

# Microwave Sintering of Aluminum-ZrB<sub>2</sub> Composite: Focusing on Microstructure and Mechanical Properties

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Microwave assisted rapid sintering was carried out to fabricate aluminum-ZrB<sub>2</sub> metal matrix composites containing 1 wt% cobalt and 10, 15 and 20 wt% ZrB<sub>2</sub> as the reinforcements at temperatures of 600, 700 and 800°C. The results showed that the highest values of density and bending strength (97% of theoretical density and 240 MPa, respectively) were obtained in a composite containing 15 wt% ZrB<sub>2</sub> and 1 wt% Co sintered at 600°C. Furthermore, the optimum values of compressive strength and hardness (350 MPa and 63 Vickers, respectively) were measured for a composite containing 20 wt% ZrB<sub>2</sub> sintered at 600°C. At all sintering temperatures, XRD analysis revealed ZrB<sub>2</sub> and aluminum as the only crystalline phases. Microstructure investigations demonstrated a homogeneous distribution of ZrB<sub>2</sub> particles in the aluminum matrix.

**Keywords:** Aluminum, Zirconium diboride, Microwave sintering, Metal matrix composites

## 1. Introduction

Aluminum (Al) metal matrix composites (AMC) have many applications in the aerospace and car industries and military applications due to their high specific modulus, high strength to weight ratio and toughness<sup>1-3</sup>. Generally, the casting, extrusion and forging routes, and powder metallurgy (PM) process have been used to produce AMC<sup>4,5</sup>. Compared with traditional methods, PM is believed to be a more appropriate process to fabricate near net shape products<sup>6</sup>. In addition, the interaction between matrix and reinforcements can be avoided because of lower processing temperatures usually associated with PM methods<sup>7</sup>. Compared with casting process, PM provides a more uniform distribution of reinforcement materials within the matrix<sup>8</sup>. However, the conventional PM processing of composites often needs long sintering time, which results in grain coarsening and poor performance of mechanical properties<sup>9</sup>.

Microwave sintering presents distinct advantages such as high energy efficiency, enhanced densification and also smaller grain size due to the faster heating rate at lower sintering temperatures<sup>10</sup>. Microwave sintering provides a uniform volumetric heating as well as smaller pores in the sintered compacts, which improves the microstructure and mechanical properties<sup>11-13</sup>.

SiC and Al<sub>2</sub>O<sub>3</sub> particles are the most commonly used materials for reinforcement of Al alloys. Furthermore, there has been growing interest of using ZrB<sub>2</sub> as reinforcement due to its high melting point, high electrical and thermal conductivity and chemical inertness<sup>14-16</sup>. In the recent years, there has been much research into various aspects of Al-ZrB<sub>2</sub> composites produced mainly by casting route and PM method<sup>17,18</sup>. The limited research has been conducted on the effect of second metallic phase as a binder or coherent phase between aluminum and reinforcement<sup>19</sup>. Some researchers investigated the effect of Cu additive as

a coating of reinforcement particles on different aluminum matrix composites<sup>20</sup>. However, to the best of the author's knowledge, there have been limited reports of the effect of Co additive on the final properties of Al-ZrB<sub>2</sub> composites<sup>21,22</sup>.

In the present work, an attempt has been made to investigate the effect of microwave sintering parameters and variable amounts of ZrB<sub>2</sub> on microstructure and mechanical properties of PM green compacts. Additionally, the properties of Al-ZrB<sub>2</sub> composite with and without 1 wt% Co have been investigated.

## 2. Experimental procedures

ZrB<sub>2</sub> (99.5% purity, 10 μm average particle size) and aluminum (MERCK Art. no. 1056 aluminum powder, <70 μm particle size, 99% purity) 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 required amounts of Al, ZrB<sub>2</sub> and Co powders were taken in four separated batches presented in Table 1.

The batches were mixed with a high energy turbula mixer-Spex (Mixer mill – 8000D) for 10 minutes in ethanol media. After mixing process, each batch was dried at 70°C to remove ethanol from the mixture. Then, the bar-shaped samples with the dimension of 25×5×5 mm were compacted at 250 MPa by an uniaxial press. The compressive strength samples with a diameter of d=1 cm and height of h=0.8 cm were prepared at 140 MPa by a hydraulic press. The samples were sintered in a graphite bed using a single-mode microwave furnace (900 W and 2.45 GHz), at 600°, 700° and 800°C without soaking time. An optical pyrometer (Model: RAYR312MSCL2G) was used as the temperature monitoring device. The bulk density of sintered samples was measured using the Archimedes' Principle. The three point bending strength and compressive strength tests were conducted with a Santam-STm 20 instrument using at least 5

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**Table 1:** Different batch compositions.

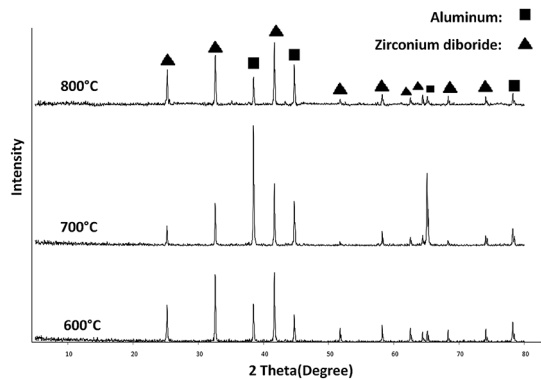
Batch	Al (wt %)	ZrB <sub>2</sub> (wt %)	Co (wt %)
A	90	10	0
B	89	10	1
C	84	15	1
D	79	20	1

and 3 samples, respectively. X-ray diffraction (XRD, Philips X' Pert System) analyses were performed to identify the phases present in the Al-ZrB<sub>2</sub> composites. Vickers microhardness values of the sintered samples were determined using a MKV-h21 Microhardness Tester under a load of 1kgf for 15 s. At least ten successive indentations were performed for each sample. Microstructural investigations of sintered samples were carried out using a SEM (Stereoscan 360, Leica Cambridge).

3. Results and discussion

3.1. XRD analysis

Figure 1 shows the XRD patterns of D series samples (table 1) sintered at 600, 700 and 800°C. The identified phases of Al and ZrB<sub>2</sub> are similar for all composites and no additional phases are found.



**Figure 1:** XRD patterns of composites containing 20 wt% ZrB<sub>2</sub> sintered at (a) 600 °C, 700°C and 800 °C.

3.2. Densification

Since in the precision of XRD analysis only Al and ZrB<sub>2</sub> phases were detected (Figure 1), therefore, the relative density of composites were measured by the rule of mixtures and the results are shown in Table 2. The variation of the measured densities was limited to ± 1 % of the average value. As Table 2 further reveals, the relative densities of B, C and D composites are nearly the same compared to A composite which is much less. It seems that the presence of Co additive in B, C and D composites caused an increase of density. The maximum relative density of 96.9±0.3 % was calculated for C composite sintered at 600 °C.

As Table 2 represents, there is no significant change in the measured density of Al-ZrB<sub>2</sub> composites containing Co with

the increase of sintering temperature. This is because of two diverse effects; the relative density increases as a result of high atomic diffusion and decreases due to the agglomeration of ZrB<sub>2</sub> particles during sintering process. Because of high melting point of ZrB<sub>2</sub>, it can be expected that ZrB<sub>2</sub> particles form aggregates with a random distribution in Al matrix, preventing complete densification<sup>14</sup>. Another possibility is the formation of alumina layer with a high melting point around Al particles and low tendency of these layers to make bonds with ZrB<sub>2</sub> particles at sintering temperatures. It seems that the formation of some porosity and cracks within the composites matrix during cooling due to the thermal expansion mismatch between Al and ZrB<sub>2</sub> particles can be prevented by adding Co particles.

3.3. Microstructural analysis

The backscattered electron images of Al-ZrB<sub>2</sub> composites are shown in figure 2. The dark and light regions correspond to Al and ZrB<sub>2</sub> particles, respectively. SEM images in figure 2-a further reveal a uniform distribution of ZrB<sub>2</sub> particles in Al matrix in C sample containing cobalt additive sintered at 600°C. It also has a less porous structure (no cracks were observed), exhibiting good bonding between matrix and reinforcement particles.

Figure 2-b shows the scanning electron micrographs of A sample (without cobalt additive) sintered at 600°C. Microcracks can be seen around ZrB<sub>2</sub> particles attributed to the thermal expansion mismatch between Al and ZrB<sub>2</sub> particles.

SEM micrographs of D composite sintered at 600°C (Figure 2-c) show that the agglomeration of ZrB<sub>2</sub> particles increased due to increasing amount of reinforcement particles from 15 to 20 wt%.

Figure 3 shows the SEM micrographs and EDS analysis of Al-15wt%ZrB<sub>2</sub>-1wt%Co composite sintered at 600°C. EDS1 point (gray region) is rich in element Al, while EDS2 point (light region) is rich in element Zr, implying the existence of ZrB<sub>2</sub> particles as the reinforcement in composite.

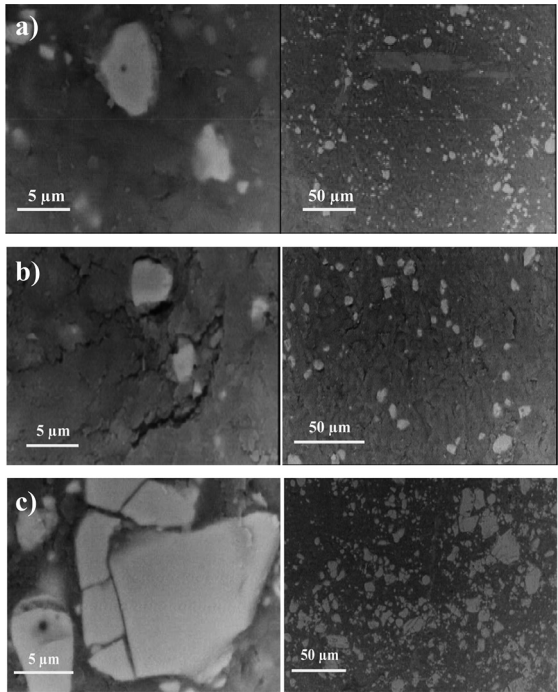
Figure 4 shows the fracture surface of C composite sintered at 600°C. It can be observed that there are many fine dimples around 5-6 μm in diameter accompanied by ZrB<sub>2</sub> particles on the surface of this sample. The rupture mechanism of C composite is the ductile fracture of ZrB<sub>2</sub> and its expanding in the substrate.

3.4. Bending strength

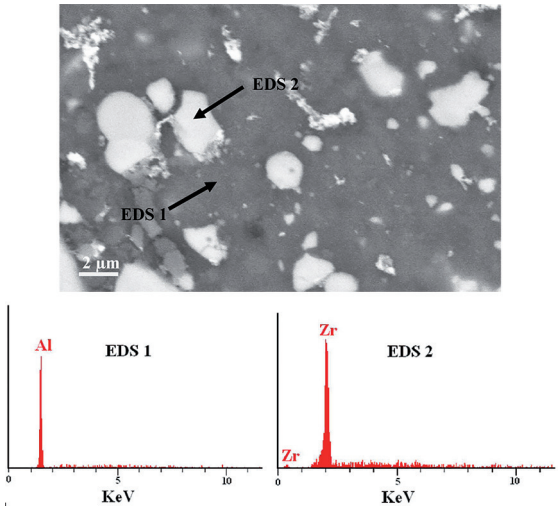
In the case of sintering apart from bonding and porous-free microstructure, the important factor affecting strength of sintered composite is the amount of ZrB<sub>2</sub> particles presented in the matrix. On the other hand, increasing amounts of reinforcements can be caused the agglomeration of particles, destroying the mechanical properties of composites. The maximum bending strength value was measured for C composite containing 15 wt % ZrB<sub>2</sub> and 1wt% Co sintered at 600°C. The presence of ZrB<sub>2</sub> particles in Al matrix could impede the movement of dislocations since these particles are stronger than Al particles. As it was considered in previous section (Figure 2-c), the agglomeration of reinforcement particles in Al-20 wt% ZrB<sub>2</sub>-1wt% Co composite could form some porosity between agglomerates, acting as crack propagation centers. The degree of strengthening produced

**Table 2:** Relative density of composites sintered at different temperatures.

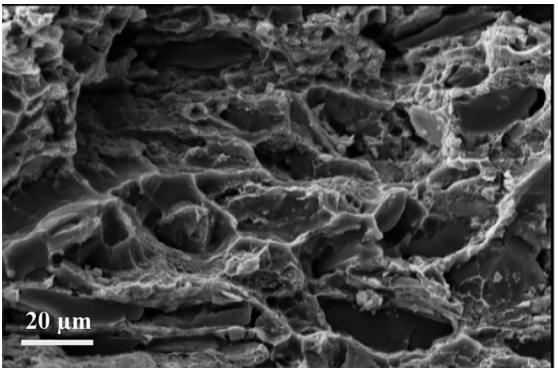
Al-20wt%ZrB <sub>2</sub> -1wt%Co			Al-15wt%ZrB <sub>2</sub> -1wt%Co			Al-10wt%ZrB <sub>2</sub> -1wt%Co			Al-10wt%ZrB <sub>2</sub>			sample
3.06			2.96			2.88			2.85			
800			700			600			800			Theoretical density (gr/cm <sup>3</sup> )
700			600			800			700			
2.92 ± 0.01			2.79 ± 0.03			2.73 ± 0.04			2.43 ± 0.03			Sintering temperature (°C)
2.91 ± 0.02			2.83 ± 0.01			2.74 ± 0.01			2.41 ± 0.04			
2.90 ± 0.03			2.87 ± 0.02			2.75 ± 0.01			2.35 ± 0.03			Measured density (gr/cm <sup>3</sup> )
95.4 ± 0.3			94.2 ± 1			94.79 ± 1.3			85.26 ± 1			
95. ± 0.6			95.6 ± 0.3			95.13 ± 0.3			84.56 ± 1.3			Relative density (%)
94.7 ± 1			96.9 ± 0.6			95.48 ± 0.1			82.45 ± 1			



**Figure 2:** SEM micrographs of: a) Al-15 wt% ZrB<sub>2</sub>-1wt% Co, b) Al-10wt% ZrB<sub>2</sub> and c) SEM micrographs of Al-20 wt% ZrB<sub>2</sub>-1 wt% Co composite sintered at 600°C.

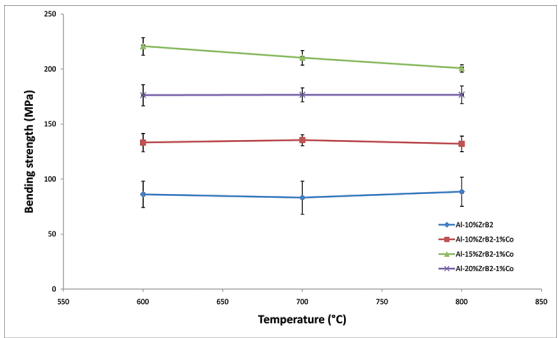


**Figure 3:** Backscattered images, and EDS spectra taken from surface of Al-15wt% ZrB<sub>2</sub>-1wt%Co sintered at 600°C.



**Figure 4:** The fracture surface of Al-15wt% ZrB<sub>2</sub>-1wt%Co sintered at 600°C.

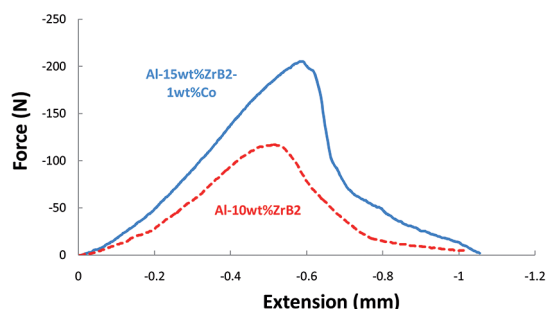
also depends on the size of particles, their distance apart and the bonding of particles. The dislocations cannot pass through the reinforcement particles, if they have higher mechanical properties than the matrix particles. Figure 5 demonstrates the change of bending strength of composites with increasing sintering temperature. The nearly high and equal values of strength of composites sintered at different temperatures can be attributed to the existence of Al and ZrB<sub>2</sub> as the only crystalline phases and it seems that changes in sintering temperature have little effect on the changes in bending strength of composites.



**Figure 5:** Bending strength vs sintering temperature.

C composites obtained higher bending strength values (Figure 5) than all other specimens because of higher density and excellent bonding of Al and ZrB<sub>2</sub> reinforcement particles in comparison with A composites (Figure 2-b). It seems that Co additive has a positive effect on the bonding between ZrB<sub>2</sub> and Al particles.

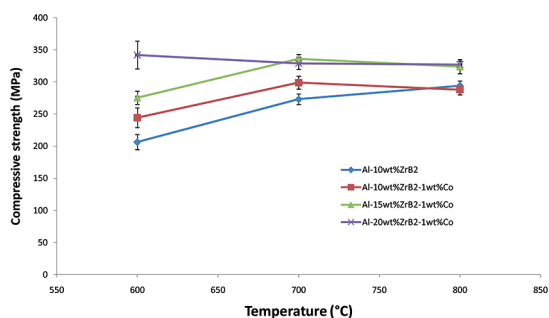
Figure 6 shows the force-extension variation for C and A composite sintered at 600°C. The surface area under force-extension curves introduces the fracture toughness of specimens. As comparison, it is clear that C composite exhibited a higher toughness rather than A composite.



**Figure 6:** The force-extension curves for Al-15wt%ZrB<sub>2</sub>-1wt%Co and Al-15wt%ZrB<sub>2</sub> samples sintered at 600°C.

### 3.5. Compressive strength

Because of a lower density, composites containing 10 wt% ZrB<sub>2</sub> without Co additive (Figure 7) had the lowest compressive strength values. With increase in reinforcement particles, the movement of dislocations is somehow halted, leading to increase in the compressive strength.

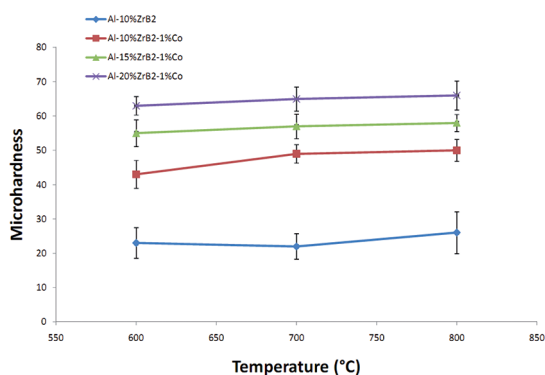


**Figure 7:** Compressive strength vs. sintering temperature.

The maximum compressive strength was measured for composite containing 20 wt% ZrB<sub>2</sub> (D series) at all sintering temperatures. It seems that the proper bonding between ZrB<sub>2</sub> reinforcement and Al matrix exists in D rather than A composites. Furthermore, the presence of maximum amounts of reinforcement in D composite leads to best compressive properties among all series of composites.

### 3.6. Microhardness

Generally, the high hardness indicates a good bonding between particle and matrix. The results presented in Figure 8 reveal that the maximum microhardness value corresponds to composite containing 20 wt% ZrB<sub>2</sub> due to the high hardness value of ZrB<sub>2</sub> particles and also the good bonding between the reinforcement particles and matrix, as shown in Figure 2. The hardness increases due to reduced plastic deformation by the reinforced particles.



**Figure 8:** Microhardness vs. sintering temperature.

Figure 8 further reveals that the microhardness and bending strength changes (Figure 5) follow the same trend. The hardness of composites sintered at different temperatures is almost similar for each composite series (A, B, C and D). The results obtained by Z. Asadipناه et al.<sup>23</sup> indicated that almost similar values of microhardness and less values of compressive strength obtained using nano ZrB<sub>2</sub> particles reinforced aluminum composite without Co additive prepared by microwave processing than those values obtained in the present work. This shows the role of Co addition in the improvement of microstructure and mechanical properties of Al-ZrB<sub>2</sub> composites.

## 4. Conclusions

The powder metallurgy combined with microwave sintering was successfully applied to aluminum-ZrB<sub>2</sub> composites and a high density was obtained at a low temperature of 600°C without soaking. Co additives had significant effect on crack and porosity degradation in microstructure of these composites. The optimum bending strength (240MPa) and microhardness (56 vickers) values were obtained for Al-15wt% ZrB<sub>2</sub>-1wt% Co composite sintered at 600°C. Aluminum and ZrB<sub>2</sub> were found to be the only crystalline phases with increasing sintering temperature.

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