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Effects of Sintering Time on Density and Microstructure of Al-15Si-2,5Cu-0,5Mg / B_4C Composites in Microwave Sintering Process

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Abstract. In this study, B_4C (5, and 10 wt.%) particle-reinforced Al-15Si-2,5Cu-0,5Mg (Ecka Alumix 231®) aluminum matrix composites were produced through cold pressing technique. Samples were sintered by microwave sintering technique. Sintering was carried out at 555 °C for 15, 30, 45, 60 minutes holding times and without holding time. Density measurement, microstructure analysis and hardness tests of the produced samples were carried out. Densities of samples before and after sintering were measured by Arshiment method according to ASTM B962-08. Optical microscope and scanning electron microscope (SEM) were used in microstructure studies. Macrohardness measurements were performed in terms of Brinell hardness by use of 31,5 kgf load and 2.5 mm ball diameter. The green density of the samples decreased with increasing B_4C wt.%. Compared to the green density values, density values of all samples had decreasing with the sintering process. The highest density values in sintered samples were obtained in 60 minutes sintering time. Al-rich solid solution, primary Si and Cu and Mg-rich phases in the microstructure were determined through SEM and EDS analysis.

Keywords: Powder metallurgy, Microwave sintering, Density, Microstructures, Hardness
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Introduction

Composite materials have become a requirement for modern production technology. It is because composite materials have improved physical and mechanical properties. The main application areas of composite materials are the aerospace, military and automobile industries. Different techniques are used for the production of metal matrix composites. The most common of these techniques is powder metallurgy (P/M) [1]. Cold pressing and sintering, one of these P/M techniques, were developed at the University of Sevilla [2]. Nowadays, different sintering techniques are used. One of these techniques is microwave sintering. Microwave heating is due to the material's ability to absorb high frequency electromagnetic energy (microwaves) and convert it into heat [3]. The microwave heating

occurs from the core and generates heat inside the sample [4]. Microwaves; refers to electromagnetic waves at a frequency of 300 MHz to 300 GHz, wavelength between 1 m 1 mm [3,4].

The use of microwave energy for processing materials has the advantages in reduced processing times and energy savings [5]. Microwave sintering has been recognized as a time-saving and cost-effective process as compared to the conventional powder metallurgy processes for sintering composite materials [6].

In this study; hypereutectic Al-Si (Al-15Si-2,5Cu-0,5Mg (Alumix 231®)) alloy has been chosen as the matrix material. B_4C was used as a reinforced. Al/B_4C composites were produced by cold pressing and microwave sintering techniques. The effect of sintering time on density, microstructure and hardness was investigated.

Experimental procedure

B_4C reinforced Al matrix composites were produced through cold pressing and microwave sintering techniques. Hypereutectic Alumix 231® powder, produced by ECKA Granulate Velden GmbH, with a sintered density of 2.67 g/cm³, was used as matrix material. The chemical composition of Alumix 231® powders are given in Table 1. The Alumix 231® powders with an average size of about ~75 µm at D50, ~22 µm at D10, and ~190 µm at D90 was produced by inert gas atomization. The reinforcement particle B_4C has a density of 2.52 g/cm³ and the average powder particle size (D_{50}) is 10 µm.

Table 1. The chemical composition (wt.%) of Alumix-231 alloy

Composition (wt%)					
Alumix 231®	Al	Si	Cu	Mg	Lubr.
Nominal	Bal.	14–16	2,4–2,8	0,5–0,8	1,5

Alumix 231 / (wt. 5-10%) B_4C powders were mixed in a triaxial mixer. The mixture powders were pressed by applying 620 MPa pressure by cold pressing technique. Samples were sintered by microwave sintering technique. Sintering was carried out at 555 °C for 15, 30, 45, 60 minutes and without holding time. The samples were subjected to the elimination of the lubricant at 400 °C for 20 minutes. Heating rates to lubricant elimination (400 °C) and sintering temperature (555 °C) were set at 5 °C / min and 10 °C/min, respectively.

The effects of sintering time on density, microstructure and hardness were investigated. After sintering, the density of the samples was measured according to the Archimedes principle (ASTM B962-08). The densities are reported as % of the theoretical densities (%TD). The theoretical density is calculated from the rule of mixtures using material composition. Optical microscope and scanning electron microscope (SEM) were used in microstructure examinations. Macrohardness measurements were performed using the Brinell hardness test with 31,5 kgf load and 2,5 mm ball diameter.

Results and discussion

Table 2 illustrated the sample codes and % TD of the produced samples. The MW sample code shown in Table 2 represents microwave sintering. The numbers 0-5-10 next to the MW code indicate the wt.% B_4C particle ratio.

Table 2. The density results

Sample code	B_4C Particle Rate (%)	Density (% TD)					
		Green	Sintering Time				
			Without holding time	15 min.	30 min.	45 min.	60 min.
MW-0	0	92,127	88,185	86,324	85,301	85,058	89,410
MW-5	5	91,793	87,804	87,247	87,035	87,233	88,982
MW-10	10	91,197	88,023	89,089	89,436	89,062	90,222

The green density of the samples decreased with increasing wt.% B_4C. Green density decreases due to increasing porosity with increasing particle ratio (Table 2). It is because, with increasing particle ratio, increasing porosity at the particle/matrix interface causes a decrease in density.

Compared to the green density values, density values of all samples had decreasing with the sintering process. The highest density values in sintered samples were obtained in 60 minutes sintering time. (Table 2). However, these changes observed in sample densities were limited to small values. These decreases in densities of sintered samples are attributed to the liquid phase formation. In studies carried out with Alumix 231®, liquid phase formation is mentioned at sintering temperatures above 500 °C. [7-10]. It was determined that the liquid phase formed due to sintering temperature had an effect on density. In this study, it is thought that the small density changes observed at different sintering times at 555 °C are caused by the accumulation of the liquid

phase in a region and the sudden dimensional change during cooling.

The SEM micrograph of MW-0 (without holding time) in Fig. 1 and EDS analysis results in Table 3 are given. Different regions were identified in the microstructure of the Alumix 231® alloy: elemental Al matrix (point 3), master alloy (point 2), primary Si (point 1) particles, Cu (point 4) and Mg-rich secondary phases. Alumix 231® is a premixed alloy powder that is composed of a mixture of elemental aluminum and master alloy (Al-Si-Cu-Mg) powders.

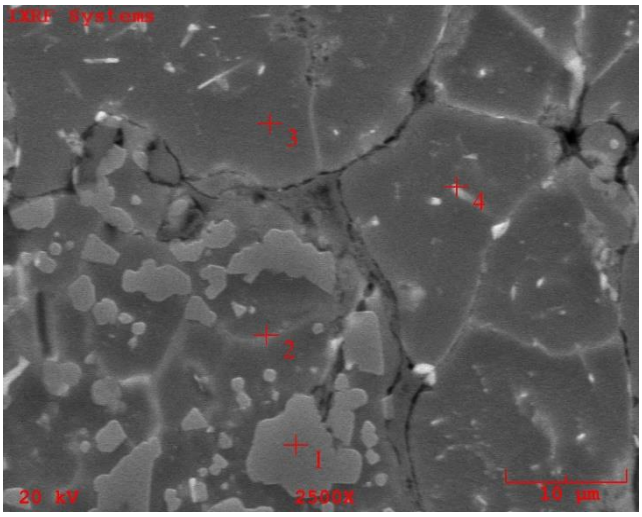


***Figure 1**. The SEM micrograph of the MW-0 (Without holding time)*

Table 3. EDS analysis (wt.%) corresponding to the point locations as shown in Fig.1

Sample	Point	Al	Si	Cu	Mg
MW-0 (Without holding time)	Point 1	4,47	94	1,37	0,15
	Point 2	76,28	21,27	2,2	0,28
	Point 3	96,13	0,017	3,4	0,45
	Point 4	95,1	0,35	4,18	0,36

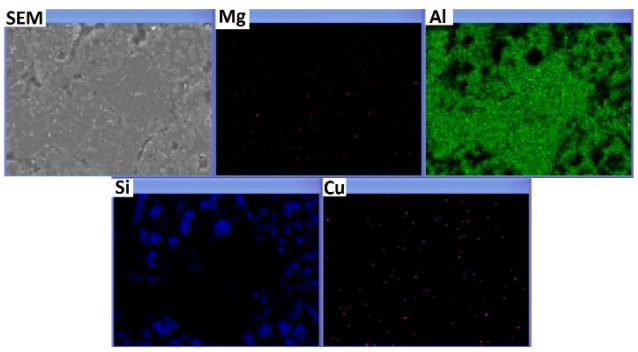


***Figure 2**. Distribution of the alloying elements inside the MW-0 (Without holding time) sample mapped by EDS analysis*

Fig. 2 presents the microstructure map of the MW-0 (Without holding time) sample. The map shows that the primary Si particles emerged in the original master alloy grains. The Cu- and Mg-rich second phases had a homogenous distribution in the master alloy and elemental aluminum regions.

The SEM micrograph of the MW-5 (Without holding time) sample is shown in Fig. 3. Master alloy, pure aluminum regions and B_4C particles are summarized in micrographs. The primary Si particles (Fig.1, point 1) have grown from the master alloy powder.

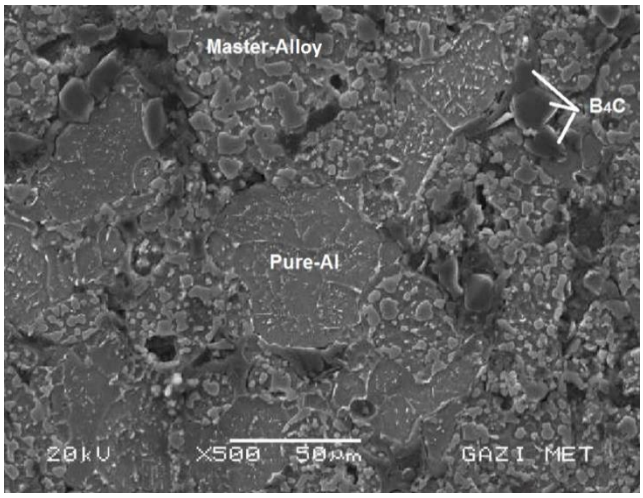


***Figure 3**. The SEM micrograph of the MW-5 (Without holding time)*

Optical micrographs of Alumix 231 samples sintered at 555 °C (60 min holding time and without holding time) are given in Fig. 4. When the micrographs are examined, in parallel with the density measurement results; it was observed that the pores shrink and decrease with 60 minutes sintering. This change in pores size and amount with increasing sintering time can be explained by the grain join and growth that occurs with the effective transport mechanisms (evaporation and condensation by atomic diffusion) in sintering. However, there is still an intense porosity and regional enlargements of pores in the microstructure. In addition, the bonding problems between the grains are seen in Fig.1, Fig.4 and Fig.5. There was not a good diffusion event and sufficient cohesion between the grains could not be achieved. Therefore, a long sintering time of 60 minutes did not show the expected improvements in microstructure and density compared to without holding time sample. As stated in the literature, the biggest advantage offered by microwave sintering technique is that it exhibits good density and microstructure properties without holding and/or in very short holding times [11-17].

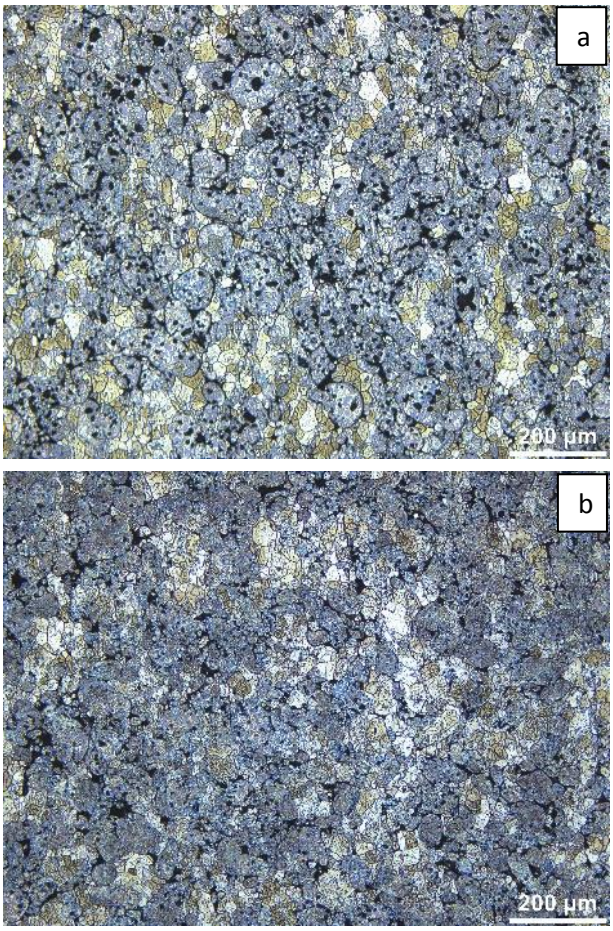


Figure 4. *Optical micrographs of MW-0 samples sintered at different times, a) Without holding time; b)* Holding time: 60 min

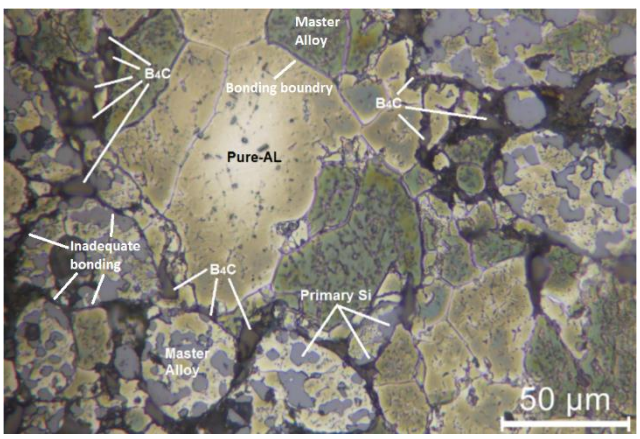


Figure 5. *Optic micrographs of MW-10 (Without holding time)*

The effect of the sintering time on the hardness of the samples is given in Fig.6. The highest hardness value was determined in the sample coded MW-0, which was without holding time at the sintering temperature at 555 °C. In general, a decrease in hardness was observed with increasing sintering time in samples. Grain coarsening with increasing sintering time and inadequate bonding between grains may be the reason for this decrease in hardness (Fig.1, Fig.4 and Fig.5). With increasing B_4C reinforcement ratio, a decrease in hardness was observed in samples. There was no significant increase in hardness in the samples MW-0 and MW-10 at 60 minutes sintering time. Although there is no significant change in density, the decrease in hardness caused by the increasing B_4C addition can be explained by the pushing of B_4C particles into the pores and the grain coarsening during sintering (Fig. 6).

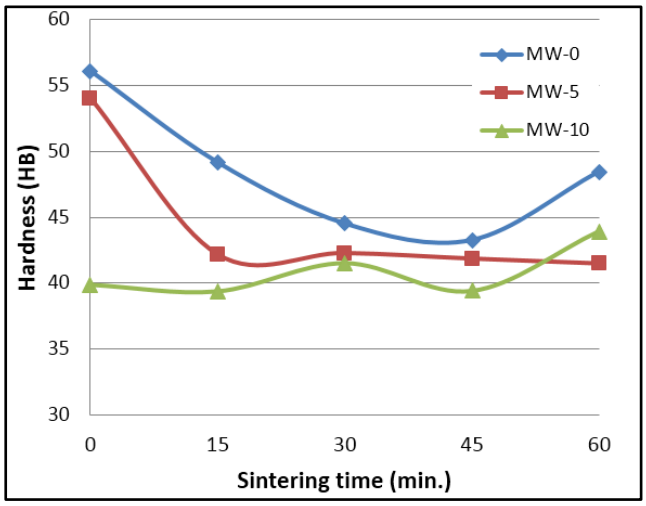


Figure 6. *Hardness change graph according to different sintering times and wt.% particle amount*

Conclusion

The main conclusions for this study are summarized below:

- B_4C (wt.% 5 and 10) particle-reinforced Ecka Alumix 231® aluminum composites were produced through the cold press/microwave sintering technique. Sintering process was carried out at without holding time and four different times.
- The green density of the samples decreased with increasing wt.% B_4C.
- Compared to the green density values, density values of all samples had decreasing with the sintering process. The highest density values in sintered samples were obtained in 60 minutes sintering time. However, these changes observed in sample densities were limited to small values.
- Different regions were identified in the microstructure of the Alumix 231® alloy: elemental Al matrix, master alloy, primary Si particles, Cu and Mg-rich secondary phases. The primary Si particles emerged in the original master alloy grains. The Cu- and Mg-rich second phases had a homogenous distribution in the master alloy and elemental aluminum regions.

- The highest hardness value was determined on the sample that did not contain B_4C and without holding time at the sintering temperature. Sample hardness was observed to decrease with increasing B_4C particle ratio.
- It was determined that B_4C particles clustered in the pores.
- The samples were produced without holding time at 555 °C gave optimum values in terms of density and hardness.

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