



Short Communication

Screw dislocation assisted spontaneous growth of single-crystalline α -Al₂O₃ microrods

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ABSTRACT

Herein we reported a simple chemical vapor deposition approach to synthesize single crystal α -Al₂O₃ microrods with diameters of 2–4 μ m and lengths of 10–30 μ m on the as-prepared SiC-Si-Al₂O₃ ceramics based on the screw dislocation growth. Two types of evidence for screw dislocation growth have been conclusively observed in the as-synthesized microrods: scanning electron microscopy analysis reveals that the representative morphology feature of the screw dislocation growth is the stepwise spiral terrace configuration and the growth kinetics of the as-synthesized microrods follows the prediction of the dislocation growth mechanism as described by Burton-Cabrera-Frank theory. This work provides not only a new method for synthesizing Al₂O₃ microrods but also a new insight into the growth mode for Al₂O₃ microrods.

1. Introduction

The formation of any micro/nano-structures relies on crystal growth, which includes three basic growth mechanisms: screw dislocation growth, layer-by-layer (LBL) growth, and dendritic growth [1]. The proceeding of a specific growth mechanism is dictated by the supersaturation of the system. The classical crystal-growth theory predicts that the screw dislocation growth only plays a vital role in the formation of all micro/nano-structures under low supersaturation conditions [2]. The reason is that the self-perpetuating step edge provided by screw dislocation growth spirals permits crystal growth to occur at supersaturations below that necessary to create two-dimensional (2D) nuclei for LBL growth [2]. Recently, the screw dislocation growth has attracted tremendous attention in the interpretation of the formation of various micro/nano-structures, including one-dimensional (1D) nanowires [3] and nanotubes [4], 2D micro/nano-plates [5,6], and three-dimensional hierarchical tree-like nanostructures [7]. In addition, the screw dislocation growth (for example, 1D micro/nano-rods) is expected to be also related to the formation of much more micro/nano-structures.

Alumina (Al₂O₃), a conventional ceramic material, has aroused considerable attentions in recent years owing to its potential applications in absorbent, catalyst carrier, and reinforcement of ceramic composites for its high strength, corrosion resistance, and chemical stability [8]. Compared with their bulk counterparts, 1D Al₂O₃ micro/nano-rods have more superior performances owing to their small

size and few defects [9], which make them exhibits more potential applications in absorbent, catalyst carrier, and reinforcement of ceramic composites. To date, much effort has been devoted to synthesizing Al₂O₃ nanorods, but the formation of these Al₂O₃ nanorods is only governed by LBL growth [10,11]. To our knowledge, the screw dislocation growth in the formation of Al₂O₃ micro/nano-rods has been rarely reported so far.

In this work, we report a facile vapor deposition approach to *in situ* synthesize Al₂O₃ microrods on the as-prepared SiC-Si-Al₂O₃ ceramics via the screw dislocation growth. The features from the terraces confirmed the presence of screw dislocations in the as-synthesized α -Al₂O₃ microrods. This was further validated by analyzing the growth kinetics of the as-synthesized α -Al₂O₃ microrods. This work provides a new strategy for synthesizing Al₂O₃ microrods and also expands the vision to understand the screw dislocation growth mode in the Al₂O₃ microrods.

2. Experimental procedure

Al₂O₃ microrods were synthesized on the as-prepared SiC-Si-Al₂O₃ ceramics by CVD method. First, SiC-Si-Al₂O₃ ceramics were prepared by hot-pressing reactive sintering in the furnace. The powders were mixed as follows: 65 wt.% Si (purity: 99.5%, size: 300 mesh), 10 wt.% Al₂O₃ (purity: analytical reagent, size: 300 mesh), and 25 wt.% graphite (purity: 99.0%, size: 320 mesh), and ball milled for 24 h in ethanol using high-purity agate spherical media. The powders were dried, screened, and pressed into pellets of 50 mm (diameter) \times 10 mm

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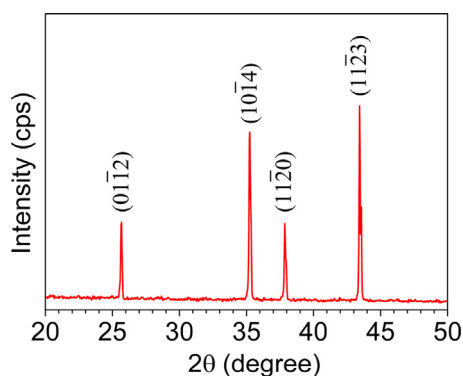


Fig. 1. XRD pattern recorded from the as-synthesized products.

(thickness) under a uniaxial pressure of 10 MPa. Then, these pellets were hot-pressed at a mechanical pressure of 30 MPa inside graphite dies in argon atmosphere at a temperature of 1700 °C for 2 h. Second, the as-prepared SiC-Si-Al₂O₃ ceramics as the precursor and substrates were encapsulated in graphite papers and placed at the center of an alumina tube in the furnace. After evacuating the furnace for 3 times, argon carrier gas (purity: 99.95%) was introduced into the system with the flow rate of 50 sccm, while oxygen (purity: 99.95%) with the flow rate of 4 sccm were used as the precursor. The samples were heated from room temperature to 1600 °C at a rate of 10 °C/min and held for 2 h, followed by furnace cooling to room temperature. To adjust the kinetic for the growth of Al₂O₃ microrods, more flow rates of the oxygen precursor (1 sccm, 2 sccm, and 3 sccm) were used. The super-saturation of the system was defined as following [12]:

$$\sigma = \frac{P}{P_e} - 1 \quad (1)$$

where P is the partial pressure of oxygen, P_e is the equilibrium partial pressure of oxygen at the deposition location. Details of the calculation process of P and P_e were reported elsewhere (Ref. 13).

The phase composition, morphology and microstructure of the as-obtained products were analyzed using X-ray diffraction (XRD; X'Pert PRO; PANalytical, Almelo, Netherlands), scanning electron microscopy (SEM, supra-55; Zeiss, Oberkochen, Germany) and transmission electron microscopy (TEM, Tecnai F30G2; FEI, Eindhoven, Netherlands) with energy dispersive X-ray spectroscopy (EDS).

3. Results and discussion

To investigate the phase composition of the as-obtained products, the as-obtained products peeled from substrate were tested by XRD. Fig. 1 depicts XRD pattern of the as-obtained products. It can be seen that all diffraction peaks can be indexed to the hexagonal α -Al₂O₃ phase with lattice parameters, $a = 0.476$ nm and $c = 1.299$ nm (JCPDS Card no. 46-1212), which implies the as-obtained products are composed of α -Al₂O₃ phase.

To view the morphology of the products on the as-prepared SiC-Si-Al₂O₃ ceramics, the samples were studied by SEM. The low-magnification SEM image (Fig. 2(a)) shows that the substrates are densely coated with many microrods. The majority of microrods have a low small aspect ratio with a typical diameter of 2–4 μ m and length of 5–20 μ m. The high-magnification SEM images (Fig. 2(b) and (c)) show that these microrods all present a hillock structure on their tops. It is worth noticing these hillocks present a stepwise spiral terrace configuration on their top surface with their diameters decreasing continuously, as shown in Fig. 2(d). This surface feature fits well to a screw

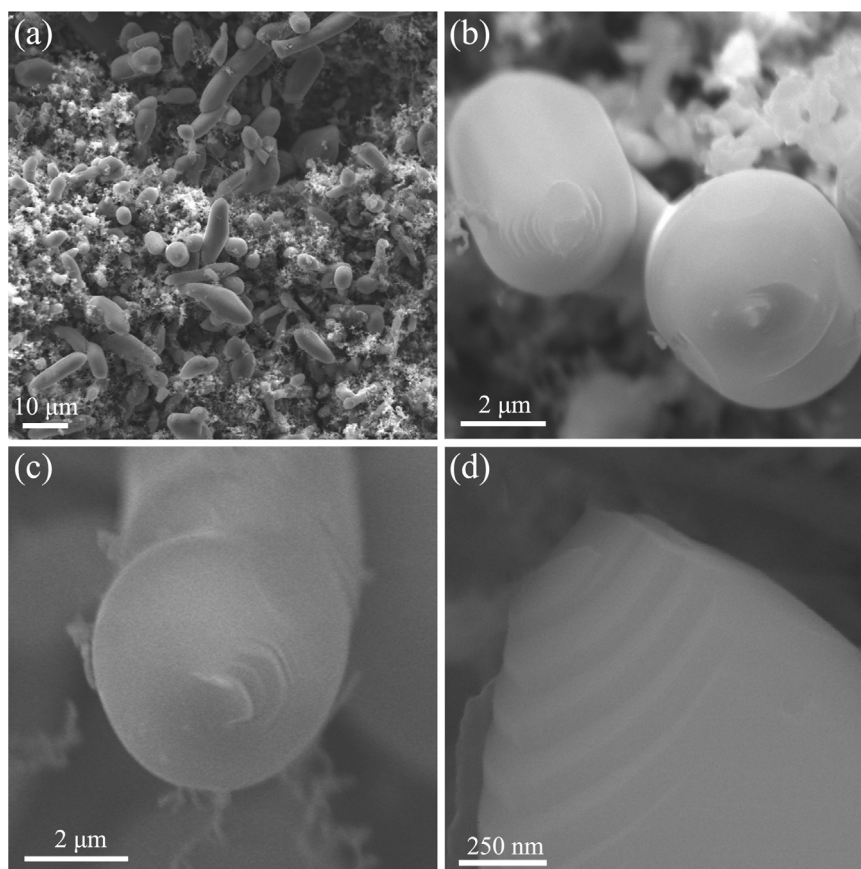


Fig. 2. SEM images of the as-synthesized α -Al₂O₃ microrods on the ceramic substrates. (a) Low-magnification; (b), (c) and (d) high-magnification.

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