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A simple method to eliminate the surface defects of diamond particles



Feng Teng ¹, Zhong Bao ¹, Peng Zhang, Guozhi Zhang, Chengshi Gong, Caitian Gao, Jiangtao Wang, Xiaojun Pan, Erqing Xie *

School of Physical Science and Technology, Lanzhou University, Lanzhou 730000, People's Republic of China

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1. Introduction

Diamond has received much attention in recent years due to its excellent physical and chemical properties, such as high mechanical strength, high thermal conductivity, wide band gap, high intrinsic resistivity, high electron and hole mobility [1], the highest breakdown field, transparent over a wide range of wavelength and so on. It was widely used in industry, arts, medicine and other related field. Synthesized diamond, which has extremely similar characteristics with natural diamond, has been widely used in cutting tools, heat spreaders, pump seals, mechanical and electrical materials. Various techniques to synthesize diamond particles were pursued by numerous research groups [2–4]. But the commercially available diamond particles have many surface defects, which limit its industrial applications. Defects on diamond surface can have a profound effect on certain properties of the diamond, enabling these properties to be tailored to the specific needs of important applications. Properties sensitive to defects include optical properties, thermal properties, and electronic properties. However, the most important aspect of the effect is that its mechanical properties become poor. The reasons are as follows. Firstly, the atoms existed in the defects can easily detach from the crystal structure because of the incomplete crystal structure caused by dangling bonds. Secondly, the bond between the dangling atoms and the internal atoms would be broken because huge stress and lattice distortion could be generated when an external force was applied to the particle. Thirdly, the dangling atoms have high activity, which can easily react with other substances with strong

ABSTRACT

Defects on diamond surface have a profound effect on certain properties of the diamond, enabling these properties to be tailored to the specific needs of important applications. A simple and practical method is described to repair the diamond particles with surface defects. The low grade diamond particles were repaired by hotfilament chemical vapor deposition with low carbon concentration in feed gases, and the defects on the surfaces disappeared because of the simple film formation and homoepitaxial growth mechanism during the CVD process. This efficient and simple method provides a way to extend its useful life during the actual application.

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oxidation, especially the oxygen. So the damage would be larger and deeper during the using process, which was named as "defective proliferative effect". In order to solve these problems, many efforts are focused into the preparation of fine diamond particles. In recent decades, there are some methods of growing diamond from gas phase at comparatively low temperatures and pressures were created, including the hot-filament chemical vapor deposition (HFCVD) [5], microwave plasma chemical vapor deposition (MPCVD) [6], combustion-flame methods [7], DC arc plasma jet [8] and so on [9]. And the growth rate of the diamond film is regulated by adjusting the concentration of carbonaceous active groups in reaction atmosphere, which can realize expected growth on the crystal surface. However, metal and the material with lower melting point used as the support matrix are not suitable to place in the reaction chamber for MPCVD, DC arc plasma iet and combustion-flame method. Therefore, in all the above mentioned methods, HFCVD is the most promising option to grow fine diamond particles with the advantages of simple device and few restrictions.

In this letter, the commercially available diamond particles $(5-50 \mu m, prepared by crushing larger grains with defects on the surface) have been treated by HFCVD with different carbon concentrations in the reaction atmosphere, and the mechanism using CVD method to repair the defects on the surface was discussed in detail. The effect is obvious, which can be observed from the SEM images and Raman spectra.$

2. Experimental

The original diamond particles are crushing diamond particles (5–50 µm, provided by Yangzhou Hanjiang Grinding Tools, Jiangsu Province, China). The apparatus used in our experiment is JZ.CD8A-450 Hot Filament Chemical Vapor Deposition equipment (Juzhi Technology

^{*} Corresponding author. Tel.: +86 9318912616; fax: +86 9318913554.

E-mail address: xieeq@lzu.edu.cn (E. Xie).

¹ F. Teng and Z. Bao contributed equally to this work.

Development Co., Ltd., Liao Ning, China) [10]. Tungsten wires with diameters of 0.60 mm twisted to a 9-turn coil of 3 mm diameter were used as the filaments. The wires need to be carbonized in a mixture of methane and hydrogen so as to be stable during the diamond growth process. Before the experimental process, the silicon substrates ultrasonically cleaned in ethanol and acetones were positioned right under the filaments with a close to monolayer diamond particle film placed on it. The filament temperature was 2200 °C measured by an infrared thermometer (Raytek) during diamond deposition. The substrate temperature, measured by thermocouple below the silicon substrate, was maintained at 800 °C. The distance between the filaments and the substrate was set to 8 mm. Hydrogen (99.99%, 200 standard cubic centimeters per minute (SCCM)) and methane (99.99%, 2 SCCM) (CH₄/H₂ volume ratio <1%) as reaction gases were introduced into the chamber and the total pressures were maintained at 2000 Pa. The base pressure is 9.0×10^{-4} Pa during the experiment. And the treatment process lasted 18 h. As comparisons, medium concentration carbon source $(CH_4/H_2$ volume ratio 1–2%) and high concentration carbon source $(CH_4/H_2 \text{ volume ratio } > 2\%)$ were used to study the influence of carbon source concentration on reparation of the surface defects.

The morphology of the samples was investigated using field emissionscanning electron microscope (FE-SEM, Hitachi S-4800). Raman spectra were recorded at room temperature using a JY-HR800 Raman spectrometer. The samples were excited by a 532 nm laser line from the yttrium aluminum garnet laser.

3. Results and discussion

3.1. Surface morphology and Raman spectra

The initial morphology of the diamond particles was shown in Fig. 1a. It is obvious that the commercially available diamond particles have a large number of surface defects and uneven edges. This morphology strongly influences the properties of diamond surfaces including surface conductivity, surface wettability and the adsorption. Fig. 1b displays the high resolution SEM image of the diamond particles in detail. The surface defects show a layered stack, which mainly relates to the crystallographic orientation. Due to the existence of the defects on the surface, flakes and scraps can be easily produced while being used, which leads to a short working life of the related tools. The diamond particles with smooth surface and little defects were prepared by repairing the surface defects using HFCVD with low carbon concentration and the results are shown in Fig. 1c-f. The surfaces are relatively flat, only seldom excrescences and defects exist, and the edges of the particles become neat. Compared to the initial particles, there is a great improvement in the appearance. Picture at high magnifications (Fig. 1d) shows that the particles treated by HFCVD have very smooth surfaces. The Raman spectra in Fig. 2 show that the diamond particles basically maintain the original single crystal structure with a small amount of graphite generated, which is reflected in that the intensity of the diamond characteristic peak (1333 cm^{-1}) does not move and

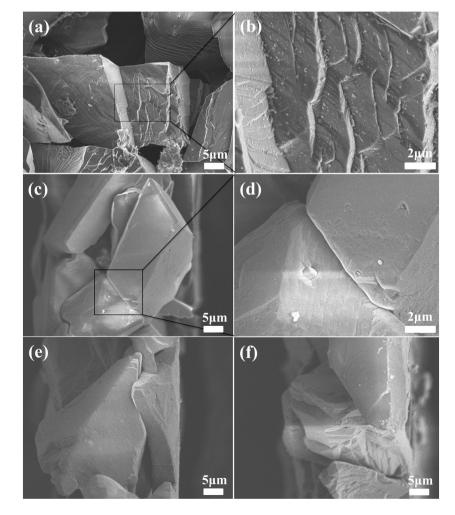


Fig. 1. SEM images of the diamond particles, (a) initial diamond particles, (b) high-resolution picture of the initial diamond particles, (c) the diamond particles treated by HFCVD, (d) high-resolution picture of the treated diamond particles, and (e)–(f) the diamond particles after treatment.

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