



Mechanochemical synthesis of NbC–NbB₂ nanocomposite from the Mg/B₂O₃/Nb/C powder mixtures

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Abstract

An experimental study was conducted on the preparation and characterization of nanocrystalline niobium boride and carbide composite (NbB₂–NbC) in the Mg–B₂O₃–Nb–C system via mechanochemical method. The thermodynamic appraisal indicated that the reduction and synthesis reactions were highly exothermic and should be self-sustaining. According to the differential thermal analysis (DTA) results, a pre-milling treatment of powder mixture up to 2 h not only reduced the reaction temperature, but also induced a different reaction path. In addition, the amount of Mg₃(BO₃)₂, as a major by-product of the thermal synthesis, was significantly decreased by applying adequate mechanical activation. The phase analysis revealed that the NbB₂ phase was achieved after 3 h high energy ball milling in self-stunning mode; meanwhile, the formation of NbC was progressively completed after a longer period of milling up to 7 h. According to the morphological evolutions, the range of particle size was within 100 nm.

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1. Introduction

NbC and NbB₂ as transition metal carbides and borides have widespread high performance applications due to their attractive combination of properties such as high melting point, high hardness, good corrosion resistance and other high temperature mechanical properties [1–3]. Carbide and boride of niobium have received a great deal of attention owing to their applications in mechanical industry, chemistry and microelectronics fields [4]. These materials are used in refractories, cutting tools, drills, abrasives and wear-resistant pieces [3,4].

At present, many processes are available for the synthesis of NbC and NbB₂ powders separately and each process varies in the characteristics of the produced powder and the processing cost. Traditionally, NbC has been prepared by various methods, such as

the direct combination of niobium metal with carbon [5], self-propagating high-temperature reaction [6], carburization of the niobium oxides [1], carburization of niobium pentahalide [7], carburization of niobium oxide (Nb₂O₅) in 20% (v/v) CH₄/H₂ [2] and mechanically induced self-propagating reaction [3,8]. On the other hand, many techniques are reported for the production of NbB₂ powder such as solid state reaction between niobium and boron [9], self-propagating high-temperature synthesis (SHS) [10–12] and chemical vapor deposition method (CVD) [13]. Tsuchida and Kakuta [14] have investigated simultaneous synthesis of NbC and NbB₂ from the powder mixtures of Nb/B/C=2/2/1 by combining mechanical activation (MA) and SHS in air (MA–SHS). In another study [15], they have investigated the effect of particle size of Nb metal, the mixing ratio of Nb/B/C and the weight ratio of sample to balls on the MA–SHS in air process. Also, they sintered the powder mixture of NbC and NbB₂ by spark plasma sintering (SPS) to obtain the NbC/NbB₂ composite compact [4].

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Due to the formation of various by-products and some general restrictions in conventional techniques such as high temperature operation, grain growth, non-uniform phase and so on, it is necessary to find an effective technique that could overcome these limitations.

Mechanochemical activation as a type of in-situ method is a solid state powder process which involves inducing chemical reactions in a mixture of as-received powders at room temperature or at least much lower than synthesis temperature. An increase in the kinetics of reactions during high energy milling can be resulted from microstructural refinement, repeated cold deformation and fracture of particles [16,17]. With the advantages of time and energy savings, simplicity, and high-purity products, the mechanochemical process represents an attractive alternative to the conventional methods in producing advanced materials, including borides, carbides, nitrides, intermetallics, etc. We have so far applied the mechanochemical technique to the synthesis of carbides and/or borides of Mo, Ti, and B [18–21]. The common point of all these reactions is a great amount of the heat created as a result of the exothermic reaction between metal oxides and aluminum/magnesium during the aluminothermic/magnesiothermic reduction. Afterwards, the elemental metals obtained from reduction process react with boride/carbide agents and synthesis reactions occur. The mechanochemical method was used to prepare NbB₂ powder via reaction between powder mixtures of Nb–B (1:2) by Iizumi et al. [22]. In addition, Tsuchida and Azuma [23] reported that NbC, Nb₂C and Nb₂N were obtained as main products by mechanically activated mixtures of Nb and C mixed in various mole fractions (C = 10–80 mol%).

In this work, the simultaneous mechanosynthesis of nanocrystalline NbC and NbB₂ composites was investigated using Mg, Nb, boron oxide and graphite. The main purpose of our work is to generate a comprehensive and consistent thermodynamic description of this system and to predict the reaction mechanism during the milling process based on the results from thermodynamic calculations, thermal analysis and the phase transformations.

2. Experimental procedure

The precursor materials were magnesium (Merck, 99.7% purity, particles size 40 ± 5 μm), boron oxide (Merck, 99.95% purity, mean particles size 30 ± 5 μm), niobium metal (Strem Chemicals, 99.8% purity, mean particles size 45 ± 5 μm) and graphite (Merck, 99.5% purity, mean particles size 50 μm). The specifications of the samples and values of the precursor materials are presented in Table 1.

The precursor materials were milled in a planetary ball mill for various durations at room temperature. Details of the ball mill machine and the milling conditions are given in Table 2. To prevent the oxidation process, the vial of milling was filled with high purity argon gas before ball milling.

In this study, amounts of the remained B₂O₃ and C were determined to understand the reaction mechanism. For calculation of the remained B₂O₃ value, firstly, the obtained powder from milling process was weighed and considered as W₁. Then,

Table 1

Specifications of the specimen and the contents of precursor materials.

Type	Compositions contents (wt%)			
	Mg	B ₂ O ₃	Nb	C
Magnesiothermic reduction	40.18	38.37	18.98	2.47

Table 2

Details of the ball mill machine and the milling conditions.

Rotation speed of vial (rpm)	500
Diameter of vial (mm)	100
Vial material	Hardened chromium steel
Ball material	Hardened carbon steel
Diameter of balls (mm)	20
Number of balls	5
Balls to powder weight ratio	20:1
Total powder mass (g)	7

the powder was washed by hot water (60–70 °C) for 1 h with a magnetic stirrer, then the solution was filtered and the residue on the filter was dried at 90 °C for 2 h, and then weighed as W₂. Hence W₁–W₂=W₃ is amount of the remaining B₂O₃. For removing probable Mg and MgO, the obtained powder was leached with 10% hydrochloric acid for 1 h. The solution was filtered after leaching and the purified products were repeatedly washed by distilled water to eliminate extra HCl acid until the pH value became about 7. Again, the residue on the filter was dried at 90 °C for 2 h and weighed as W₄. At the end, the obtained powder was oxidized at 700 °C for 30 min in the air and was weighed as W₅. Therefore, W₄–W₅=W₆ yields the amount of free carbon. Finally, to report the changes in data as percentage change, the following formula was used: [(obtained value/initial value)100]. In an attempt to reduce errors, the reported data were average of three experimental values.

XRD analysis was carried out using Cu-Kα radiation to identify different phases of the starting powders and mechanically alloyed powders. The diffractometer (Philips X-ray diffractometer) was operated at 40 kV and 30 mA. Scans were performed over 10° < 2θ < 90°. “PANalytical X’Pert High-Score” software was also used for the analysis of different peaks. The diffraction patterns of products were compared to proposed standards by the Joint Committee on Powder Diffraction and Standards (JCPDS).

The crystallite size and the lattice strain of the samples are calculated by using the Williamson–Hall method [16,17] and the equation

$$\beta_s \cos \theta = \frac{k\lambda}{d} + 2\eta \sin \theta \quad (1)$$

where β_s is the peak broadening in radians, 2θ the position of peak maximum, k the Scherrer constant (0.9), λ the X-ray wavelength (λ(CuKα₁)=1.540598 Å), d the so-called crystallite dimension and η an approximate upper limit of the lattice distortion. The instrumental broadening (β_i) was removed by

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