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Synthesis and microstructural characterization of aluminum borate whiskers

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Abstract

Porous ceramic with a framework structure of aluminum borate ($9Al_2O_3\cdot 2B_2O_3$) whiskers was in situ synthesized by firing above 1150 °C a green powder compact of a mixture of aluminum hydroxide, boric acid and an additive of nickel oxide. During sintering, the whiskers of aluminum borate grew in situ in the compact, and were bonded together. The porous aluminum borate consisted solely of whiskers with a porosity of 54–58%. The average diameters of the whiskers increased from 0.2 to 2 μm with increasing sintering temperature from 1150 to 1350 °C. However, the estimated whiskers aspect ratio decreased with increasing sintering temperature.

Keywords: In situ synthesis; Aluminum borate whiskers; Microstructure

1. Introduction

Discontinuously reinforced metal matrix composites (DRMMCs) have been widely studied during the past several decades, and successfully used in the automobile and aerospace industries due to their light weight, high wear resistance and the retention of mechanical properties at elevated temperatures [1,2]. Among the reinforcements for DRMMCs, ceramic whiskers, such as silicon carbide, silicon nitride and aluminum borate, were usually used. However, in order to promote application of MMCs, it is important to choose some cost-effective reinforcements and fabrication techniques. It has been found that the aluminum borate ($9Al_2O_3\cdot 2B_2O_3$) whisker is one of the cheapest ceramic whiskers, and therefore, used to reinforce aluminum matrix composites [3–8], whose Young's modulus, strength and creep resistance are similar to those of SiC whisker reinforced Al composites.

Ceramic whiskers were synthesized by the vapor–liquid–solid (VLS) method [9,10] and flux technique [11]. Since it is difficult to incorporate uniformly extremely fine whiskers with diameters of 0.1–0.2 μm and an aspect ratio of 10–100 in molten metal, whisker-reinforced MMCs have generally been fabricated by previously formed whiskers in a preform of porous body and then infiltrating molten metal under a preset pressure into the pores of the preform [8,12,13]. Accordingly, it is necessary firstly to prepare porous whisker preform.

In the present investigation, a new porous aluminum borate ($9Al_2O_3\cdot 2B_2O_3$) whisker preform comprised of a framework structure were synthesized in situ by firing a green powder compact of a mixture of aluminum hydroxide ($Al(OH)_3$), boric acid (H_3BO_3) and an additive of nickel oxide (NiO). The crystal structure and microstructure of this new porous ceramic was identified by X-ray diffraction (XRD) and observed through scanning electron microscopy (SEM).

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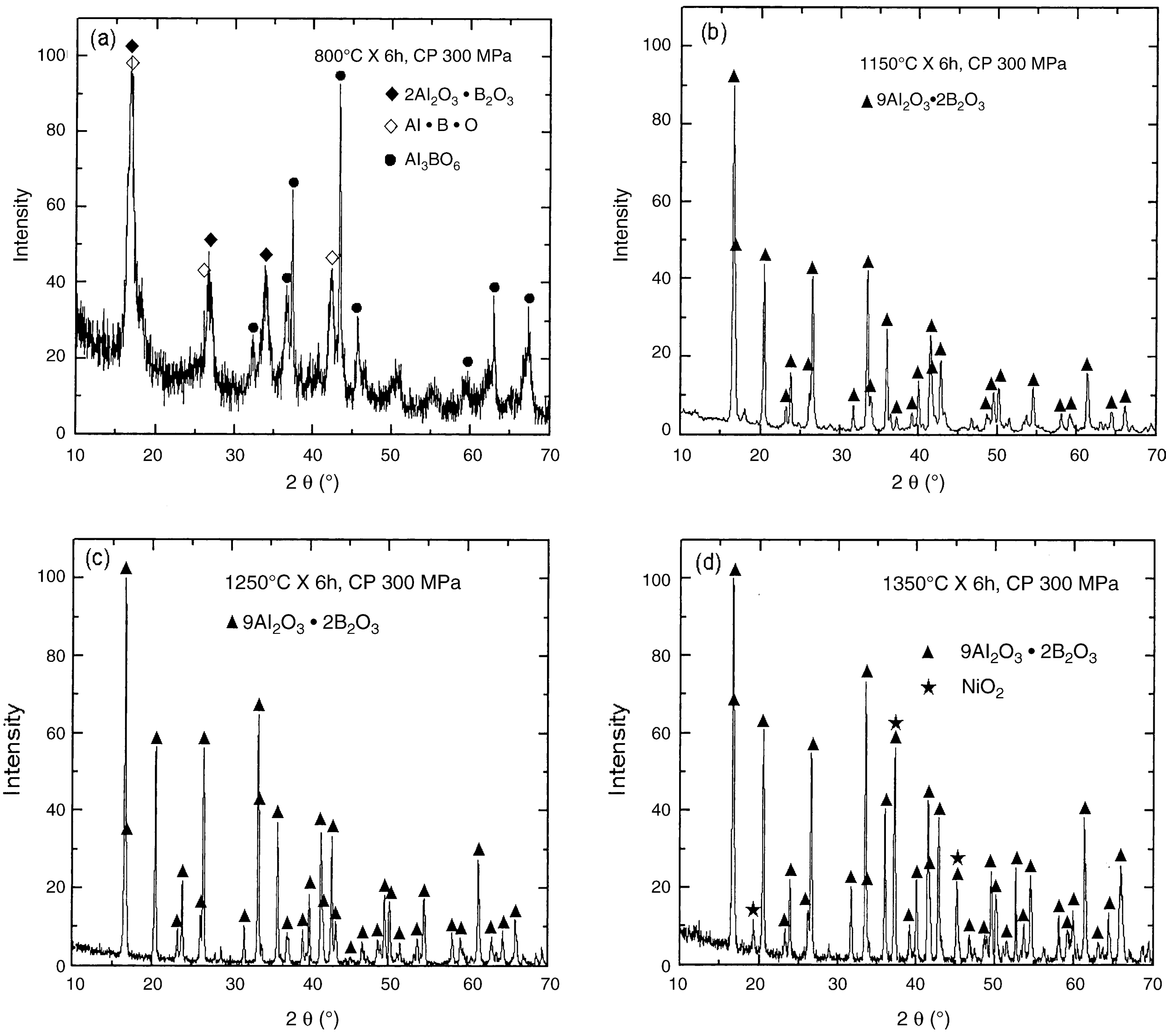

Fig. 1. XRD pattern for samples sintered in air at (a) 800 °C, (b) 1150 °C, (c) 1250 °C and (d) 1350 °C for 6 h, respectively.

2. Experimental procedures

Commercial aluminum hydroxide, boric acid and nickel oxide were finely powdered and uniformly blended, where the boric acid was added in excess with the mixing molar ratio of $Al(OH)_3$ to H_3BO_3 as 9:4 and the amount of NiO additive was 3 wt.%. The mixed powders were then uniaxially die-pressed under a pressure between 200 and 400 MPa into platelets with dimensions of 36 mm × 18 mm × 10 mm.

The compact was sintered in a furnace in air at a temperature ranging from 800 to 1350 °C for 6 h. After cooling, the sintered body was washed in hot water to remove residual boron oxide. The bulk density and porosity of the sintered body was estimated by 1-butanol ($CH_3(CH_2)_2CH_2OH$) immersion in vacuum. The microstructures of the sintered body were examined using X-ray diffraction and scanning electron microscopy.

3. Results and discussion

3.1. X-ray diffraction

The X-ray diffraction patterns of sintered compacts under different conditions were presented in Fig. 1. When the compact was calcined to 800 °C for 6 h, the reaction between non-crystalline aluminum oxide and molten boron oxide occurred, and the mixture peaks of $2Al_2O_3{\cdot}B_2O_3$, Al_3BO_6 and amorphous phase Al·B·O were confirmed in the samples (Fig. 1(a)). This was inconsistent with the results obtained by Li et al. [5], where the resultant phases contained some $9Al_2O_3{\cdot}2B_2O_3$ as well as $2Al_2O_3{\cdot}B_2O_3$ in the compact fired under the same condition as the present investigation. In the compacts sintered above 1150 °C for 6 h, only $9Al_2O_3{\cdot}2B_2O_3$ was formed (Fig. 1(b) and (c)), which has an orthorhombic structure with lattice parameters $a = 0.77$ nm, $b = 1.50$ nm and $c = 0.57$ nm, and the four rectangle surfaces are {1 2 0} and the four corner surfaces are {1 1 0} [4]. However, when the calcining temperature was raised to 1350 °C, a trace of NiO_2 was detected, which was transformed from NiO at elevated temperature and whose peaks were overlapped by the peaks of $9Al_2O_3{\cdot}2B_2O_3$ (Fig. 1(d)). It was documented [14] that $9Al_2O_3{\cdot}2B_2O_3$, which has a high melting point (1950 °C), was in equilibrium with a B_2O_3 liquid above 1035 °C on the phase diagram of Al_2O_3–$2B_2O_3$ binary system as shown in Fig. 2, and $9Al_2O_3{\cdot}2B_2O_3$ and $2Al_2O_3{\cdot}B_2O_3$ were solid phases in equilibrium below this temperature in the present Al/B molar ratio, but the former phase was stable with excess molten boron oxide during cooling.

3.2. The microstructure of porous aluminum borate

The SEM microstructures of compacts sintered from 800 to 1350 °C for 6 h are shown in Fig. 3. It can be found that the compact sintered at 800 °C consisted mainly of amorphous phase without $9Al_2O_3{\cdot}2B_2O_3$ formed. $9Al_2O_3{\cdot}2B_2O_3$ whiskers were found in all other samples sintered above 1150 °C. These were coincident with the results indicated by X-ray

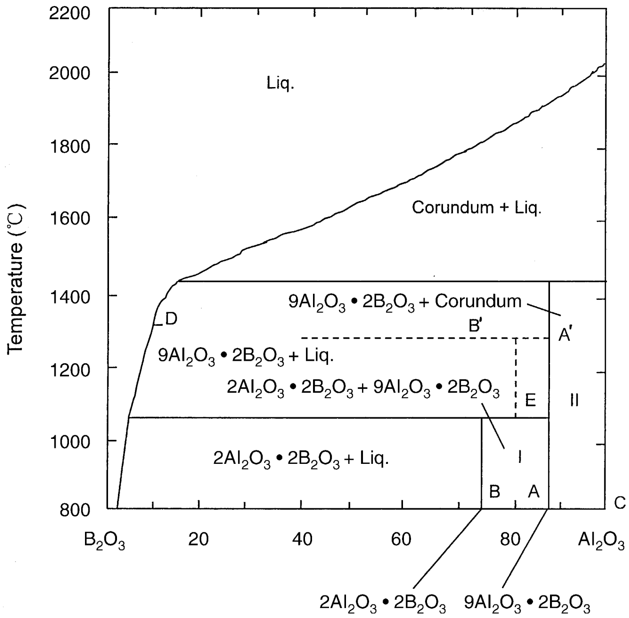

Fig. 2. Phase diagram of Al_2O_3–B_2O_3 binary system [14].

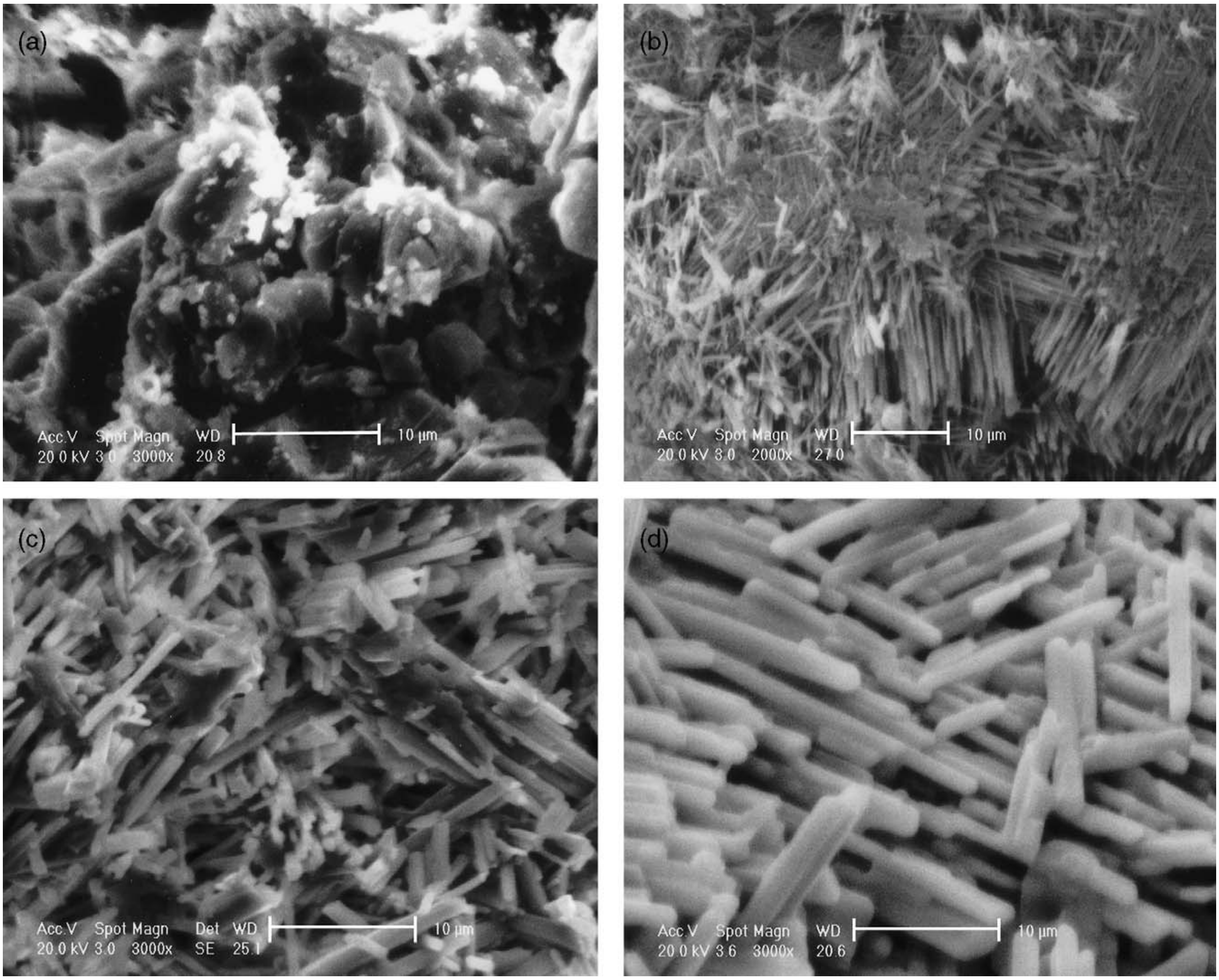

Fig. 3. Scanning electron micrographs of compacts sintered at (a) 800 °C, (b) 1150 °C, (c) 1250 °C and (d) 1350 °C for 6 h, respectively.

diffraction. The average diameters of the whiskers increased from 0.2 to 2 μm with increasing sintering temperature from 1150 to 1350 °C. Unfortunately, it was impossible to separate the whiskers from the bulk for measuring the accurate length due to the moderately high bonding strength between whiskers. In general, the estimated aspect ratio of the length to diameter of whiskers decreased with increasing sintering temperature. In the presence of excess melt of boron oxide and added nickel oxide, aluminum borate whiskers nucleated uniformly throughout the compact and grew within a dense compact. As a result, the formed whiskers distributed randomly in three dimensions and a framework or porous structure was present. The addition of nickel oxide accelerated the reaction between aluminum oxide and boron oxide at high temperature and increased the aspect ratio of the length to diameter of aluminum borate whiskers [5]. The porosity of the sintered compacted varied in a narrow range from 0.54 to 0.58, almost independent of pressure and sintering conditions.

4. Conclusion

Porous aluminum borate ($9Al_2O_3{\cdot}2B_2O_3$) whiskers with a framework structure was in situ synthesized by sintering of a green powder compact of a mixture of aluminum hydroxide ($Al(OH)_3$), boric acid (H_3BO_3) and an additive of nickel oxide (NiO) above 1150 °C. The samples consisted solely of whiskers with a porosity of 54–58%. The average diameters of the whiskers increased and the estimated aspect ratio of the length to diameter of whiskers decreased with increasing sintering temperature from 1150 to 1350 °C.

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