



Microstructural investigation of diamond-SiC composites produced by pressureless silicon infiltration



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ARTICLE INFO

Article history:

Received 24 June 2016

Received in revised form

17 November 2016

Accepted 5 December 2016

Available online 22 December 2016

Keywords:

Diamond

Silicon carbide

Composites

Microstructure

Crystallography

ABSTRACT

Superhard silicon carbide-bonded diamond materials were synthesized by liquid silicon infiltration of diamond-containing preforms. The properties of the materials were strongly influenced by the strength of the interfaces between the diamond and the silicon carbide. Interface formation was investigated through local analysis of the microstructure in the interface regions using field-emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM), and electron backscatter diffraction (EBSD) as well as X-ray diffraction (XRD). The results of these experiments revealed a pronounced orientation relationship between SiC and diamond at their interfaces and, as a result, strong bonding of the diamond particles to the ceramic matrix. There was also an orientation relationship between the nano-sized SiC grains, which were embedded in residual silicon near the diamond interfaces, and diamond. Additionally, the different morphologies and phenomena occurring in the microstructures of the diamond-SiC composites and their dependence on the infiltration temperature were studied.

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1. Introduction

Ceramics containing diamond or cubic boron nitride (c-BN) are of great interest for a variety of industrial applications. The very high hardness and wear resistance are the most important characteristics of this class of particle-reinforced ceramic materials [1]. However, classic sintering of such materials is impracticable because of the metastable character of diamond and c-BN. The requirement of mass transfer during sintering results in transformation of the metastable phases. Diamond transforms to graphite at temperatures exceeding 800 °C in oxygen-containing atmospheres and above 1600 °C in non-oxidizing atmospheres [2–5]. The transformation of c-BN is even observed at lower temperatures in the presence of a melt [6]. Thus, such materials with high diamond contents can only be sintered at high pressures that stabilize the respective metastable high-pressure phases [7,8].

The pressureless manufacturing of dense materials in which there is a high amount (up to 50 vol.%) of the metastable diamond phase in a ceramic matrix has been achieved through reaction sintering [9–11]. In this process, the diamond preform is infiltrated

with liquid silicon at 1450–1650 °C in a non-oxidizing atmosphere. The product of the infiltration is a composite material that contains SiC and diamond as well as residual elemental silicon, which deteriorates the corrosion stability of the composite in liquid media [12].

In addition to the reaction infiltration route, high pressure sintering [7,8] or silicon vapor vacuum reactive infiltration process [13,14] can be used to form these materials. For large diamond particles ($\geq 100 \mu\text{m}$), reactive hot pressing has also been used [15].

In the first results published on the microstructural investigations of diamond-SiC composites [16–20], the interface between diamond and cubic β -SiC was investigated using transmission electron microscopy. Various microstructural studies using TEM examined an orientation relationship between these two cubic phases in materials produced at high pressure and high temperature (HPHT) [18] and via silicon infiltration [16,17]. The main outcome of these studies was that an orientation relationship between diamond and SiC existed. Park et al. [17] observed the relationship $\{111\}_{\text{diamond}} \parallel \{111\}_{3\text{C-SiC}}$ and $\langle 011 \rangle_{\text{diamond}} \parallel \langle 112 \rangle_{3\text{C-SiC}}$. This corresponds to a rotation of 30° of the SiC lattice at a diamond-3C-SiC interface, resulting in minimization of the lattice mismatch (~5%).

By Yang et al. [14] the infiltration mechanisms of diamond graphite preforms by silicon vapor at 1600 °C for one hour are

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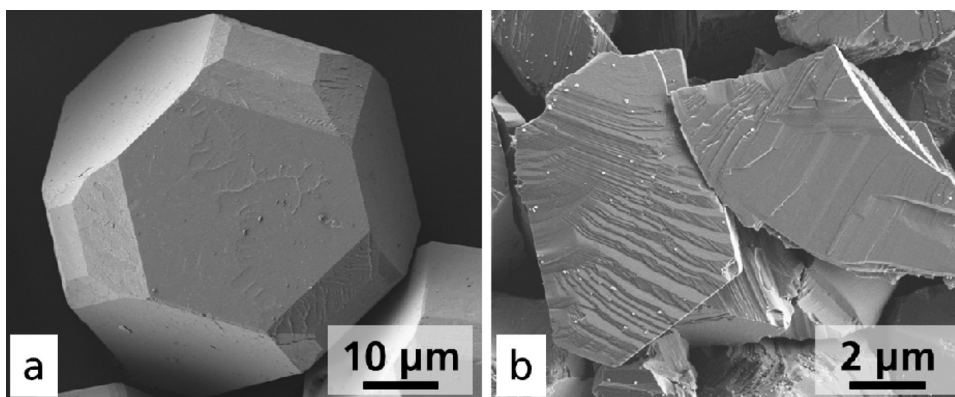


Fig. 1. Different morphologies of raw diamond powders: (a) faceted and well-grown diamond with a mean grain size of 50 μm and (b) crushed diamond powder with a mean grain size of 10 μm .

discussed. They do not find graphitization of diamond at these temperatures. Additionally, some epitaxial growth of silicon carbide on (111) surfaces of diamond was observed. However, they give no further information. The authors claimed the infiltration mechanism is a mixed mechanism of vapor and liquid infiltration. This and the fact that they have used a substantial amount of graphite and resin in the starting material results in large differences to our experiments. The kinetic data of the formation of SiC layer on diamond are measured and explained more realistically in [9,21].

In the present paper, the defect structures at the interfaces between diamond and silicon carbide, the stability of diamond in the composites, and the stability of raw diamond powder were analyzed in detail. For this purpose, the diamond preforms were infiltrated at different temperatures and analyzed using SEM, EBSD, and TEM. In addition, various diamond powders were analyzed using SEM before and after heat treatment near the stability limit. Finally, the long-term stability of heat-treated diamond-SiC composites was analyzed using various methods. The experimental results furthered the understanding of the different phenomena occurring in the formation of the microstructure in liquid silicon-infiltrated diamond-SiC composites and contributed to the refinement of a microstructural model.

2. Materials and methods

2.1. Preparation of the materials

The analyzed materials were prepared by infiltration of liquid silicon into the diamond-containing preforms. Such preforms can be produced by pressing of ceramic granules consisting of diamond and organic additives or by gel casting. The technology of pressureless preparation of graded and bulk diamond-SiC materials is reported in the literature [9,10]. For the preparation of the diamond preforms, two different synthetic diamond powders (VDiamant Ltd.) with respective average grain sizes of 50 μm and 10 μm (Fig. 1a and b) were used as starting materials. After the dia-

mond preforms were pressed and the binder was pyrolyzed at a temperature of 1000 $^{\circ}\text{C}$, most of the diamond surfaces are coated by glassy carbon. The green bodies formed in this way were infiltrated with liquid silicon at two different temperatures (1550 $^{\circ}\text{C}$ and 1600 $^{\circ}\text{C}$) in vacuum with a heating rate of 5 K/min and a holding time of 20 min at the peak temperature. The infiltration was carried out in a laboratory-scale furnace. The temperature was measured on the outsides of the crucibles; hence, the actual specimen temperature could differ slightly from the furnace temperature.

For determination of the temperature of onset of transformation of metastable cubic diamond to the thermodynamically stable phase of hexagonal graphite, a well-grown synthetic diamond powder (PDA 999 D46 element six Ltd.) with defined crystallographic facets (Fig. 1a) was heat-treated in small graphite crucibles for 20 min in a laboratory furnace at temperatures of 1550 $^{\circ}\text{C}$, 1600 $^{\circ}\text{C}$, and 1650 $^{\circ}\text{C}$ (heating rate: 10 K/min). The annealing temperature was determined with an accuracy of better than 5 K. The conditions of the heat treatment process for the diamond powder were chosen to resemble the infiltration process.

For demonstration of the long-term stability of the composites and the thermal stability of the interfaces in particular, the composite materials were additionally heat-treated at a temperature of 1525 $^{\circ}\text{C}$ for 35 h in a laboratory furnace with an argon atmosphere.

In addition to bulk materials, the starting diamond powder with a grain size of 100 μm (VDiamant Ltd.) was coated by a CVI process using SiO as a vapor phase produced in-situ by mixing of silicon and SiO₂ at a temperature of 1450 $^{\circ}\text{C}$ in an argon atmosphere. This experiment was designed to have the possibility to compare the nucleation of silicon carbide on pure diamond surfaces and in the bulk material. The crystals that formed on the surface showed a defined crystal habit, which is analyzed in detail in this study.

An overview of the infiltrated and heat-treated materials as well as the CVI-treated diamond powder sample with the diamond grain size, heat-treatment temperatures, density, and selected mechanical properties are given in Table 1. The sample nomenclature of the diamond-SiC Composites (DSc.) as well as the coated dia-

Table 1
Selected properties of prepared samples.

Sample	Diamond grain size (μm)	Residual silicon (wt.%)	Infiltration temperature ($^{\circ}\text{C}$)	Density (g/cm^3)	Hardness HK-2 (GPa)	Young's modulus (GPa)
DSc.A10	10	3.2 \pm 0.2	1550	3.25	49.6 \pm 1.6	525 \pm 5
DSc.A50	50	11.3 \pm 0.5	1550	3.15	>35	518 \pm 5
DSc.B10	10	–	1600	3.21	30.3 \pm 2.3	–
DP.C100	100	–	^a 1450	–	–	–
DSc.A50.t	50	<0.1	^b 1525	–	–	–

^a CVI gas phase reaction with SiO (g).

^b Further heat treatment of sample DSc.A50.

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