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Phase identification of Al–B_4C ceramic composites synthesized by reaction hot-press sintering

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ABSTRACT

A series of boron carbide (B_4C) matrix composites with different contents of Al, were synthesized by reaction hot-press sintering with milled B_4C and pure metallic Al powder at 1600 °C for 1 h. X-ray diffraction (XRD), scanning electron microscope (SEM) and transmission electron microscopy (TEM) were used to identify the phase constituent of the milled powders and the composites. The results have shown that parts of B_4C and Al particles were oxidized to boron oxide (B_2O_3) and alumina (Al_2O_3) during the milling. Thermit reaction occurred and B_2O_3 was reverted during hot-press sintering. A ternary phase of Al boron carbide ($Al_8B_4C_7$) was found in the composites, and the B_4C transformed to a rich boron phase ($B_{6.5}C$) because of the superfluous boron in the system.

1. Introduction

The unique combination of extremely high hardness, wear resistance, low specific gravity, and high chemical stability makes boron carbide (B_4C) ceramics a candidate material for a variety of structural applications [1]. For instance, it is employed as lightweight armor plates for the ballistic properties, and employed as wear-resistant components for the tribological property. In addition, boron carbide can also be used as a neutron absorber in nuclear reactors associated with its high boron content [2–4]. However, the prevailing covalent character of the bonds in the crystal lattice of B_4C determines both its valuable properties and low sinterability. Monolithic boron carbide, without applied pressure, cannot be sintered to obtain satisfactory densities even at the temperatures over 2280 °C [5]. Another major problem for B_4C ceramic is extreme susceptibility to brittle fracture. Researchers have known that combining B_4C with metal could solve the recognized difficulties with B_4C [6–8]. They have focused on Al because of its lightweight, ready availability and reactivity with B_4C under reasonable processing conditions [9,10]. Hence, B_4C–Al composites have the potential to combine the high stiffness and hardness of B_4C with the ductility of Al, and without defeating the goal of obtaining a strong and low density material. There are a variety of binary and ternary compounds in B_4C–Al system. Arslan et al. [11] have reported that B_4C–Al composites are composed of various combinations of Al_3BC, AlB_2, $AlB_{12}C_2$ and Al_4C_3 phases, when the composites were fabricated by infiltration of Al into porous B_4C between 985 and 1370 °C. Viala et al. [3] have also reported the chemical reactivity of Al with B_4C by Al–B_4C powder mixture at temperature ranging from 627 to 1000 °C, they found that besides Al_3BC, the $Al_3B_{48}C_2$ replaced AlB_2 at temperature higher than 868 °C. For the pressure sintering, the temperature is often over 1600 °C, few reports were found about the chemical reactivity of Al with B_4C at this temperature.

Recently, we have synthesized Al–B_4C composites at 1600 °C with the milled B_4C and metallic Al particles, and found that the chemical reactivity of Al with B_4C had been drastically changed at 1600 °C. The present study is concentrated on the reactant phase identification and the chemical reactivity of B_4C with liquid Al at 1600 °C.

2. Experimental procedure

Samples studied in this work were prepared from commercial powders of Al (purity 99.6 wt.%; grain size, d_{50} = 28.5 μm, Northeast Light Alloy Co., Ltd.), and boron carbide (stoichiometric B_4C with C traces, purity 99.4 wt.%; grain size, d_{50} = 4.5 μm, Jingangzhuan, Co., Ltd.). These two kinds of powders were loaded in a stainless steel vial and milled for 5 h using a centrifugal planetary ball mill.

A vacuum stainless steel vial (250 ml) and stainless steel balls (with the diameters of 10 and 60 mm) were used for milling. The rotational speed of the vial is 300 rpm. Four specimens with Al contents of about 5 wt.%, 10 wt.%, 20 wt.%, and 40 wt.% are pre-

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pared, named BCA5, BCA10, BCA20, and BCA40, respectively. The milled powders were put into the graphite die and were sintered in the Ar gas at 1600 °C with 35 MPa of compressive stress for 1 h. Pure B_4C sample was used as the reference.

The phases of milled powders and the composites were identified by an XRD (Philips X'pert) diffractometer with Cu Kα radiation operated at 40 kV and 40 mA, the scanning speed was 5°/min with a scan step of 0.03°. The surface morphologies of milled powders and the fracture morphologies of the composites were observed by a SEM (Hitachi S-4700). The microstructures of the composites were observed by a TEM (Philips, CM-12), operated at an acceleration voltage of 120 kV.

3. Results and discussion

Fig. 1 shows SEM images of the milled powders of B_4C mixed with different contents of Al. From Fig. 1 we can see the powders

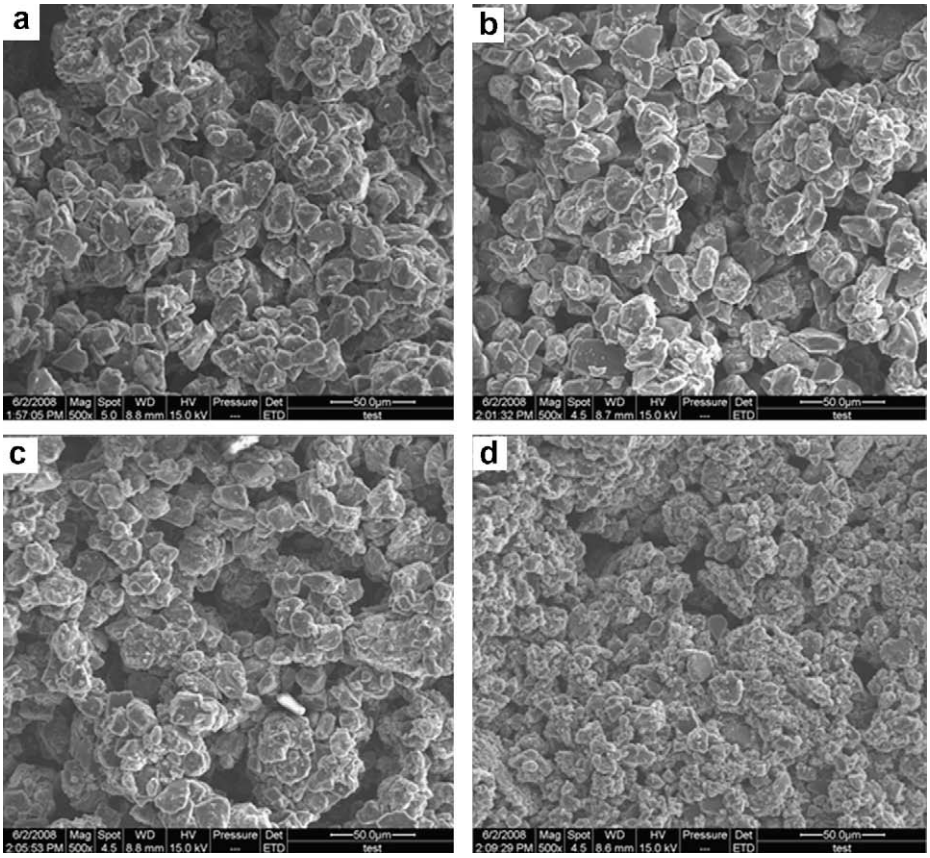

Fig. 1. SEM images of the milled powders of (a) BCA5; (b) BCA10; (c) BCA20; (d) BCA40.

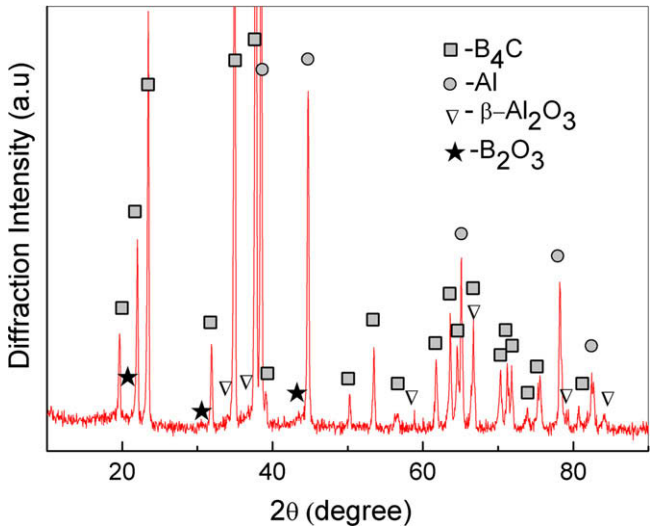

Fig. 2. XRD pattern of milled powder of BCA20.

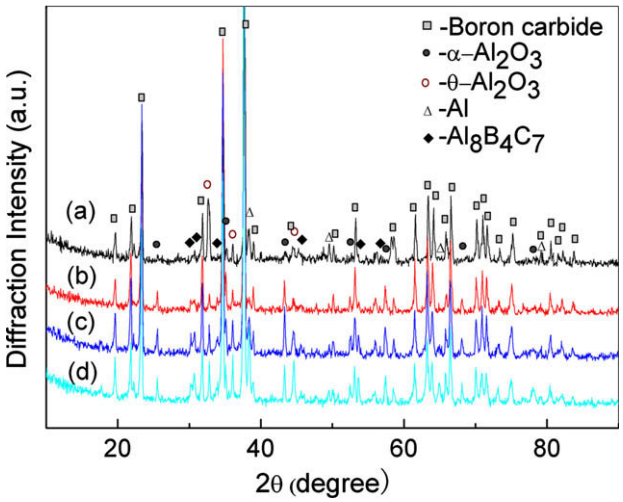

Fig. 3. XRD patterns of composites of (a) BCA5; (b) BCA10; (c) BCA20; (d) BCA40.