

# Characterization of the interior structure of synthetic diamond particles



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## ARTICLE INFO

### Article history:

Received 20 May 2016

Received in revised form

21 July 2016

Accepted 22 July 2016

Communicated by Peter Rudolph

Available online 25 July 2016

### Keywords:

Diamond

Carbonallotropes

Monocrystals

Nanocrystals

Amorphous carbon

## ABSTRACT

TEM observation was originally presented about the interior microstructure of a diamond particle, and three different kinds of carbon allotropes were found existing in diamond. The synthetic diamond particle was constituted of many tiny columnar monocrystals with the approximate diameter of 10 nm and length of more than several hundred nanometer. These nanocrystals were assembled into clusters along the  $\langle 111 \rangle$  lattice plane, while between these nanocrystals there were the amorphous carbons filled with, and this study originally revealed the microstructure of the synthetic diamond particle. Internal structure with crystal defects was also demonstrated clearly. These findings show the interior microstructure more explicitly, which may give useful inspiration to the technical progress of diamond synthesis.

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## 1. Introduction

Diamond has excellent properties such as the extremely great hardness, high wear resistance, good optical performance and excellent thermal conductivity, and it has been widely used as the raw materials in precision machining, drilling and jewelry industry [1–5]. Due to the depletion of natural diamond deposits and increasing demands of industry, synthetic diamond has caught extensive attention in public, especially when GE (General Electric) company produced the first man-made diamond in 1975 and put it into industrial production three years later. The physical properties of diamond absolutely depending on its microstructure should be deeply studied.

There are many researches about the internal microstructure of synthetic diamond particles, while the studies by TEM are scarce. Most of the results do not well clearly demonstrate its interior microstructure, thus limiting our understanding about the diamond particles. In this work, we applied in an appropriate method to prepare the thin film of diamond for TEM observation and originally presented the interior microstructure of diamond particles, and made certain of three different kinds of carbon allotropes existing in diamond. Further more, internal structure with crystal defects was also revealed clearly. These findings fully demonstrated the interior microstructure of diamond particle, which

would be useful to better understanding of diamond synthesis and various properties.

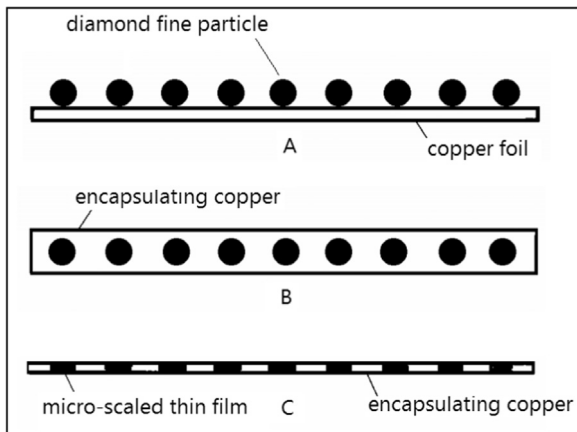
## 2. Experimental

Diamond particles (8–20  $\mu\text{m}$ ) purchased from domestic manufacture were observed through SEM (EVO18, Germany) instrument, and then Raman spectroscopy (LabRAMHR Evolution, France) characterizations were conducted with a 532 nm laser beam, a 200 mV power and a spot of 1  $\mu\text{m}$ . The Raman shift range was set from 1000 to 1800  $\text{cm}^{-1}$ . XRD studies were also conducted through the instrument (Ultima IV, Japan) with a Cu  $K\alpha$  radiation at 40 Kv and 40 mA. The measurement  $2\theta$  ranged from 10–100° with a step of 0.02° and a preset time of 0.3 s.

In a typical TEM film preparation procedure, the synthetic diamond particles were encapsulated in a copper foil by homogeneous electroplating deposition, as illustrated schematically in Fig. 1, and more details about homogeneous electroplating deposition were introduced in the literature [6], and then cut, polished to a suitable thickness for focused ion beam (FIB) milling. The thin film sample was milled by  $\text{Ar}^+$  from both sides with 15° until perforations generated, so that it was good for TEM observation. TEM (TecnaïG<sup>2</sup>F20, America), which was equipped with an X-ray energy-dispersive spectrometer (EDS) system and a high-angle angular-dark-field (HAADF) detector, was used for electron diffraction analysis, and high resolution transmission electron

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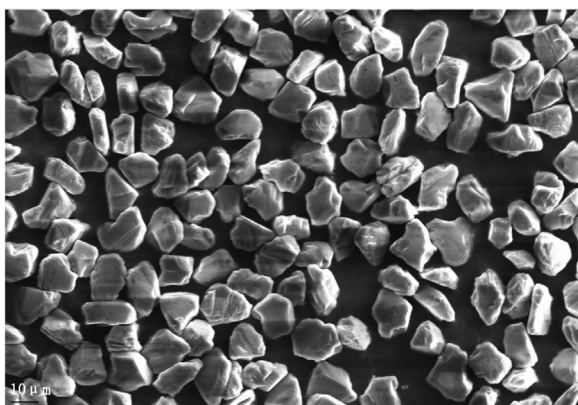
**Fig. 1.** Procedures that synthetic diamond particles were encapsulated into the copper foil (A, B) and prepared to a thin film by polishing and  $\text{Ar}^+$  milling for TEM observation.

microscopy (HRTEM) observations and chemical composition analysis were also conducted correspondingly. The EDS spectra were obtained by STEM mode. The step size for scan was 3 nm and the probe size from EDS was about 2 nm, and the results were analyzed through the affiliated TEM Imaging & Analysis software.

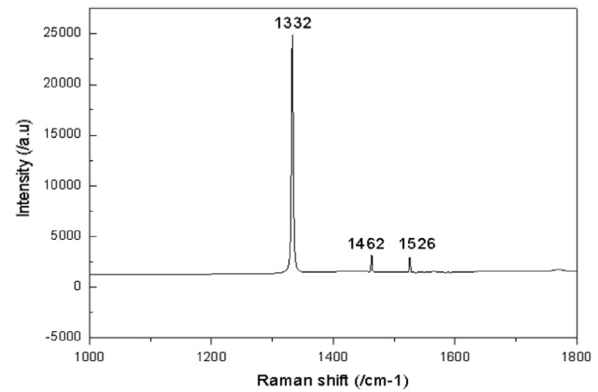
### 3. Results and discussion

**Fig. 2** revealed the microphotography of diamond particles with the average length of more than  $10\ \mu\text{m}$ . They had irregular configurations, and the boundaries of diamonds were rough, especially on the (100) faces, which indicated that the non-diamond carbon phase existed in diamond grains [7]. Moreover, these faces were partly out of flatness, and it could be seen that it was quite difficult to see the interior microstructure if without the TEM observation.

Raman spectroscopy is a most effective and powerful method to study diamond and other carbon allotropes, and the result was shown in **Fig. 3**. There was a sharp Raman peak at  $1332\ \text{cm}^{-1}$ , the characteristic of  $\text{sp}^3$  carbons corresponding to diamond [8–11], and the full width at half maximum (FWHM) of  $3\ \text{cm}^{-1}$  near the peak  $1332\ \text{cm}^{-1}$  showed the relative perfection of diamond crystal [12]. Generally, the Raman peak near  $1462\ \text{cm}^{-1}$  corresponding to the amorphous carbon with  $\text{sp}^2$  was regarded as the contamination for diamond. The peak appeared at  $1526\ \text{cm}^{-1}$  was attributed to nano-diamond carbon, and literature [8] even called the Raman peak near  $1560\ \text{cm}^{-1}$  onion-like carbon, while others [12,13] thought that it was related with amorphous carbons. The concern



**Fig. 2.** SEM photograph of synthetic fine diamond particles.



**Fig. 3.** Visible Raman spectrum of synthetic diamond particles.

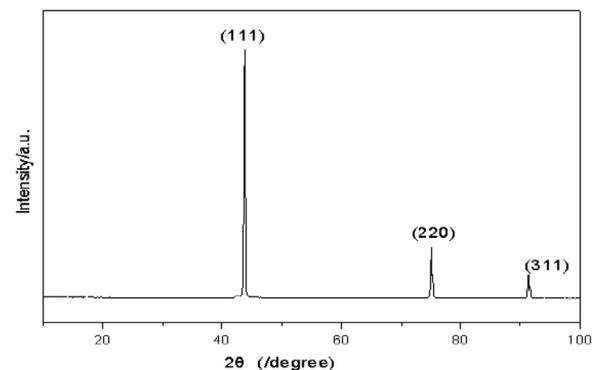
that Raman peak around  $1560\ \text{cm}^{-1}$  determines which kind of carbon allotrope is will be discussed in the following details. The visible Raman is very sensitive to the  $\text{sp}^2$  carbons, and it is available to detect the presence of the disordered carbons in diamond. Chuan [11] analyzed the effect of applying in different optical maser wavelength to detect the carbon allotropes, and it was concluded that the wavelength could not change the results but made it more obvious. In this work, we conducted the Raman experiments with a 532 nm laser beam and identified the diamond phase and other carbon allotropes.

**Fig. 4** showed the XRD characteristic (111), (220) and (311) diamond peaks, and these sharp peaks indicated a high degree of crystallization [14,15]. The appearance of strongest intensity at (111) diamond peak can be attributed to that more diamond nucleus [16] were inclined to gather and grew along (111) lattice plane during the synthetic process of transmitting carbon sources into diamond, while lattice planes such as (220) and (311) got less diamond nucleus.

**Fig. 5** indicated that a diamond field emission tip was extracted with the mentioned sample preparation technique. This TEM image demonstrated that diamond particles, as marked in the dotted rectangle, were successfully embedded into the copper foil substrate and it was good for the diamond perforations generation under the  $\text{Ar}^+$  ion milling.

**Fig. 6** represented the EDS face-scan results. The distributions of C, O and Cu by EDS face-scanning indicated that the compositions of the selected area were mainly composed of C, O and Cu, which agreed with the previous results. Element C came from diamond particles and Cu absolutely resulted from the copper substrate. The existence of O may be attributed to that Cu was easily oxidized by  $\text{O}_2$  in air [17], and cupric oxide thus came into the thin film during the sample preparation process.

**Fig. 7** showed the bright-field HRTEM images of diamond particle, and it was an astonishing finding that a diamond particle



**Fig. 4.** XRD pattern of diamond particles.

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