Effects of extrusion temperatures on mechanical properties of B4C/Al composites

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Abstract. This article studied the effects of extrusion at different temperatures on the microstructures and mechanical properties of B4C particle reinforced Al neutron shielding composite produced by powder metallurgy method. The results indicated that, the interfacial bonding between the B4C particles and the matrix was greatly improved by hot extrusion at studied temperatures. In addition, the mechanical properties of the composites were obviously improved after hot extrusion. However, by further increased the extrusion temperature, yield strength (YS) and ultimate tensile strength (UTS) of the composites decreases. Meanwhile, the fractography of extruded composite revealed that fracture mechanism changed from particle brittle cracking to matrix plastic damage as extrusion temperature increased.

1. Introduction

In the industrial field, the common reinforcements of aluminum matrix composites include silicon carbide (SiC), aluminum oxide (Al2O3), boron carbide (B4C) and so on. Among above reinforcements, B4C as a potential reinforcement is widely used in neutron absorption industry with excellent capacity for neutron absorption [1-3]. In recent years, the study of B4C/Al composites has made much progress. Many conventional fabrication methods have been used to produce the composites, such as liquid-stirring casting [4], pressure infiltration [5], powder metallurgy (PM) [6], and so on. Compared with other manufacturing techniques, the powder metallurgy (PM) method is attractive, because of the easy controlling of microstructures. However, PM processing technique normally employs lower temperature sintering, resulting in the lower diffusion rates and weak interfacial bonding strength. Another problem of the PM method is that the Al2O3 film on most commercially atomized powders can act as the barrier for the atom diffusion during sintering, especially solid phase sintering. Under that condition, it is commonly accepted secondary hot deformation like extrusion, which can significantly improve interfacial bonding strength and uniformity of added particles, and finally improve its density and mechanical properties [7-9]. As for hot extrusion, extrusion temperatures can significantly affect grain size, distribution of large particles, dynamic recrystallization (DRX) in the material, thus affect the mechanical properties of composites. However, few research exported the details of above influences.

Thus, this paper further analyzed the extrusion temperatures and their influence on the mechanical property of the composites. 12 vol.% B4C/Al composite was successfully prepared by using fine atomized Al powders and B4C particles and then followed hot extrusion process. The study of the microstructure and tension property of the composites were carried out.
2. Experimental

2.1. Raw materials

The raw materials used in this study were B₄C particles (99% in purity) and fine atomized Al powders (99.9% in purity). The average size of B₄C particles and Al powders were 4.3μm and 1.5μm, respectively. Figure 1 shows scanning electron microscopy (SEM) of B₄C particles and Al powders.

![Figure 1. The SEM micrographs of Al and B₄C particles with an insert of particle size distribution (a) Al-1.5μm, (b) B₄C-4.3μm.](image)

2.2. Processing

The aluminum powders were mixed with about 12 vol.% B₄C particulates in alcohol medium for 2 hours by using an attritor at 150 rpm/min. The ball to powder weight ratio was 6:1. The mixed powders were dried in air and compacted by cold isostatic pressing (CIP) at 400 MPa. The specimens were heated in a vacuum furnace with a heating rate of 10°C per minute to 450°C for 12 hours. The B₄C/Al billets with dimension of Φ 75 mm and thickness of 100 mm were machined from the as-sintered samples. And then conducted the preparation for the extrusion experiments. The surfaces of the billets, extrusion die and dummy block were coated with a graphite-base lubricant. The billets were put into the container and the extrusion container was heated to given temperatures in a pit-type furnace. Soaking time of 1 hour was set after the container attained the predetermined temperature. Billets were extruded with an extrusion ratio of 22:1 as well as a ram speed of 2 mm/s and then cooled in air. Three extrusion temperatures (540, 570 and 600°C) were employed.

2.3. Testing

Microstructural examination was carried out by scanning electron microscopy (SEM) with energy dispersive spectrometer (EDS) and electron back-scatter diffraction (EBSD) system. Specimens were cut parallel to extrusion direction with a size of 2×4×8 mm³. The specimens for SEM and EBSD were polished by mechanical polishing and followed by ion beam (IB-09020CP, JEOL) polishing. Room temperature tensile test samples were machined into rectangular specimens, conforming to ASTM E8M standard. Tensile tests were carried out at a strain rate of 1×10⁻³ s⁻¹ on an MTS-LANDMARK 370.25 test system. The density of the as-sintered and the as-extruded samples were measured by the Method of Archimedes.

3. Results and discussion

3.1. Microstructures

Figure 2 shows the SEM micrographs of B₄C/Al composites before and after extrusion at different temperatures. Clusters of B₄C particles (red circles) and micro-pores (red arrow) are observed in the
samples before extrusion (Figure 2a and e). Meanwhile, adjacent spherical aluminum powders and the poor interface between the boron carbide and matrix can be clearly observed. By comparing with SEM micrographs of B$_4$C/Al composites before and after extrusion, the B$_4$C particles distribution is significantly improved by extrusion. For all extruded samples, it is hard to see the clusters of B$_4$C particles (Figure 2b, c and d). And the interface between B$_4$C particles and matrix is clean and no cracks which can be seen in figure 2f, g and h. As can be seen from the longitudinal section, the number of pores in the as-extruded composite were completely eliminated in all set temperatures.

The distribution of reinforcement particles depended on the blending and the consolidation procedures as well as the matrix-to-reinforcement particle size ratios [10]. In this study, micro-pores cannot be completely eliminated during sintering due to the Al$_2$O$_3$ film on the surface of fine atomized Al powders. During the hot extrusion, shearing enlarges the surface of the powders, and breaks up the Al$_2$O$_3$ film. Besides, the severe plastic deformation of the matrix, leading to the densification of aluminum matrix composites and rearrangement of the reinforcements [11-12].

![SEM micrographs for the composites before extrusion and being extruded at different extrusion temperatures.](image)

Figure 2. SEM micrographs for the composites before extrusion and being extruded at different extrusion temperatures: (a and e) before extrusion, (b and f) 540 °C, (c and g) 570 °C, (d and h) 600 °C.

Figure 3 shows the EBSD maps and grain statistical distribution of the composites extruded at different temperatures. The average grain size of composites increases as extrusion temperature increased. The average grain sizes of the composites extruded at temperatures of 540, 570 and 600 °C are 0.7, 0.9 and 2.2 μm, respectively. In the E540 sample, only fine grains are observed, indicating that the full dynamic recrystallization (DRX) occurred during deformation. In the E570 and E600 samples, the grains became coarser, caused by higher extrusion temperature.

In a word, for 12vol.% B$_4$C/Al composites, when the temperature of hot extrusion reaches or exceeds 540°C, the micro-pores in the matrix can be completely eliminated and the interface between B$_4$C particles and matrix is smooth. Meanwhile, hot extrusion at this temperature could result in a uniform particle distribution. And the density of extruded samples increases as the extrusion temperature increases. In addition, extrusion temperature greatly affects the size of matrix grains, the average grain size of composites increases as extrusion temperature increased. So that the hardness of being extruded samples decrease as the extrusion temperature increased.
Fig. 3. EBSD maps of the composites extruded at different extrusion temperatures: (a) 540°C, (b) 570°C, (c) 600°C. The grain size distribution obtained from EBSD measurements ($d_{EBSD}$) are shown in (d), (e), (f), respectively.

3.2. Mechanical properties of the extruded composites

Figure 4 shows the tensile mechanical properties of the composites extruded at different extrusion temperatures. The ultimate tensile strength of the E540 sample is 256 MPa, which is almost three times compared with sample before extrusion in this study (90 MPa). That is mainly attributed to the fine grain, uniform B$_4$C distribution and improved interface bonding strength between B$_4$C and matrix. As the extrusion temperature increasing, the yield strength (YS) and ultimate tensile strength (UTS) of the composites decrease, while the elongation of the composites increases.

Fig. 4. Mechanical properties of as-extruded composites: (a) YS and UTS, (b) elongation.

The mechanical properties of composites depend on interface bonding strength, particle dispersion and matrix grain size. A strong interfacial bonding helps to improve composites strength by transferring external load from soft matrix to hard reinforcements[13,14] Besides, the grain boundaries and reinforced particles in the composites act as the obstacle for dislocation movement to improve the composites’ strength [15].

3.3. Fracture behavior of the composites

Figure 5 shows the fractograph of the composites extruded at different temperatures. In figure 5a, lots of fractured B$_4$C particles exist on the fracture surface (marked by red arrows) and shallow dimples in
the matrix can be observed. As the extrusion temperature increased, the number of the fractured B$_4$C particles reduce (Figure 5b) and even disappear (Figure 5d), while the dimples are deeper (Figure 5e and 5f). At the same time, in the E540 sample (Figure 5d), large number of B$_4$C particles, especially with high aspect ratio, fractured and induced micro-cracks in the area near to tensile fracture. But, in the E600 sample (Figure 5f), there exists lots of micro-pores in the aluminum matrix and adjacent reinforcing phases without any fractured particles. There is a large mismatch between pure aluminum matrix and B$_4$C particles, and the plastic deformation of the soft matrix would be limited by the hard and brittle particles. Therefore, the fracture of composites is a combination of ductile fracture and brittle fracture.

![Fractographs](image)

**Figure 5.** Fractographs of the composites and morphologies of fracture sides of composites extruded at different extrusion temperatures: (a and d) 540°C, (b and e) 570°C, (c and f) 600°C.

4. Conclusions

In this work, the effects of extrusion temperature on the microstructures and mechanical properties of 12 vol.% B$_4$C/Al composite were studied. Several conclusions have been drawn:

1. For as-extruded 12 vol.% B$_4$C/Al composites, When the temperature of hot extrusion reaches or exceeds 540°C, can effectively reduce the number of micro-pores, increase the bonding strength of the interface between the B$_4$C particles and matrix, and uniform distribution of B$_4$C particles.

2. The 12 vol.% B$_4$C/Al composites extruded at 540°C had the highest strength (UTS=256 MPa, YS=190 MPa), which is about three times compared with sample before extrusion (90 MPa). The average grain size of composites increases as extrusion temperature increasing, resulting in the decrease of the yield strength and tensile strength.

3. The 12 vol.% B$_4$C/Al composites matrix strength decreases with the increased extrusion temperature, the fracture mechanism changes from particle brittle cracking to matrix plastic damage.

Acknowledgments

This work was founded by Shenzhen Engineering Laboratory of Nuclear Materials and Service Safety.

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