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ARTICLE

ZrB_2-TiB_2 Nanocomposite Powder Prepared by Mechanical Alloying

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Abstract: Diborides, including zirconium diboride (ZrB_2) and titanium diboride (TiB_2), have a number of desirable ceramic qualities that make suitable for preparing ceramic-matrix composites. However, synthesizing a composite based on these materials usually requires high temperatures and complex synthetic methods. In the present study, a nanocrystalline ZrB_2-TiB_2 powder was synthesized via mechanical alloying (MA) of the mixture of elemental Zr, Ti, and B powders mixed at a Zr/Ti/B mole ratio of 1:1:4, a 10:1 ball-to-powder weight ratio and 500 r/min rotational speed in a planetary ball-mill under argon atmosphere using a ZrO_2 vial and balls. The effect of milling time on the phase change was investigated by X-ray diffraction (XRD), and the microstructure evolution of the powder mixture was monitored by field emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM). It is found that after 120 h of milling, a nanoscale composite powder with ~20 nm mean particle size can be obtained. Moreover, TEM examination clearly shows the composite powder is composed of the nanoscale TiB_2 and ZrB_2 particles. Finally, the milling mechanism was discussed.

Key words: ZrB_2-TiB_2; nanocomposite; mechanical alloying

Diborides from the transition metals IVB group, such as zirconium diboride (ZrB_2) and titanium diboride (TiB_2), exhibit high strength, excellent hardness, high melting point, high thermal and electrical conductivity, good thermal shock resistance and good chemically stability [1-14]. Such a series of interesting properties make TiB_2 and ZrB_2 useful materials for commercial applications. Additionally, ceramic-matrix composites (CMCs) have gained increasing interest due to their enhancing intrinsically low fracture resistance of monolithic ceramics [11-17]. The use of ZrB_2 and TiB_2 ceramics in composites is expected to offer a variety of high-temperature thermal and structural applications compared to individual ceramics. In order to improve mechanical properties, it is vital to prepare ZrB_2-TiB_2 powder with fine and homogeneous particles used as composite precursors.

Since ZrB_2 and TiB_2 are covalent materials with low self-diffusion coefficients and high melting points, it is difficult to fabricate ZrB_2-TiB_2 composites using conventional sintering methods [11-15, 17-19]. Currently, ZrB_2-TiB_2 composites are usually fabricated using a mechanical activation assisted self-propagating high-temperature synthesis of Zr/Ti/B powder blends [11, 13, 15, 19], pressureless sintering [18] or spark plasma sintering [12, 15]. However, the methodology for producing fine structure ZrB_2-TiB_2 ceramic powders via mechanical alloying (MA) has not been covered in depth. MA is a solid-state powder processing technique involving repeated welding, fracturing, and re-welding of powder particles in a high-energy ball mill [20, 21]. MA is superior to other methods due to its low

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cost, simplicity and high output. Several ceramic nano-composites, including TiB_2-TiC nanocomposite powders[22], nanocrystalline ZrB_2 powder[8], and nanocrystalline Ti(C,N) powder[23] others[24] have been successfully fabricated using MA.

The present work described the preparation of ZrB_2-TiB_2 nanocomposites powders at ambient temperature using MA. The mole ratio of Zr:Ti:B used were 1/1/4. Effects of MA duration on the composition and the microstructure features of the composite powders was evaluated.

1 Experiment

Metallic zirconium powder (Zr, 99.5% purity, Sinopharm Chemical Reagent Co., Ltd, China), titanium powder (Ti, 99.99% purity, Sinopharm Chemical Reagent Co., Ltd, China), and amorphous boron powder (B, 99% purity, Sinopharm Chemical Reagent Co., Ltd, China) were used as the raw materials. They had irregular shapes and particle sizes between 1~50 μm (Fig.1a~1c).

The powders were mixed to obtain the mixture with a Zr/Ti/B mole ratio of 1:1:4. The MA experiments were carried out in a planetary ball mill (QM-3PS2, Nanjing NanDa, China) using 8 mm diameter zirconia balls in a 250 mL zirconia vial with a ball-to-powder weight ratio (BPR) of 10:1. The powder mixture and the balls were placed inside the vials and sealed in a glove box (ZKX1, Nanjing NanDa, China) under Ar gas to prevent surface oxidation and contamination of the powder blends from the surrounding atmosphere. The formation of amorphous and nanocrystalline structures by MA is strongly dependent on the processing parameters. The most important processing parameters are the milling time, milling atmosphere, BPR and rotation speed. In the present work, we only changed the milling time while keeping the other parameters nominally constant: BPR at 10:1, rotation speed at 500 min^{-1}, and an argon shield. The MA process was interrupted at regular time intervals of 0, 10, 20, 40, 60, 80, 100, and 120 h, and a small portion of the powder was removed from the vial in the argon-filled glove box for characterization. Herein we defined the reaction initiation time when a sharp increase in the vial temperature was identified.

The phase evolution with milling time was investigated using an X-ray diffraction (XRD) analyzer (D8 Advance, Bruker, Germany) with Cu Kα radiation. The scanning speed was 2°/min and two-theta was recorded across the range of 20° to 90° with a 0.02 step size. The morphologies of the powder mixtures after milling for different time intervals were observed via field-emission scanning electron microscopy (FESEM, NoVa Nano SEM 430, FEI Company, USA) and transmission electron microscopy (TEM, Tecnai G2 F20, FEI Company, USA).

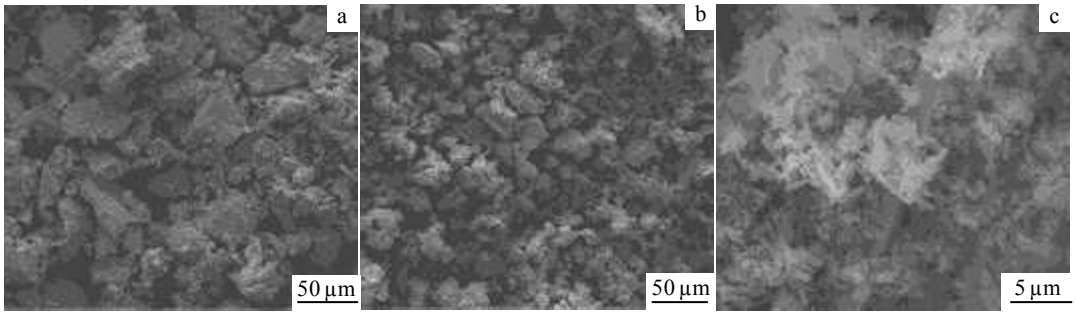

Fig.1 SEM morphologies of the starting powders: (a) Zr , (b) Ti, and (c) B

2 Results and Discussion

2.1 XRD analysis of the powder mixtures

Fig.2 shows the XRD patterns for the mixtures of Zr, Ti, and B at a 1:1:4 mole ratio after MA for 0, 10, and 20 h. There is no boron peak due to its amorphous nature. It is evident in Fig.2 that no new phases are formed, the peak intensities decrease sharply after only short milling duration. The peak broadening in the XRD spectra results from increased internal strain and reduced grain size due to the

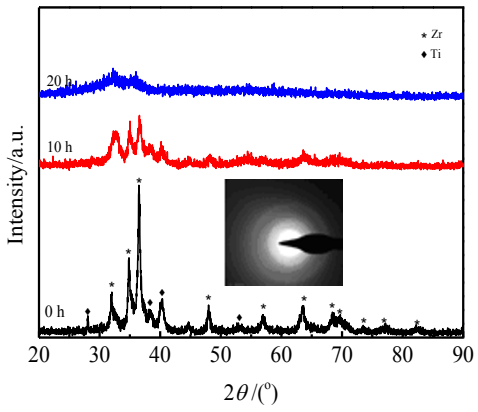

Fig.2 XRD patterns of the Zr/Ti/B powder blend at a mole ratio of 1:1:4, milled for different time (the inset shows the SAED pattern of the powder mixture milled for 20 h)