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Full Length Article

Thermochemical micro imprinting of single-crystal diamond surface using a nickel mold under high-pressure conditions

Yuji Imoto, Jiwang Yan*

Department of Mechanical Engineering, Faculty of Science and Technology, Keio University, Hiyoshi 3-14-1, Kohoku-ku, Yokohama 223-8522, Japan

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ABSTRACT

Single-crystal diamond is an important material for cutting tools, micro electro mechanical systems, optical devices, and semiconductor substrates. However, the techniques for producing microstructures on diamond surface with high efficiency and accuracy have not been established. This paper proposes a thermochemical imprinting method for transferring microstructures from a nickel (Ni) mold onto single-crystal diamond surface. The Ni mold was micro-structured by a nanoindenter and then pressed against the diamond surface under high temperature and pressure in argon atmosphere. Results show that microstructures on the Ni mold were successfully transferred onto the diamond surface, and their depth increased with both pressure and temperature. Laser micro-Raman spectroscopy, transmission electron microscopy (TEM) and electron energy loss spectroscopy (EELS) analyses indicate that a graphite layer was formed over the contact area between diamond and Ni during pressing, and after washing by a mixed acid, the graphite layer could be completely removed. This study demonstrated the feasibility of a cost-efficient fabrication method for large-area microstructures on single-crystal diamond.

1. Introduction

Single-crystal diamond has the highest hardness, excellent thermal conductivity, optical property, electric insulation and chemical stability. It is now used as an ultraprecision cutting tool material, and is expected to be applied to semiconductor substrates, micro electro mechanical systems (MEMS), and optical devices in the future. For these applications, cost-efficient precision micromachining of single-crystal diamond is demanded. Currently, laser machining, focused ion beam (FIB) machining, and reactive ion etching (RIE) methods are used for fabricating microstructures on diamond [1–4]. However, these methods lead to very high processing cost and low productivity, which limits the applications of diamond.

Although diamond is extremely hard, and difficult to be mechanically processed, it might be thermochemically machined in an easier manner. It has been reported that the tool wear is very severe when cutting transition metals, such as Ni, Co, Ti, Fe, and so on, using a diamond tool [5–9]. A thermochemical reaction occurs between diamond and transition metals, where carbon atoms in diamond are diffused into the transition metals. In recent years, surface processing of diamond utilizing the aforementioned thermochemical reaction has gathered extensive attention [10–16]. For example, a patterned Ni layer was used to react with diamond to provide surface patterning in a large area [17] without necessity of using strict vacuum environment. However, it is time-consuming to perform Ni coating/patterning on diamond, and to remove the Ni coating after the thermochemical reaction.

In this study, we propose a novel thermochemical imprinting process for single-crystal diamond. A micro-structured Ni mold surface is pressed onto diamond surface under high temperature and pressure to transfer the microstructures from the Ni mold to the diamond workpiece through interfacial thermochemical reaction. The shape, depth, and distribution of the microstructures on the Ni mold are flexibly and precisely controlled by a nanoindentation system, and the thermochemical reaction between the mold and the diamond workpiece is controlled by pressure and temperature for various processing depth.

This paper presents experimental results of microstructure formation behavior on single-crystal diamond in the thermochemical imprinting process. The influence of pressure and temperature on the imprinting depth will be examined. The feasibility of cost-efficient fabrication of high-precision microstructures on single-crystal diamond by the proposed method will be demonstrated.

2. Process mechanism

Fig. 1 illustrates schematically the process mechanism for thermochemical imprinting. First, micro dimples are formed on a Ni

* Corresponding author.
E-mail address: yan@mech.keio.ac.jp (J. Yan).

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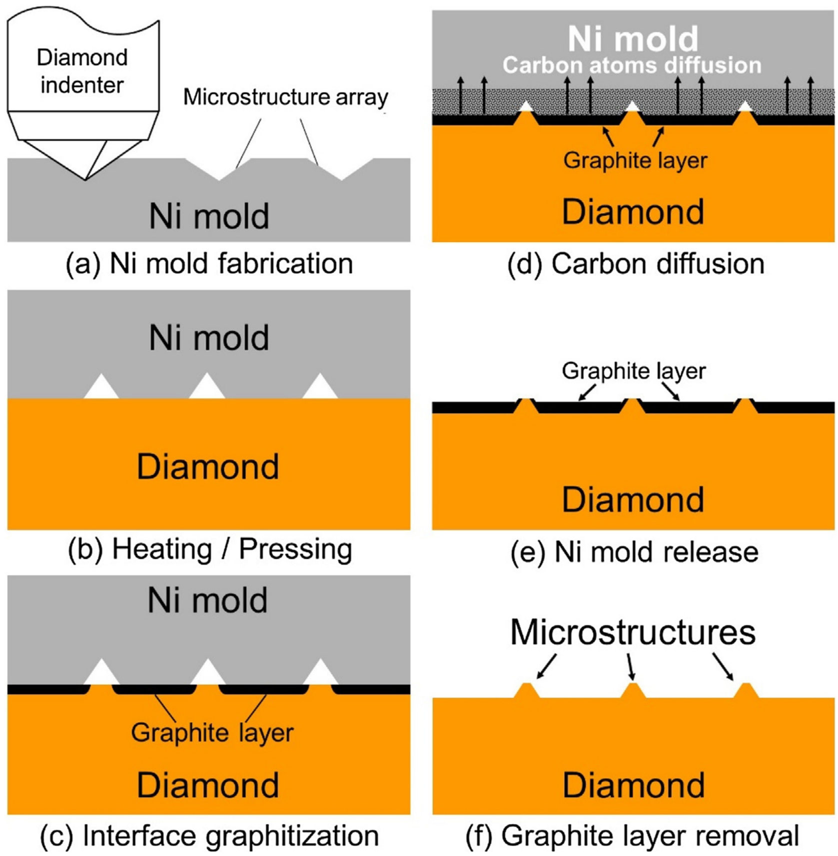

Fig. 1. Schematic diagram of micro imprinting process based on thermomechanical reaction.

mold surface by a diamond indenter (a). After the Ni mold gets into contact with a diamond substrate, they are pressed together and heated (b). At high temperature, the diamond surface will be easily wetted by nickel. Under the catalytic effect of nickel, diamond tends to undergo a phase transformation from diamond to graphite [18,19], and a thin layer of graphite is formed at the Ni-diamond interface (c). Subsequently, the graphitic carbon at the Ni-diamond interface will diffuse into the nickel mold (d). The rise of temperature reduces the energy barrier for graphitization and also improves the solubility of carbon in nickel. As a result of continuous graphitization and diffusion, the diamond surface is locally machined deeper and deeper. After this, the sample is cooled down and the Ni mold is removed from the diamond substrate by immersing the sample in hydrochloric acid HCl (e). Finally, the graphite layer is removed by a mixed acid, and microstructures are obtained on the diamond surface (f).

3. Experimental procedures

3.1. Materials

Single-crystal diamond square pieces with a dimension of 3.0 mm × 3.0 mm × 1.5 mm were used as specimen. The top planes of the diamond samples were (100). The diamond surfaces were then polished to a mirror finish by a cast iron scaif. The Ni mold material, provided by Nilaco Co., Japan, contains 99.6% Ni, 0.20% Mn, 0.07% Fe, 0.05% Si and 0.008% C by mass. The Ni molds were formed from a Ni rod to a dimension of ϕ6 mm × 2 mm by a wire electric discharge machine, Mitsubishi MV2400S (Mitsubishi Electric Co., Japan). The surface of the cut Ni pieces were polished to mirror surface by a polishing machine, EJW-400IFN-D (Engis Japan Co., Japan). Concentrated hydrochloric acid (HCl), nitric acid (HNO_3) and sulfuric acid (H_2SO_4) provided by Junsei Chemical Co., Ltd, Japan were used as solvents for washing the processed samples.

3.2. Micro-structuring of molds

A nanoindentation system, ENT-1100a (Elionix Inc., Japan), which was originally designed for evaluating material mechanical properties, was used to fabricate microstructures on the Ni molds. The indentation force and loading rate can be precisely controlled, enabling generation of micro dimples with various depths. Unlike a cutting process, the temperature increase of the diamond indenter is extremely small during nanoindentation, thus the thermochemical reaction between diamond and the Ni mold can be suppressed. Additionally, the shape of microstructure is decided by the shape of the diamond indenter. A photograph of an indented Ni mold is shown in Fig. 2(a), where the microstructures were formed in the mold center within an area of 1 mm × 1 mm. Fig. 2(b) and (c) show a conical dimple array and a pyramidal dimple array formed on the Ni mold. It should be noted that pileups might be generated around the dimples during indentation, especially when indenting deep dimples. However, in the subsequent press molding stage, the pileups can be pressed and flattened by the diamond surface, thus have little effect on the imprinting process.

3.3. Molding conditions

Fig. 3 illustrates the high-precision molding machine GMP211 (Toshiba Machine Co. Ltd., Japan) used in this study. The machine can control precisely the molding temperature and force in different atmospheric gases. Heating is realized by infrared lamps and temperature was monitored by a thermocouple with ±1 °C accuracy. The pressing force ranges from 0.2 kN to 20 kN with a res-