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Short communication

Large-scale synthesis of aluminium borate nanowires by a simple molten salt method

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Abstract

Aluminum borate nanowires were synthesized in a molten salt method at 1100 °C in the air by using ammonium aluminum carbonate hydroxide (AACH) and H_3BO_3 as starting materials. The as-synthesized samples were examined by XRD and SEM. The results showed that $Al_{18}B_4O_{33}$ nanowires with an orthorhombic structure were formed at 1100 °C. A self-catalytic mechanism was proposed for the growth mechanism of the nanowires on the basis of the experimental phenomena.

Keywords: A. Sintering; Nanocrystalline materials; Ceramics; Aluminum borate nanowire; Molten salt

1. Introduction

One-dimensional nanostructure materials, such as nanowires, nanofibers, nanotubes, nanobelts and whiskers, have received extensive attention due to their interesting mechanical, electrical and optical properties [1–5]. Among these materials, aluminum borate ($Al_{18}B_4O_{33}$) nanowires are excellent and cheap one-dimensional ceramic materials with high tensile strength, elastic modulus, remarkable resistance to corrosion and chemical stability [6,7]. Recently, various useful methods have been developed to synthesize aluminum borate nanowires and whiskers by using different starting materials [8–15]. The molten salt method is a simple way to prepare one-dimensional ceramic materials, such as $Al_{18}B_4O_{33}$ nanowires [8,9] formed by active Al_2O_3.

It is interesting to find that active Al_2O_3 can be obtained by the thermal decomposition of ammonium aluminum carbonate hydroxide (AACH) [16]. It implies that $Al_{18}B_4O_{33}$ nanowires could be prepared from AACH. Here, we reported a novel way to synthesize $Al_{18}B_4O_{33}$ nanowires in a large scale through a simple molten salt method by using AACH and H_3BO_3 as starting materials. Compared to many other methods [4,5], no any catalysts are used in this method.

2. Experimental

2.1. Reagent and apparatus

All chemicals were reagent-grade pure and purchased from the Sinopharm Chemical Reagent Co. Ltd., China. X-ray powder diffraction (XRD) was performed at a scanning rate of 5°/min from 5° to 70° for 2θ at room temperature. A Rigaku D/max 2500 V diffractometer was used for the study and equipped with a graphite monochromator by utilizing monochromatic Cu$K\alpha$ radiation (λ=0.154178 nm). The morphologies of the samples were examined by S-3400 scanning electron microscopy (SEM). Samples were mounted on an alumina slice coated with Au.

2.2. Preparation of $Al_{18}B_4O_{33}$ nanowires

AACH was synthesized by solid-state reaction at low-heating temperature [16]. Ground AACH (20 mmol, 2.780 g) and H_3BO_3

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(8 mmol, 0.495 g) (molar ratio of H_3BO_3:AACH=1:2.5) were mixed and carefully ground together for 10 min. The reaction mixture gradually became damp and darker. Then the mixture was heated at 100 °C for 5 h to generate the precursor powder. Following a typical method for synthesizing $Al_{18}B_4O_{33}$ nanowires, the precursor powder (2.0 g) and K_2SO_4 (4.0 g) were put in a mortar and carefully ground for 30 min. Then the reaction mixture was calcined at 1100 °C for 2.5 h to generate the $Al_{18}B_4O_{33}$ nanowires.

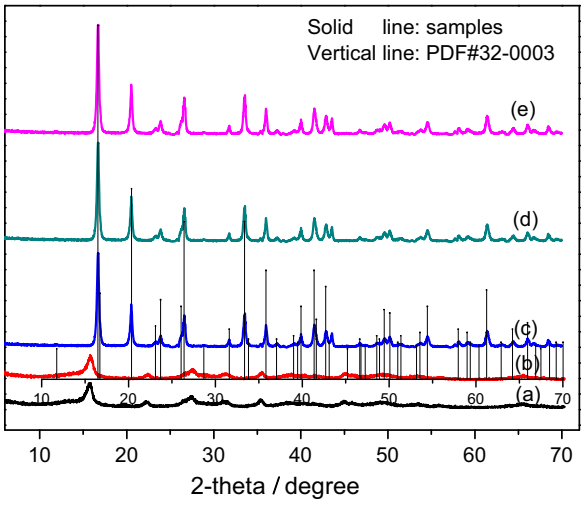

Fig. 1. XRD patterns of PDF#32-0003 and samples: (a) AACH, (b) precursor (molar ratio of AACH:H_3BO_3=2.5:1) and samples calcined at 1100 °C for different time (x): (c) x=2.5 h; (d) x=3.0 h; (e) x=4.0 h.

3. Results and discussion

Fig. 1 shows the XRD patterns of AACH dried at 80 °C for 5 h, the precursor, and the samples resulted from calcining the precursor at 1100 °C in a molten salt (K_2SO_4) by different time. In pattern (c), only several weak diffraction peaks of Al_2O_3 could be observed, which there were two peaks around 43.6° and 57.7° of 2-theta. The first peak may be a combined peak of aluminum borate and alumina, and second peak is a peak of aluminum. Two lattice parameters could be obtained from patterns (a) (AACH) and (c) (product calcined at 1100 °C) in Fig. 1; (i) for pattern (a): a=0.665078 (6), b=1. 2.02531 (2), c=0. 575945 (3) nm, and $\alpha=\beta=\gamma=90°$, which are in agreement with those of orthorhombic $NH_4Al(OH)_2CO_3$ (space group *Cmcm* (63), cell parameters: a=0.6618, b=1.1944, c=0.5724 nm, $\alpha=\beta=\gamma=90°$ (PDF card 76-1923)); (ii) for pattern (c): a=0. 768761(1), b=1.50224 (3), c=0.567163 (4) nm, $\alpha=\beta=\gamma=90°$, which are in agreement with those of orthorhombic $Al_{18}B_4O_{33}$ (space group *Amam* (63) and cell parameters: a=7.6874, b=15.0127 and c=5.6643 nm, $\alpha=\beta=\gamma=90°$ (PDF card 32-0003)). These results illustrated that the sample was a phase of $Al_{18}B_4O_{33}$. As shown in Fig. 1, all calcined samples have similar XRD patterns except the diffraction peak intensities, indicating that 2.5 h's calcining is enough for forming $Al_{18}B_4O_{33}$. Pattern (b) is the XRD pattern of the precursor and is the same as the pattern (a), indicating that besides AACH, no other crystals exist in the precursor. Fig. 1S shows FT-IR spectra of the sample calcined at 1100 °C for 4.0 h. Between 1500 and 1200 cm^{-1}, three intense bands appear at 1427, 1332 and 1263 cm^{-1}. The presence of these bands may be caused by crystalline aluminum borate shown in X-ray [17]. In

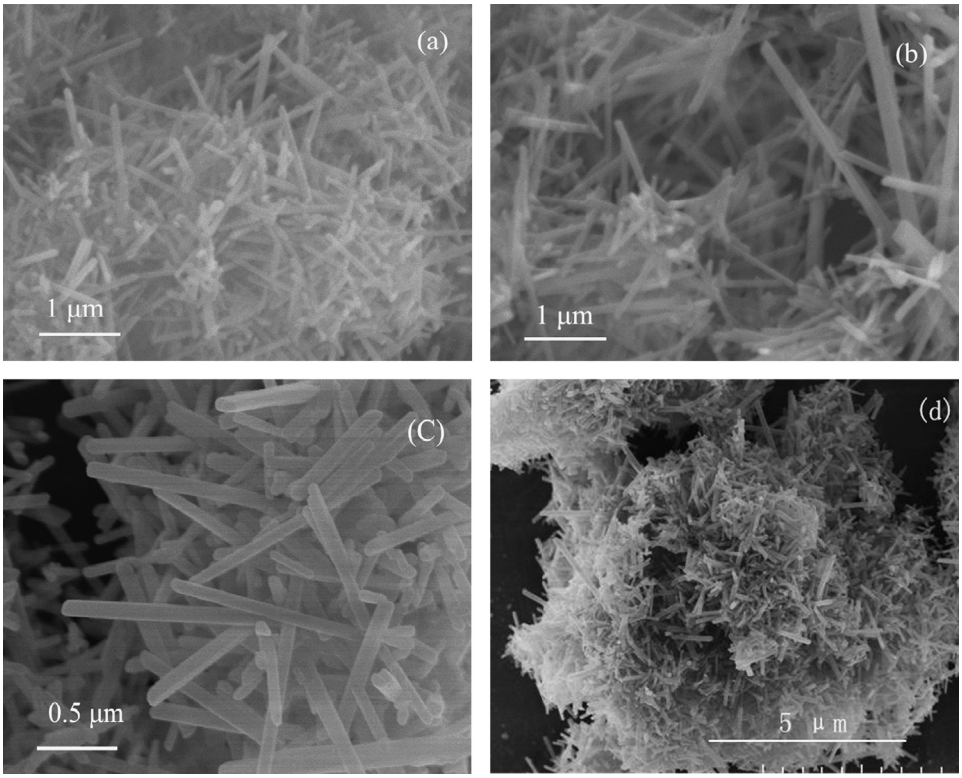

Fig. 2. SEM images of samples (molar ratio of H_3BO_3:AACH=1:2.5) calcined at 1100 °C for different time (x): (a) x=2.5 h, (b) x=3.0 h, (c) x=4.0 h.