



## Research Letters

# Micro-grooving into thick CVD diamond films via hollow-cathode oxygen plasma etching

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## Abstract

Hollow-cathode oxygen plasma etching was proposed as a promising means to make micro-texturing into CVD diamond coatings. Oxygen ions and electrons were confined in the hollow-cathode to have higher ion and electron densities in the order of  $10^{17}$  to  $10^{18} \text{ m}^{-3}$  in the inside of hollow-cathode. Quantitative plasma diagnosis proved that direct reaction of oxygen atom or radicals with carbon in the diamond coating should drive the reactive ion etching (RIE) process. A diamond-coated WC (Co) disc specimen was employed to describe the RIE-behavior with aid of stainless steel mask with the line width of  $100 \mu\text{m}$ . Surface depth profile measurement as well as the Raman spectroscopy demonstrated that a micro-groove was precisely etched into the diamond film as a sharp-edged profile. © 2016 Society of Manufacturing Engineers (SME). Published by Elsevier Ltd. All rights reserved.

**Keywords:** CVD diamond coating; Micro-grooving; Hollow-cathode oxygen plasma; Plasma etching; High etching rate

## 1. Introduction

Micro-electro-mechanical or nano-electro-mechanical devices (MEMS/NEMS) was first developed by the so-called silicon technology for fabrication; their structural components were also made of fragile silicon with relatively low stiffness and tribological properties [1]. Diamond coating by CVD (Chemical Vapor Deposition) was selected as the second candidate material for MEMS/NEMS, owing to high stiffness and more wear toughness [2]. As a promising bio-sensor, the nano-grained CVD diamond film was found to be preferable to equip various kinds of polymer accepters to proteins [3]. In parallel with direct use of diamonds as a structural member, a protective diamond coating on the stamping die was also used to imprint the micro/nano-patterns onto metallic sheets [4]. In the above appli-

cations, the micro-texturing method becomes an essential key technology to reform the original diamond film to the integrated actuators, sensors and die-unit.

In the literature, the reactive ion etching of silicon by lithographic process was first employed as a standard means to fabricate the MEMS-pattern [5]. Since then, there have been developed various surface processing technologies; e.g. the femto-second laser micro-machining [6], the focused ion beam method (FIB) [7], and  $\text{CF}_4\text{-O}_2$  plasma etching using the aluminum masks [8]. Although each of them has its own merits and demerits; the promising micro-texturing process into CVD-diamond films must satisfy three features: sharp-edge configuration with high aspect ratio, short-term process, and, large-area green manufacturing.

In the present study, the hollow-cathode oxygen plasma etching is proposed as a promising means to make micro-texturing into diamond coatings as the eco-processing not to use the hazardous gas [9]. First, its system configuration is stated with comments on the role of hollow-cathode. A

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quantitative plasma diagnosis is employed to prove that high ion and electron densities are attained by this hollow-cathode oxygen plasmas. On-line monitoring of the CO-peak intensity with time via spectroscopy is also used to describe the chemical etching process in the present micro-texturing. CVD-diamond coated WC (Co) disc specimen with the diameter of 10 mm is used as a substrate, and, micro-line patterned stainless sheet with the thickness of 50  $\mu\text{m}$ , as a mask for micro-grooving. The initial diamond coating thickness is nearly constant by 19  $\mu\text{m}$ . The diamond micro-groove formed on the WC (Co) substrate has a square cross-section of  $97 \times 19 \mu\text{m}^2$  with the sharp edge nearly normal to the substrate. The etching rate reaches to 9.3  $\mu\text{m}/\text{h}$ , eight times faster than that by  $\text{CF}_4\text{-O}_2$  system in [8]. The above micro-grooving textures are formed on the whole surface of WC (Co) substrate surface just in correspondence to the original line-pattern with the width of 100  $\mu\text{m}$ .

## 2. Experimental procedure

### 2.1. Hollow-cathode oxygen plasma system

A hollow-cathode oxygen plasma system was shown in Fig. 1. This system consists of the vacuum chamber, the RF-generator, the DC-generator, and a hollow metal tube. The chamber was neutral in electricity. The RF dipole electrodes and DC-bias worked independently to generate RF and DC plasmas, respectively. Different from the conventional plasma generator, the present system did not have mechanical matching box. The input and output powers were automatically matched by frequency adjustment around 2 MHz. In particular, the DC-bias was also applied

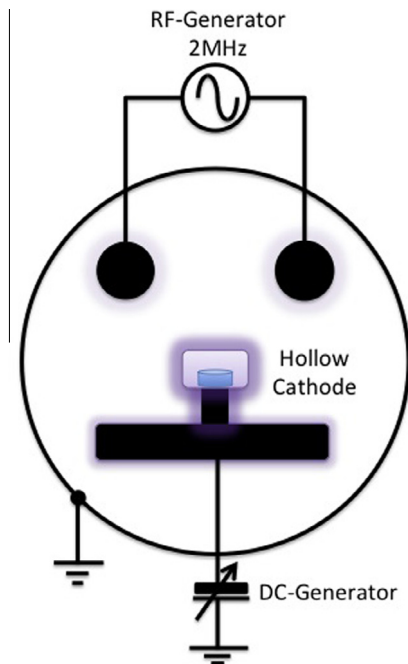


Fig. 1. Schematic view of the hollow-cathode oxygen plasma etching system.

to this hollow tube. Oxygen gas was introduced to the chamber. Due to the pressure gradient between the inside of chamber and the metal tube, the oxygen gas flew into a metal tube. The oxygen plasma was selectively ignited in this metallic hollow by switching on the RF electrode and the DC bias.

The quantitative plasma diagnosis instruments were used to characterize the current plasma state during the etching process. The Langmuir probe (Impedans, Co. Ltd.) was first used to measure electron and ion densities in the hollow cathode oxygen plasma. The probe was directly inserted into this hollow-cathode plasma to monitor the change of plasma state by varying the processing conditions. For an example, the pressure was varied to investigate its influence on the electron and ion densities. Optical Emission Spectroscopy (OES by Hamamatsu, Co. Ltd.) was also used to measure the emissive light spectrum from the hollow-cathode plasma and to analyze the generated species in the plasmas. A detector was mounted on the quartz window of chamber to transfer the detected signals to computer for analysis. In the similar way to the above Langmuir probe measurement, this OES worked to make on-line monitoring on the generated species during the etching process.

### 2.2. Observation and measurement

Optical microscopy (NICON, Co. Ltd.) was used to observe the specimen surface before and after etching. Surface profilometer (Keyence, Co. Ltd) was employed to measure the surface profile of etched micro-groove. The Raman spectroscopy (Renishaw, Co. Ltd.) was also utilized to prove that the whole CVD diamond was removed even at the bottom of micro-grooves.

### 2.3. Masking technique

A stainless steel sheet with the thickness of 50  $\mu\text{m}$  and the line width of 100  $\mu\text{m}$  was utilized as a mask to make micro-grooving into the CVD diamond coating. This masking sheet was directly placed and glued onto the diamond film surface.

### 2.4. Specimens

WC (Co) disc specimen with the diameter of 10 mm and the thickness of 5 mm was prepared as a substrate material for CVD (Chemical Vapor Deposition) coating of diamonds. The coated CVD diamond film was around 19  $\mu\text{m}$  thick.

## 3. Experimental results and discussion

### 3.1. Quantitative plasma diagnosis on the hollow-cathode oxygen plasmas

The pressure ( $p$ ) was varied to investigate its influence on the electron and ion densities or Ne and Ni. Fig. 2(a)

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