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Consolidation of nanostructured $NbSi_2$–SiC composite synthesized by high-frequency induction heated combustion

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Abstract

Dense nanostructured $NbSi_2$–SiC composite was synthesized by high-frequency induction heated combustion synthesis (HFIHCS) method within 2 min in one step from powders of NbC and 3Si. Simultaneous combustion synthesis and densification were accomplished under the combined effects of an induced current and mechanical pressure. Highly dense $NbSi_2$–SiC with relative density of up to 99.8% were produced under simultaneous application of a 60 MPa pressure and the induced current. The average grain size and mechanical properties (hardness and fracture toughness) of the composite were investigated.

Keywords: High-frequency induction heated combustion; Composite materials; Intermetallic; Sintering; Nanofabrication; Mechanical properties

1. Introduction

Refractory metal silicides have received considerable attention in recent years for their potential application as high-temperature structure materials [1]. Their properties provide a desirable combination of a high melting temperature, high modulus, high oxidation resistance in air, and a relatively low density [2,3]. Silicon carbide also is one of the most attractive high-temperature materials under investigation because of its excellent creep resistance and oxidation behavior.

However, as in the case of many intermetallic compounds, the current concern about these materials focuses on their low fracture toughness below the ductile-brittle transition temperature [4–6]. To improve on their mechanical properties, it is more effective to use as form of composite with other carbide, boride and silicide such as TiC–SiC, TiB_2–SiC, TiB_2–TiC, $MoSi_2$–SiC and WSi_2–$MoSi_2$ more than individual phases [5–12]. Furthermore, SiC little influence the oxidation resistance of metal silicides and forms a protective SiO_2 layer to show outstanding oxidation resistance [13]. Therefore, SiC may be the most promising additive as a reinforcing material for $NbSi_2$-based composites.

Many similar high-temperature dense composites are usually prepared in a multistep process [14,15]. However, the method of field-activated and pressure-assisted combustion synthesis has been successfully employed to synthesize and densify materials from the elements in one step in a relatively short period of time. This method has been used to synthesize a variety of ceramics and composites, including $MoSi_2$–ZrO_2, Ti_5Si_3 and its composites, WSi_2 and its composites, and WC–Co hard materials [16–21]. These materials that are generally characterized by low adiabatic combustion temperature cannot be synthesized directly by the self-propagating high-temperature synthesis (SHS) method. More recently, a new approach has been developed in which synthesis and densification can be effected simultaneously. This new process, referred to as the high-frequency induction heated combustion synthesis (HFIHCS), has been successfully used to synthesize and densify, in one step, some materials in a relatively short period of time (2 min) [22–24]. The purpose of this work is to produce dense nanophase $NbSi_2$–SiC composite within 2 min in one step from mixtures of NbC and 3Si powders by using this method and to evaluate its mechanical properties.

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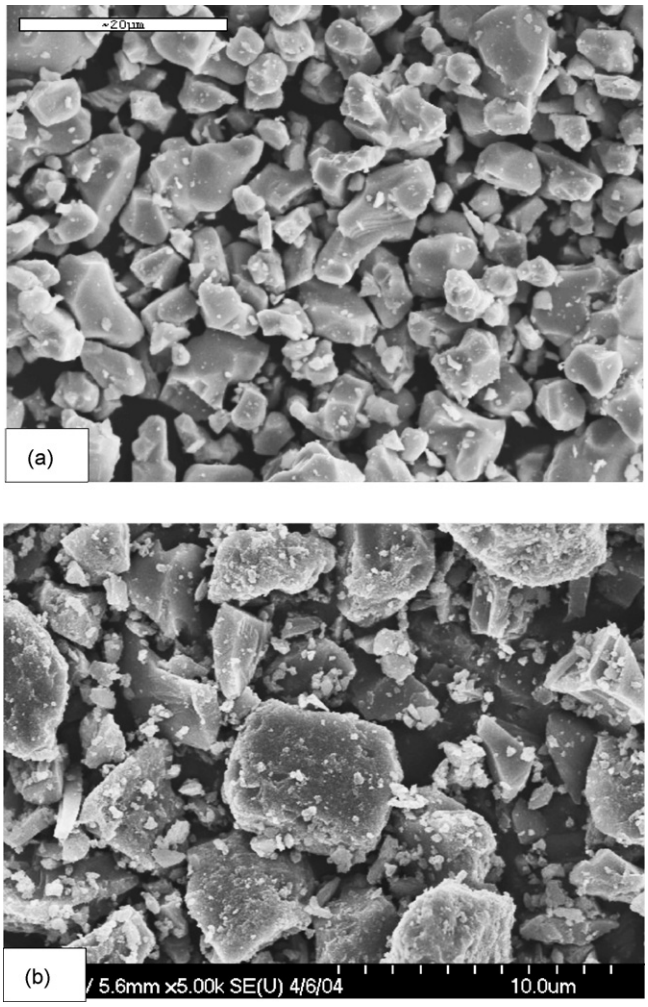

Fig. 1. Scanning electron microscope images of raw materials: (a) niobium carbide and (b) silicon powder.

2. Experimental procedure

Powders of 99.5% niobium carbide (−325 mesh, Cerac Products) and 99.5% pure silicon (−325 mesh, Alfa Products) were used as a starting materials. Fig. 1 shows the SEM images of the raw materials used. NbC and 3Si powder mixtures were first milled in a high-energy ball mill, Pulverisette-5 planetary mill with 250 rpm and for 10 h. Tungsten carbide balls (5 mm in diameter) were used in a sealed cylindrical stainless steel vial under argon atmosphere. The weight ratio of ball-to-powder was 30:1. Milling resulted in a significant reduction of grain size. The grain size and the internal strain are calculated by Strokes and Wilson's formula [25]:

$$b = b_d + b_e = k\ell/(d\cos\theta) + 4\varepsilon\tan\theta$$

where b is the full width at half-maximum (FWHM) of the diffraction peak after instrument correction; b_d and b_e are FWHM caused by small grain size and internal stress, respectively; K is constant as 0.9; ℓ is wavelength of the X-ray radiation; d and ε are grain size and internal stress, respectively; and θ is the Bragg angle. b and b_s follow Cauchy form with the relationship: $B_0 = b + b_s$, where B_0 and b_s are FWHM of broadened Bragg peaks and the standard sample's Bragg peaks, respectively. Fig. 2 shows XRD patterns of raw powders and milled NbC + 3Si powder. The FWHM of milled powder is wider than that of raw powder due to an internal strain and a reduction of grain size. The average grain size NbC measured by Stoke–Wilson equation was about 40 nm.

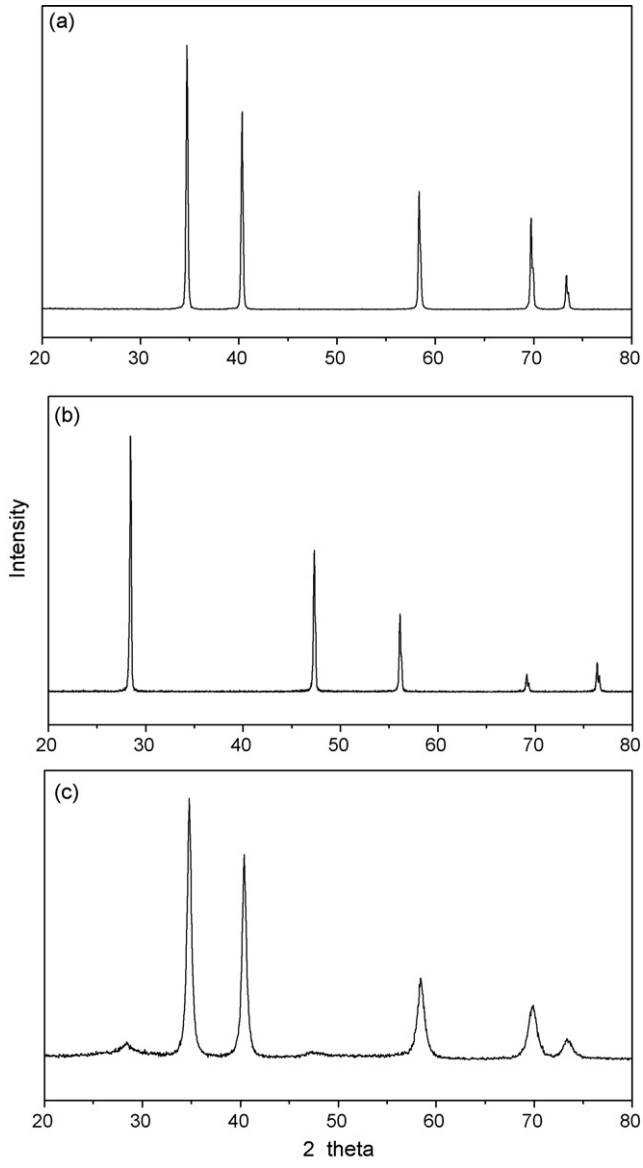

Fig. 2. XRD patterns of raw materials: (a) NbC, (b) Si and (c) milled NbC + 3Si.

After milling, the mixed powders were placed in a graphite die (outside diameter, 45 mm; inside diameter, 20 mm; height, 40 mm) and then introduced into the high-frequency induction heated combustion system, shown schematically in Fig. 3. The four major stages in the synthesis are as follows. The system was evacuated (stage 1). And a uniaxial pressure of 60 MPa was applied (stage 2). An induced current (frequency of about 50 kHz) was then activated and maintained until densification was attained as indicated by a linear gauge measuring the shrinkage of the sample (stage 3). Temperature was measured by a pyrometer focused on the surface of the graphite die. At the end of the process, the sample was cooled to room temperature (stage 4). Typical parameters for the process are presented in Table 1. The process was carried out under a vacuum of 40 mTorr.

The relative densities of the synthesized sample were measured by the Archimedes method. Microstructural information was obtained from product samples which were polished and etched using a solution of HF (15 vol.%), HNO_3 (35 vol.%) and H_2O (50 vol.%) for 1 min at room temperature. Compositional and micro structural analyses of the products were made through X-ray diffraction (XRD) and scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDAX). Vickers hardness was measured by performing indentations at load of 10 kg and a dwell time of 15 s on the synthesized samples.