Original Article

Investigation on microstructural and mechanical properties of B₄C–aluminum matrix composites prepared by microwave sintering

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ABSTRACT

B₄C reinforced aluminum composites were fabricated by microwave heating of the mixture of B₄C (10, 15 and 20 wt%) and aluminum powders at 650, 750, 850 and 950 °C. The effect of different amounts of B₄C on the microstructure and mechanical properties of aluminum matrix was examined. The maximum bending (238 ± 10 MPa) and compressive strength (330 ± 10 MPa) values were measured for composites sintered at 950 and 750 °C, respectively. The maximum hardness (112 Vickers) was measured for Al–20 wt% B₄C composite sintered at 850 °C. XRD investigations showed the decomposition of boron carbide and also the formation of Al₃BC by heating the composites at 850 °C. SEM micrographs showed uniform distribution of reinforcement particles in Al matrix.

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1. Introduction

Among metal matrix composites, aluminum alloy based composites are known as very promising light materials with enhanced mechanical properties. In fact, by introducing a hard phase either in particulate or fiber form, the modulus and wear resistance of aluminum alloys could be significantly improved [1–3]. Among different parameters affected by the final properties of the composites, the nature of the reinforcement and processing route play a paramount role [4,5]. Boron carbide is an interesting material for many reasons. It has low density (2.51 g/cm³), excellent chemical resistance and is

extremely hard [6]. Additionally, boron carbide (B₄C) has a high neutron absorption cross-section making it a valuable material for nuclear applications [6]. Despite these advantages, the monolithic B₄C does not seem to be introducing well as an advanced structural ceramic due to high brittle nature related to shock induction localized at high strain rates [7]. It is known that combining the B₄C with a metal can mitigate the problems associated with brittleness. By far, the most popular metal used in joining B₄C for this purpose is aluminum [8–12]. Aluminum wets B₄C well at elevated temperatures. Molten aluminum has been shown to form a variety of binary and tertiary phases when in contact with B₄C such as Al₃BC, AlB₁₀ and Al₄C₃. Among these phases, Al₃BC is the most commonly

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observed phase [13,14]. The sintering of composite materials by microwave processing has been known to reduce heating temperature and time and lead to fine microstructures and improved mechanical properties [15–22].

The boron carbides react strongly with liquid aluminum, resulting in a variety of compounds, including Al_3BC, $AlB_{24}C_4$ (AlB_{10}), $Al8B_4C_7$, $Al_3B_{48}C_2$ (b-AlB_{12}), AlB_2, $AlB_{12}C_2$, $AlB_{48}C_2$, Al_4C_3 and a-$AlB_{12.7}$ [23]. Some studies have reported on the reactivity of B_4C in aluminum processed by infiltration and powder metallurgy techniques and on the formation of different compounds at different processing temperatures [14,24,25,26]. The main aims of the present work are the feasibility of the formation of aluminum–B_4C composites by using microwave heating and investigation of the mechanical and structural properties of these composites.

2. Experimental procedures

B_4C (Aldrich-378100) and aluminum (1056-merck) powders were used as the starting materials and cobalt powder (99.8% purity and 5 μm mean particle size) was used as an additive. The composition of composite samples is given in Table 1.

The mixing of powders presented in Table 1 was carried out using the Spex instrument (8000D, Mixer mill). The mixed powders were compacted to prepare the bar shape samples with the dimension of $25\,mm \times 5\,mm \times 5\,mm$ at 250 MPa using a uniaxial press. The compression strength test samples were fabricated using a hydraulic press type to obtain cylindrical preforms with a diameter of 10 mm and height of 8 mm. The sintering of samples was carried out using a microwave furnace (900 W and 2.45 GHz), which monitored temperature using an optical pyrometer (Model: RAYR312MSCL2G) at 650 °C, 750 °C, 850 °C and 950 °C without soaking time in a graphite bed. The bulk density of sintered samples was measured using the Archimedes' Principle. The three point bending and compressive strength measurements were examined by Santam-STm 20. For bending and compressive strength tests, five and three specimens were used, respectively. X-ray diffraction (Philips (30 kV and 25 mA) diffractometer system with CuK_α radiation ($\lambda = 1.5405$ Å)) analyses were performed to identify the phases present in the Al–B_4C composites. Vickers microhardness values of the sintered samples were determined using a Microhardness Tester (MKV-h21, Akashi) under a load of 1 kgf for 15 s. At least ten successive indentations were performed for each sample. Microstructural investigations and EDS analyses of sintered samples were carried out using a SEM (Stereoscan 360, Leica Cambridge).

Table 1 – Different compositions of composite samples.

Composite	Al (wt%)	B_4C (wt%)	Co (wt%)
A	90	10	0
B	89	10	1
C	83.5	15	1.5
D	78	20	2

3. Results and discussion

3.1. XRD analysis

For studying the effect of sintering temperature on the phase structure and chemical compound of the composites, four D composites (Table 1) were sintered at 650, 750, 850 and 950 °C. As Fig. 1 shows, the identified phases were Al, B_4C and Al_3BC. It seems that the formation of Al_3BC begins from the temperature of 850 °C [13].

3.2. Density

As can be seen in Fig. 2, there are no sharp changes in density of different composites sintered at the same temperature. Fig. 2 further reveals that composite A with no Co additive has the minimum density, which implies that Co additive can act as a binder between Al and B_4C particles. Meanwhile, it should be noted that the accurate calculation of relative density of samples is impossible due to partial decomposition of B_4C.
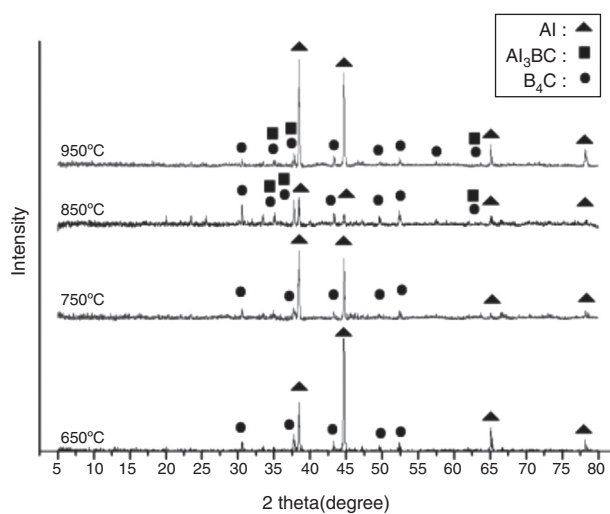

Fig. 1 – XRD patterns of composite containing 20 wt% B_4C sintered at 650 °C, 750 °C, 850 °C and 950 °C.
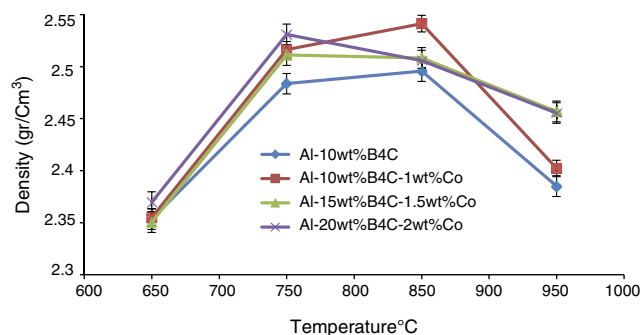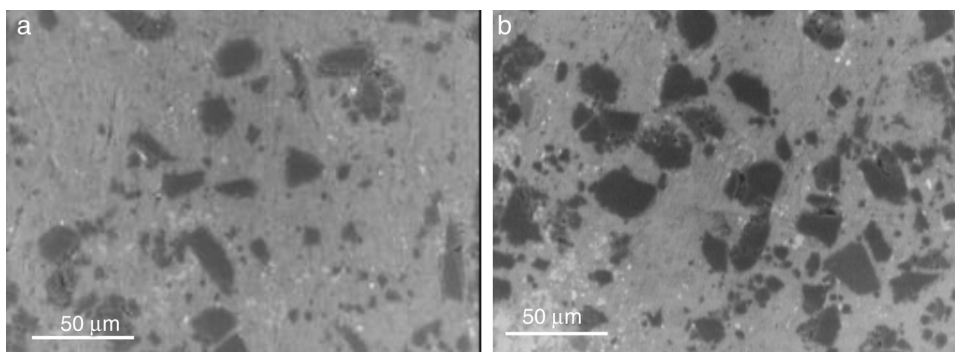

Fig. 2 – Density versus sintering temperature.

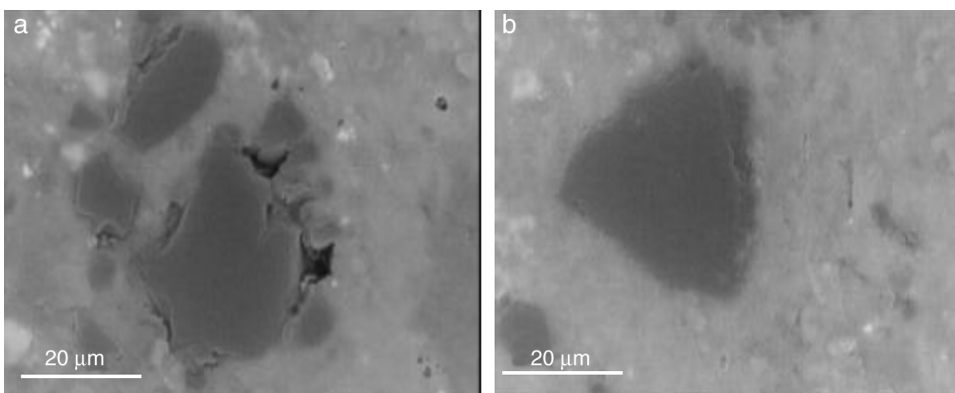**Fig. 3 – Backscattered images of B and C composites sintered at 850 °C: (a) Al–10 wt% B$_4$C–1 wt% Co and (b) Al–15 wt% B$_4$C–1.5 wt% Co.**

Fig. 4 – Backscattered images of A and B composites sintered at 750 °C: (a) Al–10 wt% B$_4$C and (b) Al–10 wt% B$_4$C–1 wt% Co.

3.3. Microstructural analysis

Mechanical and physical properties are highly affected by the microstructure of sintered composites. Figs. 3 and 4 illustrate the SEM micrographs of A, B and C composites. Homogeneous distribution of B$_4$C reinforcements can be seen typically in Fig. 3. From Fig. 4(a) and (b), it is observed that A composite in contrast to B composite has some porosity around the B$_4$C reinforcement particles. It seems that Co additive can improve the adherence of Al matrix to B$_4$C particles.

Fig. 5 shows SEM and EDS micrographs of Al–20% B$_4$C–2% Co composite sintered at 850 °C. Also, the EDS spectra, from some spots labeled 1 through 3, are shown in Fig. 5. Since the EDS analysis is incapable of detection of carbon and boron elements, the detection of Al peaks in the EDS spectra can be used as a criterion for proofing of B$_4$C existence in the microstructure. In spot 1, there are not any peaks, while at spot 2, Al peak is seen, which probably seems that the interfacial reaction and formation of Al$_3$BC phase have occurred. In spot 3 stronger peak of Al is observed in the matrix of composite. Finally, it seems that the dark and light regions are B$_4$C and Al particles, respectively and Al$_3$BC is probably formed at the interface of these particles.

3.4. Bending strength

The matrix hardening is mainly the consequence of three effects: (1) smaller grain sizes in the AMC matrix than in the alloy due to the reinforcement tangle. The hardening follows
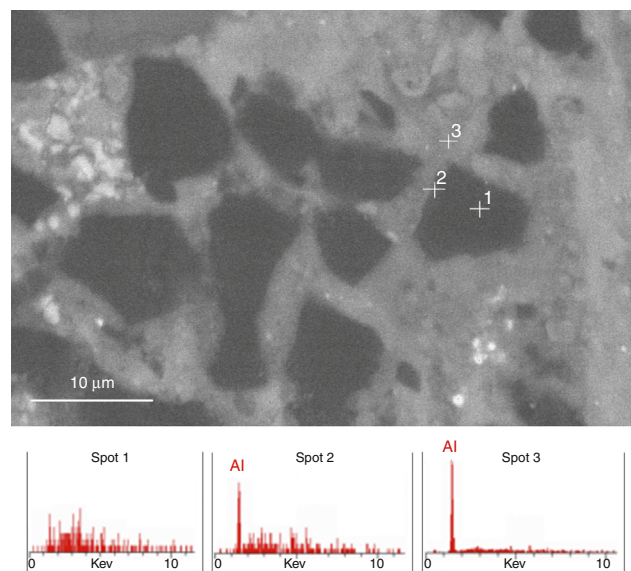

Fig. 5 – Backscattered image and EDS of Al–20% B$_4$C–2% Co composite sintered at 850 °C.

the Hall–Petch law: $\Delta\sigma^y \propto 1/\sqrt{D}$, where D is the grain size. (2) Higher dislocation density generated by the CTE mismatch between matrix and reinforcements. (3) Chemical reaction between the reinforcements particles and Al matrix, as it can have a significant effect on the interfacial characteristics and hence on the mechanical properties of the composite [27,28].
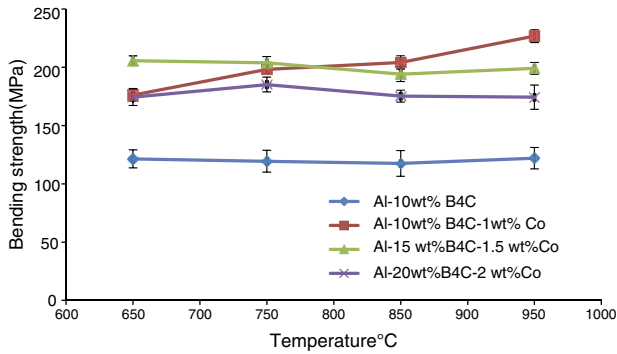
Fig. 6 – A plot showing variation in bending strength of composites with sintering temperature.

The bending strength of B_4C–Al composites fabricated in the present work was found to be in the range of 123–238 MPa for B_4C weight percent fractions in the range of 10–20%, respectively. Fig. 6 illustrates the bending strength changes of composites versus sintering temperature. As it is clear in Fig. 6, composites have the lowest bending strength among others and Co additive might promote the adhesion of Al to B_4C particles as observed in SEM micrographs (Fig. 4).

The C composites sintered at 650 and 750 °C have higher bending strength; however, with the raising temperature bending strength of B composites increases (Fig. 6). The interfacial reaction and formation of Al_3BC and also more cohesion between Al matrix and reinforcement as a result of higher sintering temperature which causes higher atomic diffusion could be the possible reason for the above mentioned. It is obvious that many parameters including interfacial reaction, porosity, microcracks, grains size, etc. affect on mechanical properties of composite; so the evaluation of each individual parameter regardless of the impact of others is unreasonable.

3.5. Compressive strength

The compressive strength results as a function of sintering temperature (Fig. 7) show that an increase in the amount of B_4C and sintering temperature lead to an increase in the compressive strength. As it was discussed in the bending strength section, the smaller grain size will lead to more grain boundaries, which can act as strong obstacles to the dislocation motion, leading to the increase of compressive strength.
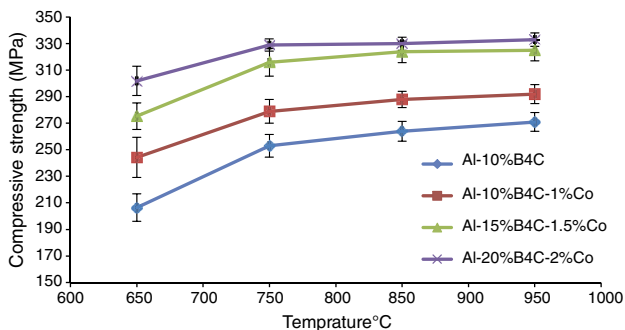

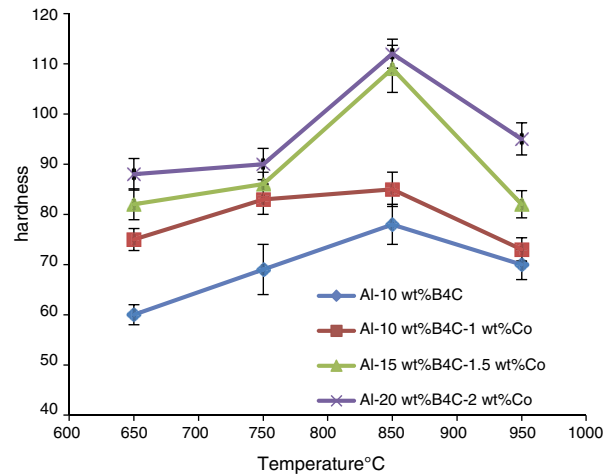**Fig. 7 – The effect of sintering temperature on compression strength.**

Fig. 8 – the effect of sintering temperature on microhardness on sintered samples.

The performed studies on the composites fabricated by microwave sintering revealed a narrow grain size with uniform distribution of the reinforcements, and well-bonded interfaces. This may be correlated with the advantage of microwave sintering, which have been proved to produce the composites with small grain size, homogeneous distribution of reinforcements and proper interfacial characteristic [29,30].

3.6. Microhardness

For all tested samples, the average microhardness of the microwave sintered composites was higher than the corresponding value for the reference 6061 Al alloy and the hardness increased with the increasing amount of B_4C. This can be explained by the law of mixtures in the following equation [19]:

$$H_c = H_m F_m + H_r F_r$$

where H_c, H_m, and H_r are the hardness of the composite, matrix and reinforcement, respectively, f_m and f_r are the fraction of matrix and reinforcement, respectively (Fig. 8).

The microhardness increased with increasing reinforcement, since B_4C is inherently harder than Al matrix and the fairly well distribution of the reinforcements improves the ability of the soft matrix to resist deformation. Sintering temperature is another parameter, which influences on the microhardness of the composite. In the Al/B_4C composites, with raising sintering temperature to 850 °C the microhardness increased due to decrease in porosity and enhancement of interfacial bonding. At sintering temperature of 950 °C, the microhardness of composites decreased probably due to the formation of Al_3BC and the grain coarsening with increasing sintering temperature.

4. Conclusions

Microwave sintering was used successfully to produce Al/B_4C metal matrix composite. At sintering temperature of 850 °C and more, the interfacial reaction between Al and B_4C causes

the formation of Al_3BC. 1 wt% cobalt could improve the microstructural and mechanical properties of the composites. In all composites, the microhardness and compressive strength values increased with increasing the weight fraction of B_4C. Microwave heating could produce Al/B_4C composites along with saving energy and time.

Conflicts of interest

The authors declare no conflicts of interest.

REFERENCES

[1] Ibrahim IA, Mohamed FA, Lavernia EJ. Particulate reinforced metal matrix composites – a review. J Mater Sci 1991;26:1137–56.

[2] Clyne TW, Withers PJ. An introduction to metal matrix composites. Cambridge: Cambridge University Press; 1993.

[3] Lloyd DJ. Particle reinforced aluminum and magnesium matrix composites. Int Mater Rev 1994;1:1–39.

[4] Salvador MD, Amigo V, Martınez N, Busquets D. Microstructure and mechanical behaviour of Al–Si–Mg alloys reinforced with Ti–Al intermetallics. J Mater Process Technol 2003;143–144:605–11.

[5] Busquets-Mataix D, Martınez N, Salvador MD, Amigo V. Mechanical and tribological properties of Ti_3Al reinforced aluminum matrix composites. Adv Compos Lett 2004;13:17–25.

[6] Thévenot F. Boron carbide - A comprehensive review. J Eur Ceram Soc 1990;6(4):205–25.

[7] Chen M, McCauley JW, Hemker KJ. Shock-induced localized amorphization in boron carbide. Science 2003;299:1563–6.

[8] Pyzik JA, Aksay IA. Multipurpose boron carbide–aluminum composite and its manufacture via the control of the microstructure. U.S. Patent No.: 4,702,770 (1987).

[9] Pyzik JA, Nilsson RT. B_4C–Al cermets and method for making same. U.S. Patent No.: 5,039,633 (1991).

[10] Halverson DC, Landingham RL. Infiltration processing of boron carbide, boron, and boride-reactive metal cermets. U.S. Patent No.: 4,718,941 (1988).

[11] Gazza GE. Ceramic-metal systems by infiltration. U.S. Patent No.: 3,864,154 (1975).

[12] Pyzik JA, Deshmukh UV, Dummead SD, Allen TL, Rossow HE. Light weight boron carbide–aluminum cermets. U.S. Patent No.: 5,521,016 (1996).

[13] Halverson DC, Pyzik JA, Aksay IA, Snowden WE. Processing of boron carbide-aluminum composites. J Am Ceram Soc 1989;72(5):775–80.

[14] Viala JC, Bouix J, Gonzalez G, Esnouf C. Chemical reactivity of aluminium with boron carbide. J Mater Sci 1997;32:4559–73.

[15] Sutton WR. Microwave processing of ceramic materials. Am Ceram Soc Bull 1989;68(2):376–86.

[16] Clark DE, Folz D, Folgar C, Mahmoud M. Microwave solutions for ceramic engineering. Westerville, OH, USA: ACerS Pub.; 2005. p. 22–7.

[17] Janney MA, Calhoun CL, Kimrey HD. Microwave sintering of solid oxide fuel cell materials. I. Zirconia–8 mol% yttria. J Am Ceram Soc 1992;75(2):41–6.

[18] Goldstein A, Travitzky N, Singurindi A, Kravchik M. Direct MW sintering of yttria stabilized zirconia at 2.45 GHz. J Eur Ceram Soc 1999;19(12):2067–74.

[19] Roy R, Agrawal D, Cheng J, Mathis M. Microwave processing: triumph of applications-driven science in WC-composites and ferroic titanates. Ceram Trans 1997;80:3–26.

[20] Binner JGP, Vaidhyanathan B. Microwave sintering of ceramics: what does it offer? Key Eng Mater 2004;264–268:725–30.

[21] Goldstein A, Ruginets R, Geffen Y. Microwave sintering of amorphous silica powders. J Mater Sci Lett 1997;16(4):310–2.

[22] Ruginets R, Fischer R. Microwave sintering of boron carbide composites. Bull Am Ceram Soc 1995;74(1):56–8.

[23] Viala JC, Bouix J. J Mater Sci 1997;32:4559.

[24] Oh S-Y, Cornie JA, Russel KC. Wetting of ceramic particulates with liquid aluminium alloys. Part I. Experimental techniques. Metall Trans A 1989;20:527–32.

[25] Oh S-Y, Cornie JA, Russel KC. Wetting of ceramic particulates with liquid aluminium alloys. Part II. Study of wettability. Metall Trans A 1989;20:533–41.

[26] Marchi CS, Kouzeli M, Miserez A, Moser B, Rossell A, Weber L, et al. In: Sang J, Yin R, editors. Proceedings of the advanced materials conference (session 3), ICETS-2000 ISAM. Beijing: New World Press; 2000. p. 74–82.

[27] Taya M, Arsenault RJ. Scr Metall 1987;21:349–54.

[28] Tham LM, Gupta M, Cheng L. Effect of limited matrix reinforcement interfacial reaction on enhancing the mechanical properties of aluminium–silicon carbide composite. Acta Mater 2001;49:3243–53.

[29] Clark DE, Folz DC, West JK. Processing materials with microwave energy. Mater Sci Eng A 2000;287:153–8.

[30] Clark DE, Sutton WH. Microwave processing of materials. Annu Rev Mater Sci 1996;26:299–331.