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EBSD characterization of the growth mechanism of SiC synthesized via direct microwave heating

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ABSTRACT

Well-crystallized 3C-silicon carbide (SiC) grains/nanowires have been synthesized rapidly and conveniently via direct microwave heating, simply using silicon dioxide powders and artificial graphite as raw materials. The comprehensive characterizations have been employed to investigate the micro-structure of the obtained 3C-SiC products. Results indicated that, different from the classic screw dislocation growth mechanism, the 3C-SiC grains/nanowires synthesized via high-energy vacuum microwave irradiation were achieved through the two-dimension nucleation and laminar growth mechanism. Especially, the electron backscattered diffraction (EBSD) was employed to characterize the crystal planes of the as-grown SiC products. The calculated Euler angles suggested that the fastest-growing crystal planes {211} were overlapped gradually. Through the formation of the {421} transformation plane, {211} finally evolved to {220} which existed as the side face of SiC grains. The most stable crystal planes {111} became the regular hexagonal planes in the end, which could be explained by the Bravais rule. The characterization results of EBSD provided important experimental information for the evolution of crystal planes.

1. Introduction

Silicon carbide (SiC) has extracted extensive interest over the recent years due to its numerous outstanding properties, such as excellent mechanical strength, high thermal conductivity and stability. Nowadays, owing to its extraordinary properties exhibited in extreme conditions, SiC also plays a key role in the field of thermal protection [1,2]. Besides, with its wide band gap, high critical breakdown electric field, low dielectric constant and high saturated electron mobility, as well as strong anti-radiation, good mechanical property and other characteristics [3–6], SiC is also an essential semiconductor material for making transistors and sensors.

It is well known that material properties depend not only on chemical compositions, but also on microstructure. Meanwhile, during the formation process of the materials, especially the crystal nucleation and the subsequent growth, many factors can seriously affect the microstructure. Therefore, the studies on synthesis and properties of the SiC with different morphologies and crystal forms have drawn significant focus. Thus far, many ways to synthesize SiC have been developed, such as carbon–thermal reduction [7,8], physical vapor transport [9], template synthesis [10], shape memory synthesis [11], sol–gel method [12,13], arc-discharge method [14], chemical vapor deposition [15,16], polymer pyrolysis route [16], microwave irradiation [17,18] and so on. At the same time, some growth mechanisms have been deduced. The most common modes are solid–liquid–solid (SLS) growth [6], vapor–solid (VS) growth [7,10,14], vapor–liquid–solid (VLS) growth [19], composite mechanism of VLS and VS [8,16], or oxide assisted growth mechanism [20]. The classic screw dislocation mechanism [21,22] is usually used to explain the primary nucleation and subsequent growth of SiC. But with different synthesis approaches and conditions, the formation mechanism of SiC may change as well. For example, the formation of SiC platelets prepared via microwave irradiation heating or double-heating technique can be properly explained by the two-dimensional nucleation and growth mechanism [17,23].

In recent years, microwave has been widely used for chemical synthesis, especially for the preparation of novel functional materials [17,18,24]. By high-energy microwave irradiation, products with high purity can be synthesized directly without catalyst, template or substrate. The SiC nanowires obtained via direct microwave irradiation exhibited excellent photoluminescence property [18]. Different from the traditional heating approaches, the high-energy microwave irradiation heating is a dielectric polarization/loss process occurring in the interior of the substance which can absorb microwave. During the irradiation process, the microwave energy can be rapidly transformed to heat. So, microwave thermal treatment has the advantages of high speed, selectivity, homogeneous heating, high purity of products, easy to be scaled up and industrialized, etc. [17]. For example, Carassiti et al. obtained

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3C-SiC nano-fibers as small as 5 nm in diameter in a multimode cavity, and large crystallites of α- and 3C-SiC in 1 min in a single mode cavity [17]. Moreover, because of the complex reaction systems and the multiple growth mechanisms under high-energy microwave irradiation, the obtained specimens would exhibit various morphologies and sizes. It is quite easy to obtain SiC grains and nanowires simultaneously via high-energy microwave irradiation [18,25].

For the special synthesis occurring in the strong electromagnetic field which derived from high-energy microwave irradiation, the growth mechanism hasn't been investigated in detail. In the present work, high-energy vacuum microwave irradiation was directly applied to prepare SiC, and the growth mechanism was discussed. In comparison with our previous studies [18,25], the components of the raw materials were simplified, just covering SiO_2 powders and artificial graphite. Results indicated that the pure and well-crystallized 3C-SiC nanowires and grains were achieved successfully. After the comprehensive micro-structural characterizations, the formation mechanism of the SiC nanowires and grains was explored. It is worthy to note that, under high-energy vacuum microwave irradiation, the formation of 3C-SiC can be properly explained by the mechanism of two-dimension nucleation and laminar growth. Among the comprehensive characterizations, the in-situ technique of electron backscattered diffraction (EBSD) was especially used to characterize the crystal plane information of the observed SiC grains. The detailed EBSD investigation provided the experimental evidences for the evolution of crystal planes during the growth process.

2. Experimental

2.1. Synthesis of 3C-SiC

The synthesis of 3C-SiC products was performed in a multimode microwave vacuum sintering furnace (Jiequan Microwave Co., China), with the adjustable powers ranging between 0 and 10 kW. The temperature of raw materials was measured by an optical pyrometer (Reytek) on the top of the insulation glass.

The raw materials containing SiO_2 and artificial graphite powders were mixed at a molar ratio of 1:1 and they were transferred into a ceramic crucible. The exact masses of SiO_2 and graphite powders were 12.0 and 2.4 g, respectively. Then the crucible filled with specimens was placed in the center of the multimode resonant cavity of the microwave furnace. After the resonant cavity was evacuated to approximately 2×10^4 Pa by a water circulating pump, the raw materials were heated with microwave power of about 3.0 kW. Although SiO_2 cannot interact with microwave, artificial graphite can be applied as an outstanding microwave absorbing agent, namely it absorbs the microwave energy and converts it into heat. By adjusting the microwave power, the raw materials can be heated to over 1000 °C in several minutes. After being heat-treated at 1450 °C for 80 min, the power was turned off to stop the microwave irradiation, and the specimens with the furnace were naturally cooled to room temperature. Although we can obtain SiC with a good crystalline nature in a short time, the subsequent EBSD characterization cannot be carried out if the sizes of specimens are very small. In order to ensure that the as-grown SiC products are big enough for the EBSD characterization, the time of microwave heating was 80 min.

2.2. Characterization

A Sirion field emission scanning electron microscope (SEM) (FEI Ltd., Netherlands) with the attached GENESIS 60S energy dispersive spectrometer (EDS) (EDAX. Co, America) was employed to investigate the morphology and the composition of the products. Before investigation, the samples were coated with a thin layer of platinum. A FEI Tecnai T20 transmission electron microscope (TEM) (FEI, Eindhoven, Netherlands) was employed to observe further the amplified morphology and characterize the crystal structure of the specimens. X-ray diffraction (XRD) patterns were achieved by a X'TRA diffractometer (ARL Co., Switzerland) with Cu Kα radiation (λ = 1.5418 Å). Data were collected over the 2θ range of 10–80°, with the scanning speed of 10°/min and output power of 2.2 kW.

An EBSD system (TSL Co., America), also attached with a SEM apparatus, was applied to characterize the crystal structure. The test was performed when the tilt angle of the specimen was up to 70°, with a work distance of 8 mm. Supported by the software of OIM Data Collection 5.3 (TSL Co., America), the crystal structure information could be obtained. The confirmation process was composed of two steps: (i) the Kikuchi bands were calibrated to gain the crystal forms of the selected specimen, according to the matching degree counted automatically by the software; and (ii) the Euler angles ($\varphi_1, \varphi_2, \phi$) were calculated with formulas (1), (2) and (3) to obtain the plane index of the crystal.

$$h = n \sin \varphi_2 \sin \phi \tag{1}$$

$$k = n \cos \varphi_2 \sin \phi \tag{2}$$

$$l = n \cos \phi \tag{3}$$

where, n is an integer. With the two steps and further analysis, the in-situ characterizations of morphology and the corresponding growth information could be realized [26].

3. Results and discussion

3.1. Phase composition and morphologies of products

3.1.1. Morphologies of products prepared by high-energy microwave irradiation

The XRD pattern of the product is shown in Fig. 1. Compared with the standard diffraction card (JCPDF No. 29-1129), the strongest peak centered at about 35.6° can be attributed to the close-packed plane (111) of 3C-SiC. The peaks at 41.8, 60.4, 72.1 and 75.9° are also the characteristic peaks of 3C-SiC, which can be identified to the (200), (220), (311) and (222) crystal planes, respectively. Besides, the low-intensity peak centered at about 34.1°, common in the studies of SiC, may be attributed to the stacking faults [6,18,22]. Corresponding to the standard values (JCPDF No. 74-1302), such a low-intensity peak is the (101) lattice plane of 6H-SiC. Besides, 6H-SiC shares the same peaks centered at 35.6, 60.4 and 72.1° which can be assigned to the (102), (110) and (202) crystal planes. Therefore, the existence of 6H-SiC might also contribute to the common peaks. In addition, no characteristic diffraction peaks of raw materials could be found in the XRD pattern, indicating that the reaction was complete and the obtained SiC was pure. That is, by directly heating the raw materials with high-energy microwave, the products consisting mainly of 3C-SiC can be achieved. Although some other crystal forms for SiC might coexist in the specimens, the synthesis process still exhibited the superiority of being fast and effective.

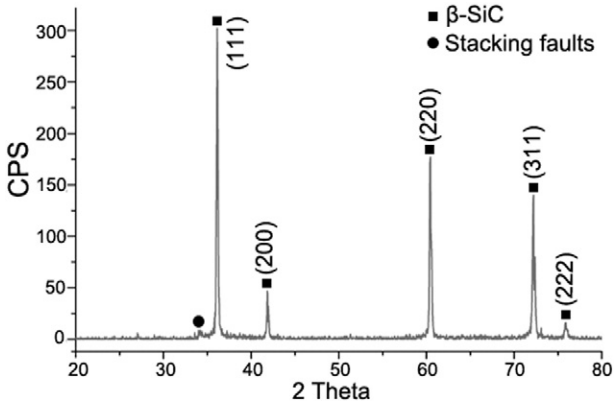

Fig. 1. XRD pattern of the sample.