared to the abrasive processes that involve friction heating of the diamond, material removal efficiency was extremely low because the removal mechanisms were based on the chemical reaction under wet conditions at low temperature. So, a pre-stage polishing technique prior to the finishing technique is strongly needed for preparing an atomically smooth diamond surface with high efficiency.

To prepare an ultra-smooth diamond surface with a high crystallographic nature, various surface preparation techniques, such as ion beam figuring [6–8], laser processing [9–11], and plasma etching [12,13], have been developed and demonstrated. However, these techniques can generate subsurface damage and roughness on the processed diamond surface, introduced by ion bombardment or thermal attack. Moreover, the cost of their processing equipment is very high, because these processes require specific circumstances, such as high vacuum, high temperature, and various gas ambients.

In addition to these processes, thermochemical polishing has been studied and demonstrated. Yoshikawa et al. developed a thermochemical polishing technique for chemical vapor deposition (CVD) diamond substrates using a hot metal plate at temperatures higher than 750 °C in controlled ambient environments, and succeeded in obtaining a smooth diamond surface with low pressure and relatively low speed [14,15]. Zaitsev et al. demonstrated a thermochemical polishing technique for CVD diamond films using a vibrating polishing plate at a temperature of 1070 K, which provided a smooth diamond surface [16]. Furushiro et al. proposed precision polishing of diamond by utilizing a thermochemical reaction with copper. It is a very simple method of bringing a diamond tool into contact with a copper plate heated in air [17]. Iwai and co-workers proposed and developed the dynamic friction polishing (DFP) method for diamond, using a SUS304 stainless steel plate at high pressure and high relative speed of both the sample and the plate in the atmosphere. A removal rate of 0.94 mm^3/min was reached [18]. However, in these polishing processes, it is difficult to control the process parameters, such as the polishing pressure, the relative speeds of the polishing plate and sample, the gas pressure, the degree of vacuum, the process-ambient, the heating condition of the polishing plate, and so on.

From the results of these studies on thermochemical polishing of diamond substrates, we have decided to simply conduct the polishing of diamond substrates using an iron plate under atmospheric conditions

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at room temperature as a pre-stage polishing technique. This technique is simple, low-cost, and based on the thermochemical reaction during friction between a diamond surface and a metal plate with high carbon solubility at elevated temperatures.

The aim of this study is to examine the feasibility of removing and smoothing a single-crystal diamond substrate by a two-step polishing technique utilizing the chemical reaction with an iron plate in an atmosphere (first-step polishing; rough processing) and in an H_2O_2 solution (second-step polishing; finish processing).

2. Experimental method

Fig. 1 shows a schematic of the experimental setup for two-step polishing of diamond substrate. Fig. 1(a) shows the first step polishing setup, which includes an iron plate placed on a rotating table. The sample was attached to the sample holder, which was placed on the iron plate at a controlled load of 3000 g. The polishing pressure was applied onto the sample by adding a suitable weight. The rotating speeds of the table and sample holder were set to 300 rpm and 500 rpm, respectively. The polishing time was 2 h. During the first stage, the sample and the iron plate directly contacted each other, and rubbed together under atmospheric conditions at room temperature. The thermochemical reaction between the interfaces of the sample and the iron plate occurred by friction heat. Thus, many scratches and much evidence of damage on the pre-processed diamond were effectively removed by carbon diffusion and graphitization at elevated temperatures during friction.

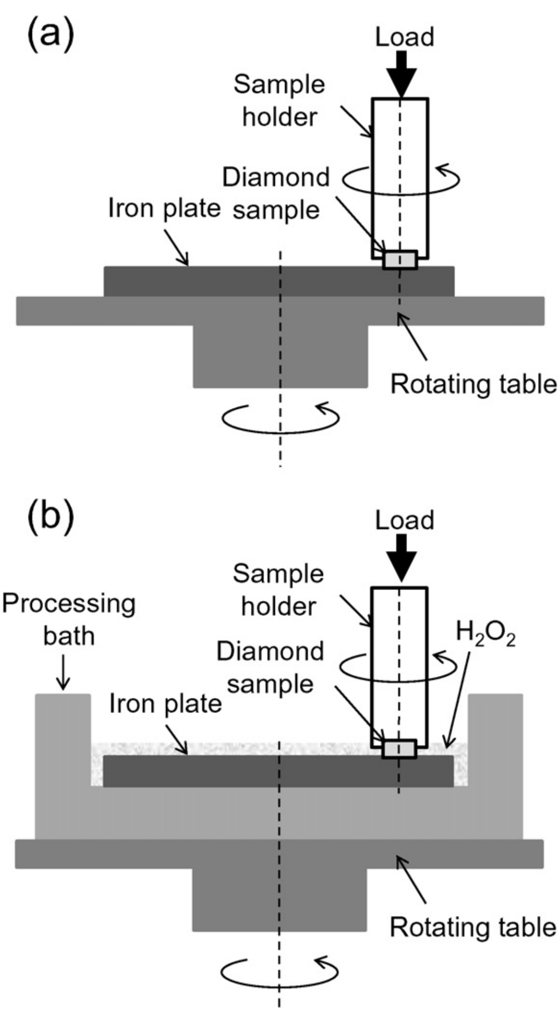

Fig. 1. Schematic diagram of experimental setup for (a) first-step polishing with an iron plate in atmospheric circumstances, and (b) second-step polishing with an iron plate under H_2O_2 circumstances.

Fig. 1(b) shows the second-step polishing setup. An iron plate was set in a processing bath, which was placed on a rotating table. The processing bath was filled with an H_2O_2 solution. The sample was attached to the sample holder, which was placed on the iron plate at a controlled load of 3000 g. The rotating speeds of the table and sample holder were set to 120 rpm and 250 rpm, respectively. The polishing time was 3 h. The second-step polishing uses catalytically generated hydroxyl radicals (OH radicals), which are generated by the decomposition of H_2O_2 on the iron plate. First, iron is immersed in the H_2O_2 solution, and ionized into ferrous iron (Fe^{2+}). The reaction occurs as follows:

$$Fe^{2+} + H_2O_2 \rightarrow OH\bullet + OH^- + Fe^{3+}. \quad (1)$$

This reaction is well known as the Fenton reaction. Generated OH radicals (OH•) are a strong oxidation species. Thus, OH radicals react with the topmost area on the sample substrate to form a modified layer. This modified layer needs to be mechanically and chemically removed. Finally, an atomically smooth and damage-free diamond surface was considered to be obtained.

In this study, single-crystal (100) high-pressure high-temperature diamond substrates (Sumitomo Electric Type-Ib) were used as a sample. Sample was a square of 3 mm with thickness of 1.5 mm. Before the experiment, the removal track was fabricated by a polishing method with a small iron tool [5]. The material removal rate was determined by measuring the cross-sectional profiles of the removal track before and after polishing. The surface morphology and depth of removal track on the diamond surface were measured and evaluated by phase-shift interferometric microscopy (Zygo Corp., ZYGO NewView 7300) and atomic force microscopy (AFM; Shimazu Co. Ltd., SPM-9700), respectively. Moreover, the crystallographic nature of the polished sample was observed by high-resolution transmission electron microscopy (TEM; Tecnai F20, Philips Electron Optics).

3. Results and discussion

3.1. Surface morphology and material removal rate at each polishing stage

We applied the proposed methods to smooth a diamond substrate using the polishing equipment illustrated in Fig. 1. Fig. 2(a) and (b) shows a height distribution image and the slope X map of the pre-polished diamond surface, Fig. 2(c) and (d) shows that of the first-step polished diamond surface, and Fig. 2(e) and (f) shows that of the second-step polished diamond surface. Slope X map shows the differential image in the horizontal direction. As observed in Fig. 2(a) and (b), many scratches, resulting from mechanical polishing with diamond abrasives, existed, and the removal track formed by the small iron tool can be seen on the center of the diamond substrate. The depth of the removal track was 354.1 nm. Additionally, growth sector boundaries [19] on the diamond substrate are clearly visible on both sides of the diamond surface. As shown in Fig. 2(d), although growth sector boundaries on the first-step polished diamond surface were clearly visible, it is confirmed that the sector boundary parts shown in the lower right and upper right of Fig. 2(f) have clearly disappeared. Moreover, the scratches on the diamond surface were removed markedly over the entire area. To investigate the surface morphological changes on the growth sector boundary before and after polishing, the obtained diamond surface was observed in detail. Fig. 3 shows magnified images of the pre-processed, first-step polished, and second-step polished diamond surface (of the circle in the lower right of Fig. 2(b), (d), and (f), respectively). The measurement area is 696 μm × 514 μm. In Fig. 3(a) and (b), it is revealed that surface roughness improved from the carbon diffusion reaction, but the boundary of that sector on the diamond substrate was clearly observed. The step height of the sector boundary part shown in Fig. 3(b) was 5.5 nm. On the other hand, the boundary of the sector part was obviously removed by second-step polishing, as shown in Fig. 3(c). This result implies that the topmost areas of the