Microstructure and Properties of Al-B₄C Functionally Graded Materials Produced by Powder Metallurgy Method

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Abstract In the present work, three different types-layered Al-B₄C functionally graded materials were fabricated via powder metallurgy. The Al-B₄C powder mixtures were cold pressed in two different pressures (400 and 500MPa) and sintered at 600°C for 3h under argon atmosphere. After investigating the pressure and sintering process of the mixed powder with different compositions, the density, microstructure, elemental distribution and hardness variation of the produced FGMs were evaluated. A detailed characterization of the microstructure of the FGMs was carried out using scanning electron microscopy and energy dispersive X-ray analysis. The results revealed that the mechanical properties of the Al-B₄C FGMs system strongly depend on constitutional variation, and display various graded distributions as well. The microstructure of FGMs so produced exhibits a gradual change of hardness from 165BHN in aluminum to 250BHN in B₄C region.

Keywords Functionally Graded Materials, Powder Metallurgy, Metal Matrix Composite, Boron Carbide

1. Introduction

As modern technology develops, more demands on material properties are raised in advanced engineering applications. On some occasions, service conditions require materials performance to vary with locations [1]. Functionally graded materials (FGMs) provide a novel solution to this requirement and realize multifunctions that cannot be achieved by monolithic or homogeneous materials [2]. By definition, FGMs are used to produce components featuring engineered gradual transitions in microstructure and/or composition. They accommodate a gradient transition of the properties of different materials from one end to another, where the property mismatch (mechanical, thermal, or electrical) can be reduced to minimum [1, 3]. The introduction to FGM has been expected to have a strong impact on the design and development of new components and structures with better performance [4]. So far, FGMs

have been extensively applied in many areas, including structural [5], thermal [6], electrical [7], biomedical [8] and military [9]. Bhattacharyya et al. [6] studied that Synthesis and characterization of Al/SiC and Ni/Al₂O₃ functionally graded materials. They reported that Al/SiCFGMs fabricated by powder metallurgy route, exhibit microstructures with a homogeneous distribution of fragmented and clustered SiC particles.

The fabrication process is one of the most important fields in FGM research. A large variety of production methods have been developed for the processing of FGM, such as, powder metallurgy, sheet lamination, laser cladding, and chemical vapor deposition [5]. It is noted that the powder metallurgy method is one of the most commonly employed techniques due to its wide range control on composition and microstructure and shape forming capability. In general, the forming methods used include slip casting [10, 11], tape casting [12,13], centrifugal casting [14], thixotropic casting [15], electromagnetic separation [16, 17], electrophoretic deposition [18], combustion [19], and die pressing followed by cold isostatic pressing [20]. The sintering techniques employed include pressureless sintering [21], hot pressing [22], and hot isostatic pressing [23].

Composites of boron carbide and aluminum offer the excellent combination of high hardness and toughness in a lightweight structure. There are several methods which have been used for manufacturing of metal matrix composites [24-35]. Rodriguez-Castro et al. [24] investigated that Microstructure and mechanical behavior of functionally graded Al A359/SiCp composite. They revealed that SiC reinforcement fracture and de-cohesion, with particle fracture increasing as particle concentration increases. The wettability of B₄C particles by the molten aluminum alloys is generally poor below 1100 °C making their incorporation difficult for casting techniques. On the other hand, powder metallurgy is the most commonly employed techniques due to its good control capability on the composition and microstructure of the FGMs and the reliable shape forming capability. Chu et al. [8] examined that hydroxyapatite-Ti functionally graded biomaterial fabricated by powder metallurgy method. They has been successfully produced the

hydroxyapatite-Ti (HA-Ti) functionally graded biomaterial (FGM) by the powder metallurgical process. They stated that FGM microstructure varies gradually with the diversification of chemical composition, which eliminates the macroscopic interfaces of HA-Ti bonding. A large number of works has been reported on aluminum-based FGM with alumina, silicon carbide, titanium carbide, and titanium diboride as reinforcements. Synthesis of Al-B₄C monolithic composite is also reported in the literature [32, 34, 36, 37]. B₄C is an excellent reinforcement for Al-based composites having high hardness close to diamond, excellent thermal stability, and remarkable inertness.

The most of the studies on FGMs have devoted Al₂O₃ and SiC reinforced FGMs [38-40] and research into B₄C reinforced FGMs is relatively limited [41]. Fabrication of aluminium-based FGMs with B₄C reinforcement was made by centrifugal casting and studied the effect of centrifugal force on B₄C gradient. However, Al-B₄C FGMs produced by powder metallurgy method has not been reported in the literature. Therefore, a specific attempt was made in this work to fabricate Al-B₄C FGMs by powder metallurgy. In this work, the desired B₄C gradient was obtained.

The purpose of this work is to develop fundamental constitutional control technique of FGM by powder metallurgy, expecting to optimize FGM fabrication process and further improve its performance. The microstructure and the composition of the fabricated FGM by powder metallurgy were studied and the green and sintered density and the Brinell hardness of each layer in the FGM were measured. The relationship between microstructure, composition and some other physical and mechanical properties were discussed.

2. Experimental

B₄C and aluminium powders were used as raw materials to fabricate the FGMs. The as-atomized Al powders (99.9% purity) with an average particle size of 113µm (Gündoğdu Exotherm Company, Turkey) were used as the matrix materials, and B₄C powders with an average particle size of 102µm (Wacker Ceramics Kempten GmbH, Germany) were used as the reinforcements. Al powders, with different weight percentages of B₄C powders (0, 5, and 10 wt. %, was blended in mixer for 0.5h. The FGM was designed to comprise three different type layers. In addition to pure Al, Al-5wt. % B₄C, and Al -10wt. % B₄C single-type-layers, the intermediate structure consisted of two different type layers with different ratios of B₄C and Al, namely, Al-5wt. % B₄C + Al, Al-10wt. % B₄C+Al two-type-layers, Al-10wt. % B₄C + Al-5wt. % B₄C+Al three-type-layer, respectively, as shown in Fig. 1.

The mixed powders were filled and uniaxially cold pressed in a steel die at 400 and 500MPa. Then the green formed FGMs compacts were subjected to sintering at 600°C for 3h under argon atmosphere. The experimental densities of the FGMs samples were measured Archimedes's method. The theoretical densities were calculated using the mixture rule according to the weight fraction of the B₄C powders. Then, green density, sintered density and densification percent were calculated considering densities of 2,70g/cm³ and 2,52 g/cm³ for Al and B₄C, respectively. The microstructure and elemental distribution on the cross-section of FGM were observed by means of optical microscope and Zeiss Evo LS10 scanning electron microscope, respectively. In addition, image analysis and computerised scanning X-ray tomography were employed to quantitatively determine the B₄C distribution. The hardness of the FGM samples was measured using the Brinell hardness method under a load of 31.25kg for a dwell time of 20s.

Figure 1. The designed layer types. (a) Single-type-layer, (b) Two-type-layer, (c) Three-type-layer

Figure 2. Microstructure of a cross-section for Al-B₄C FGM; (a) back scattered mode of three-type-layer (b) back scattered mode of two-type-layer

Figure 3. X-ray attenuation profiles showing effect of powder metallurgy technique on B₄C distribution in Al-B₄C FGMs.

3. Results and Discussion

Fig. 2 shows the general view and different interfaces details of microstructures of a cross section for this FGM. There are no indications of cracking with in individual layers or of decohesion at interfaces in any of the specimens. In the microstructures, dark gray is the B₄C ceramic phases and light gray is Al phase. With the variation of composition, the microstructure of interlayer changes in a stepwise manner from Al side to Al-10wt. %B₄C side. From the microstructures in the details for the different interfaces of the FGM, one can be seen that both the metal and composites are constant in microstructures. Thus there is no macroscopic interface in the FGM. This good continuity of microstructure can eliminate the disadvantage of traditional macroscopic interface in metal/composites joint and reflects the design ideal of FGM.

No cracking can be found in the FGMs in this study (Fig. 2), indicating that the FGMs have good strength, good thermal stability and thermal shock due to the better metal-ceramic bond, continues in microstructure at the interfaces of FGMs, as reported in previous studies [39, 43]. The results of the topography study are presented in Fig.3. The X-ray attenuation decreased with B₄C weight fraction thus enabling the B₄C distribution to be monitored. Clearly, the X-ray attenuation is reduced step by step as the B₄C content decreases in the FGM specimen whereas the X-ray attenuation trace for the specimen shows a much smoother and more B₄C distribution. Small peaks on the attenuation curves could be correlated with surface defects or discontinuities as seen in the SEM micrographs.

The results of microstructural characterization studies, conducted on the polished FGMs for all the specimens, revealed the following features: (i) presence features, (ii)

clustering of B_4C and (iii) good integrity at the interfaces of aluminium matrix-reinforcing particulates. In this study, there is no evidence of other phases either at the particle-matrix interface or in the matrix suggesting that no reaction occurs between B_4C and Al during manufacturing (Fig. 4a and 4b). The interface chemistry of the B_4C -Al couple has been addressed in several studies [44–48]. Boron carbide and aluminum react at temperatures greater than 700 °C producing AlB_2 and aluminum boron carbide, Al_3BC , which has only recently been identified [45, 49]. Other more complicated phases develop at temperatures greater than 900 °C; however, such temperatures can easily be avoided during processing since the melting point of aluminum is 660 °C. The decomposition mechanisms of boron carbide in liquid aluminum between 700°C and 900°C have been summarised by Viala et al. [45]. The formation of these phases degrades the particles and results in composites with poor mechanical properties [44, 45, 49]. These phases may form during prolong working at high temperatures as well as during processing if the production temperature is high. It is therefore important to develop low temperature production methods for B_4C -aluminum alloy composites [50].

The experimental density measurements in Table 1 showed that the density values decreased with an increase in the weight percentage of B_4C along the deposition direction and this contradicted with the theoretical reasoning whereby specimens from the lower B_4C end should be denser than those from the high B_4C end. This observation can be rationalized in term of porosity levels, which increased with

the weight percentage of B_4C . The densification of the brittle B_4C powders is due to powder sliding followed by powder fragmentation. The literature reveals that the use of additives, such as liquid and solid lubricants, substantially increase fluidity and therefore densification [51, 52]; however, the ceramics were cold pressed without additive showing unbonded interparticle contacts. Although a decrease was also seen in the experimental densities, the values are lower than those of the theoretical ones (Table 1). The density measurements showed that FGMs contain some porosity, and the amount of porosity in the FGMs increased with the increasing weight fraction of the B_4C powders (Table 1). In general, the B_4C powder clustering increased with B_4C content as did the associated porosity. The increase in porosity levels with an increase in B_4C powders can be attributed to the combined effects of the increase in the clustering tendency with increasing weight percentages of B_4C powders and the increasing friction between Al- B_4C powder mixtures leading to a lower densification extent. At the highest pressing pressure (500MPa), the green and sintered densities of FGMs increase (Table 1) while the porosity values increase. However, It is seen from these values that the green and sintered densities of FGMs decreased with increasing B_4C powder content in Table 1 whereas the porosity values increasing B_4C powder content in Table 1. This means that, the higher the weight fraction of B_4C powders added to the aluminium matrix, the higher porosity of the functionally graded composites, similar to the observations of some previous studies [38, 53].

Figure 4. Interface between B_4C and Al. (a) X-ray attenuation profiles showing a B_4C particle in the Al matrix; (b) EDX spectrum of B_4C particle in the vicinity of interface.

Table 1. Physical and mechanical properties of FGMs

Coded samples	Theoretical density (g/cm ³)	Green density (g/cm ³)	Sintered density (g/cm ³)	Brinell Hardness (BHN)
Al _{single} (400)	2.7000	2.524	2.600	170
Al _{single} (500)	2.7000	2.600	2.610	189
Al-5 _{single} (400)	2.6900	2.508	2.513	201
Al-5 _{single} (500)	2.6900	2.587	2.596	213
Al-10 _{single} (400)	2.6820	2.490	2.498	226
Al-10 _{single} (500)	2.6820	2.567	2.583	250
(Al+Al-5) _{two} (400)	2.6950	2.516	2.523	170-201
(Al+Al-5) _{two} (500)	2.6950	2.593	2.602	195-226
(Al+Al-10) _{two} (400)	2.6910	2.506	2.514	175-213
(Al+Al-10) _{two} (500)	2.6910	2.587	2.597	195-241
(Al+Al-5+Al-10) _{three} (400)	2.6906	2.500	2.510	166-195-220
(Al+Al-5+Al-10) _{three} (500)	2.6906	2.587	2.590	185-220-250

The influence of the varying microstructure on the hardness of the FGM sample is obvious, as shown in Table 1. As the content of B₄C increases from 0 wt. % to 10 wt. %, the hardness changes and the hardness increases substantially. This is mainly caused by the gradual variation of the microstructure in the functionally graded composites. As compared to the Al matrix, the hardness of the FGMs was found to be greater, and the addition of B₄C particles increases the hardness of the Al [54, 55]. An increase in the hardness of the Al-B₄C functionally graded composite with increasing weight fractions of B₄C from the matrix to B₄C graded regions, for both the FGM fabricated at 400 and 500MPa, respectively (Table 1), as observed in the previous investigations [40, 56]. The effect of the amount of B₄C (wt.%) on the hardness can occur in at least two ways: first, by influencing the load carrying between the matrix particles and second, by influencing inhibit dislocation movement.

4. Conclusions

Three different-type-layered Al-B₄C FGMs has been successfully fabricated by powder metallurgy method. Microstructural examination showed that the B₄C distributions were generally homogeneous in the Al matrix while in the higher B₄C content layers some large B₄C clustering were also present. No cracking in the FGMs showed that the composites and the FGM fabricated in this study have good graded composition and good strength due to the better metal-ceramic bond and continuous in microstructure at the interface of FGM. The density of FGMs was decreased with increasing B₄C weight fraction and decreasing pressing pressure, but the porosity and hardness of the FGMs were increased with increasing particle content.

However, the porosities of FGMs were decreased with increasing pressing pressure whereas the hardness values were increased with increasing pressing pressure SEM observations indicated that there was no interface reaction between B₄C powders and Al matrix during production.

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