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High-rate ultrasonic polishing of polycrystalline diamond films

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ABSTRACT

We report on fast polishing of polycrystalline CVD diamond films by ultrasonic machining in a slurry with diamond particles. The material removal mechanism is based on diamond micro-chipping by the bombarding diamond particles subjected to action of an ultrasonic radiator. The treated samples were characterized with optical profilometry, SEM, AFM and micro-Raman spectroscopy. The developed method demonstrates the polishing rate higher than those known for mechanical or thermo-mechanical polishing, particularly, the surface roughness of 0.5 mm thick film can be reduced in a static regime from initial value $Ra \approx 5$ μm to $Ra \approx 0.5$ μm for the processing time as short as 5 min. No appearance of amorphous carbon on the lapped surface was revealed, however, formation of defects in a sub-surface layer of a few microns thickness was deduced using Raman spectroscopy. The polishing of a moving workpiece confirmed the possibility to treat large-area diamond films.

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1. Introduction

Polycrystalline and single crystals diamond films and wafers produced chemical vapor deposition (CVD) are currently accessible for a variety of applications, including optics, electronics, thermal management, tools and biology [1–4]. In many cases the grown material should be polished down to the tolerances required by a specific application, that is difficult in view of the extreme hardness and chemical inertness of diamond. As the consequence, the treatment procedure can be a rate-determining step in production of the diamond components. The polycrystalline diamond (PCD) films, advantageous for their large dimensions, possess, however, high roughness, typically from a few to tens microns for the films of a fraction millimeter thick, the grain size and roughness increasing with the thickness [5]. In comparison with polishing of single crystal (SC) diamond [6–8] the classical mechanical treatment of the PCD films is more difficult due to random grain orientation and the absence, for this reason, of "soft" polishing directions present in SCs. Also, PCD area can often exceed 20–30 cm^2, much larger that for SC [9–11], this demanding a higher load. Even if SC and PCD sizes are comparable, the grain misorientation strongly slows down the PCD treatment. The PCD materials require novel processing techniques, and a number of alternative methods for diamond polishing have been developed and tested (see [9,11–14] for a review).

The polishing techniques based on different chemical and physical principles, include, in particular, (i) chemical-mechanical polishing [15–17], (ii) thermo-mechanical etching by metals actively dissolving carbon, such as manganese powder [18] or molten lanthanum-nickel eutectic [19], (iii) electrical discharge machining [20–22], (iv) laser smoothing [23–26], (v) ion beam polishing [27–29], (vi) abrasive liquid jet polishing [30]; (vii) float polishing in a water-based slurry containing colloidal silica [12]. More recently, a dynamic friction polishing process, that integrates mechanical, thermal and chemical interactions into a single process, has been developed [3,31,32]. The procedure is based on diamond dissolution in a metal polishing wheel (steel, Ti), rotated at high speed, owing to high temperature achieved by the workpiece upon the polishing. Yet, there is a need in development of other effective high-rate polishing/smoothing methods that do not require sophisticated equipment. Here we report on polishing of PCD films using ultrasonic machining (USM) in a diamond slurry, the material removal being realized via surface damage caused by impact action of diamond microparticles in intense ultrasonic wave. A high rate polishing is demonstrated, reducing the initial surface roughness by an order of magnitude in a few minutes. While an ultrasonic polishing of interior surfaces of diamond drawing dies is used for many decades, and USM of a variety of brittle materials is well developed [33], the polishing of PCD films by USM techniques has not been described so far, to our best knowledge.

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2. Experimental

A translucent ≈0.5 mm thick polycrystalline diamond film was grown in CH_4/H_2 gas mixture on a polished Si substrate (3 mm thick, 63 mm in diameter) using a microwave plasma CVD system [5], operated at 2.45 GHz frequency under the following process parameters: 2.0% methane concentration in the mixture, total gas flow rate 1000 sccm, pressure 100 Torr, microwave powder 5000 W, substrate temperature 730 °C as measured by a two-color pyrometer (Williamson). The freestanding diamond wafer was obtained by chemical etching of the substrate, and 8 × 8 mm squares were laser cut for the test. The thermal conductivity of ≈1760 ± 140 W/m·K was evaluated for this sample with a laser flash technique [34]. In addition one test for polishing with moving workpiece was performed for 30 mm diameter and 0.5 mm thick diamond disk of black diamond with dominant (100) grain orientation, the polished track extending across the whole disk.

The phase purity of the samples was analyzed with micro-Raman spectroscopy using LabRam HR800 (Horiba Jobin Yvon) instrument at 473 nm excitation wavelength by focusing Nd:YAG laser beam in a spot ca. 1 μm in diameter at the growth side of the film. The surfaces morphology and roughness of the virgin and treated samples were characterized with optical microscopy, scanning electron microscopy (SM-7001F, JEOL), atomic force microscopy (Ntegra Spectra, NT-MDT Ltd), and optical profilometry (NewView 5000, ZYGO).

The rough growth side of the diamond films was lapped by ultrasonic machining (USM) via the process schematically displayed in Fig. 1a. The vibrating tool (sonotrode) placed in close proximity to the workpiece mounted on a X-Y positioning table, vibrates with ultrasonic frequency of 22 kHz. A water-based slurry of 20/28 μm synthetic diamond grit (diamond mass concentration of 17 g/*l*) is fed to the interaction zone into the narrow gap between the tool face and the workpiece. The tool was a 1 cm diameter rode made of stainless steel 16X16N3MAD (Fe-16.0 Cr-3.0Ni-1.2Cu-1.1Mn) with a moderate hardness (HV = 3200 MPa) and high ultimate strength (1620–1810 MPa) that provided tolerable (low) wear rate of the tool face. The tool hammers abrasive diamond particles against the diamond film causing microcracks, micro-chipping, cavitation in the slurry and the material removal (Fig. 1b). More details on the USM abrasive process of the hard–brittle materials can be found in the reviews by Thoe et al. [33] and Zhang et al. [35]. The sonotrode ultrasonic vibration amplitude up to ≈40 μm, and acoustic power up ≈50 W at preload of 2.5 N were used in the experiment. The gap of ~30–40 μm between the tool and workpiece was close to the diamond grit size. The appearance of cavitation bubbles in the radiator-sample gap at high enough ultrasonic power indicated the beginning of the treatment process. The workpiece translation with respect to the radiator allows USM process for large area. Here, the treatment of smaller (8 × 8 mm) diamond sheets with size similar to the radiator diameter, was performed in a static regime, without X-Y table moving.

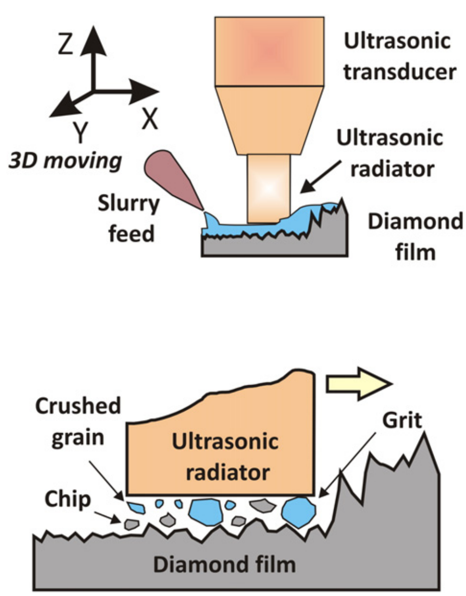

Fig. 1. The schematic of ultrasonic assisted lapping of diamond surface in a flow of microdiamond-based slurry (a), and zoom-in tool-workpiece contact zone (b).

3. Results

3.1. SEM observations

The topography on the growth side of a virgin diamond film displays well faceted crystallites with predominant (110) orientation and mean size of ~90 μm, some grains having dimensions up to ≈200 μm, as seen on SEM inspection (Fig. 2a). A striking difference in the relief was observed after merely 1 min USM treatment: the crystallite's apexes are mostly removed or rounded, and the surface becomes essentially smoothed (Fig. 2b). However, many intergrain valleys remain intact, as can be clearly seen at a higher magnification (Fig. 2c). After the longer, 5 min treatment process, the surface shows even better smoothing, although some most deep cavities between crystallites in the primary relief, still survive (Fig. 2d).

A severe damage of the crystallite surface is revealed both of the tips and facets (Fig. 2c), presumably taking place via microchopping. This leads to a certain roughening of the initially smooth facets, but, simultaneously, to a reduction in global roughness due to the tips and edges smoothening. The preferential removal of the tips can be ascribed to stronger impact effect of the grit squeezed in narrow gap between the tool (radiator) and the protrusions, and, possibly, to a more rapid removal of debris.

3.2. Optical profilometry

The average surface roughness *R*a ≈ 5 μm and peak-to-valley value R_z up to 27 μm were measured with the optical profilometer on area of 0.9 × 0.7 mm for the virgin samples. The surface reliefs for the films polished for 1 min and 5 min are compared in Fig. 3. A "rounding" of surface features and pitting of the facets are observed already after 1 min treatment process (Fig. 3a,b), this resulting in a slight decrease of the roughness to Ra ≈ 1.77 μm as measured on area of 0.9 × 0.7 mm. The size of imaged area was chosen to contain many (> 50) grains in order to get enough statistics. The more pronounced smoothing, down to *R*a ≈ 0.51 μm, was revealed after 5 min polishing (Fig. 3c,d), this being in accordance to the SEM observation (Fig. 2b). Similar correlations of the profilometry and SEM images have been found for all the treated samples. To assess the PCD removal rate one of the samples was polished for 5 min on one half only, leaving the rest surface intact, and the resulting step of ≈17 μm has been measured by optical profilometer. The achieved material removal rate ≈ 200 μm/h is several times higher compared to ~30–50 μm/h reported by Zhou et al [36] for super-high speed polishing with low-carbon stainless steel used as the polishing plate material. Much lower removal rates are known for pure mechanical polishing of polycrystalline diamond plates with comparable thickness, in particular, Chen et al. [37] measured the reduction for *R*a from the initial 4.5 μm to ≈0.56 μm for 20 h, while Ollison et al. [38] reported on decrease of the average surface macro-roughness *R*a from 9 μm to ~1 μm in 36 h of mechanical polishing of 10 × 10 mm^2 samples.

The grain boundaries contribute significantly to the roughness of the USM-polished surfaces. If the relief measurements were restricted by a single grain region, with exclusion of the grain boundaries, the roughness as small as *R*a ≈ 240 nm on the measured 70 × 52 μm [2] area was revealed after 5 min processing (Fig. 3e,f), that seems to be a

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