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Effect of Heating Method on Microstructure and Mechanical Properties of Zircon Reinforced Aluminum Composites

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The mechanical properties and microstructure of aluminum matrix composites containing of zircon (10, 15 and 20 wt.%) as a reinforcement particles and cobalt additive (1, 1.5 and 2 wt.%) were investigated. The aluminum matrix composites were prepared by powder metallurgy technique and sintered in both conventional and microwave furnaces at temperatures higher than the melting point of aluminum. The results revealed that the density and mechanical strength of aluminum were increased by introducing zircon and cobalt particles. The maximum strength was obtained by adding 10 wt.% zircon sintered in microwave furnace at 950°C. The phase analysis and scanning electron microscopy of sintered composites were examined and found some interesting data about intermetallic compounds.

Keywords: *aluminum matrix composite, zircon particles, cobalt, microwave sintering*

1. Introduction

Particle reinforced aluminum matrix composites have received considerable attention in aerospace and automobile industries because of high specific modulus, good wear resistance and high specific strength[1-3]. Aluminum matrix composites have been usually produced via powder metallurgy and casting, while achieving a homogenous distribution of reinforcement in the base alloy is more accessible with powder metallurgy in comparison with casting technique[4-6]. Reinforcement segregation, clustering, high localized residual porosity and poor interfacial bonding are problems found by other forming techniques[7]. The other composite production methods, such as spray forming, are expensive which limit their applications[8]. Producing net-shape components and minimizing machining process are the other advantages of using powder metallurgy. Four advantageous, known as low density, high conductivity, high toughness and low price have been made aluminum as one of the best materials in some metal matrix composites in comparison with other light metals such as magnesium[9,10]. In spite of all these advantages, the low strength of aluminum leads to limitation of its applications[11].

Ceramic particles (e.g. Al_2O_3, SiC, TiC and zircon) have been added to aluminum matrix in order to overcome its low tensile strength and attain better hardness and tolerate high temperatures[12-16]. Zircon is a ceramic with high chemical stability, corrosion resistance and excellent thermal shock resistance. It is cheap and available, and also is a suitable candidate for applications under elevated temperature conditions[16,17].

Ravi kumar et al.[18] reported the synthesis and properties of 15 wt.% zircon particles dispersed in pure aluminum. Their composites showed a linear increase in the abrasive wear resistance with increasing zircon content in the matrix. Aluminum alloy matrix composites reinforced with zircon and alumina particles were successfully synthesized by stir casting method and results showed that hybrid reinforced composites have better hardness and tensile strength than aluminum[19]. Ejiofor et al.[20] reported an improvement of the ultimate tensile strength, yield strength and hardness of aluminum alloy composites by adding 15 vol% zircon particles (size < 200 µm). The results of Abdizadeh et al.[16,21] experiments also showed that the mechanical properties of aluminum-zircon composites can be optimized with 5 wt% zircon reinforcement at sintering temperature of 650°C.

Several advantages such as reducing time and sintering temperature, uniform heat distribution and etc., have been reported on the application of microwave energy for processing of ceramics, metals and composites. Aluminum matrix composites reinforced with iron oxide[23] and Silicon carbide[24] were prepared using microwave energy.

In the present work, the effect of zircon particles as a reinforcement and cobalt powder as an additive on the microstructure and mechanical properties of aluminum matrix was investigated. Also, the effect of different heating methods (microwave and conventional heating) on the properties of prepared composites was studied. Samples were sintered at temperatures much above the melting point of aluminum in a short time.

2. Materials and methods

Aluminum (MERCK Art. no. 1056 aluminum powder, 250 mesh, 99% purity), zircon (99.5% purity, d50=5 µm) and cobalt (99.8% purity and 5 µm mean particle size) powders

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were used as raw materials. The required amounts of Al, zircon and Co powders were taken in separated batches with the compositions given in Table 1.

Table 1: Different compositions of batches.

Batch	Al (wt.%)	zircon (wt.%)	Co (wt.%)
A	90	10	-
B	89	10	1
C	83.5	15	1.5
D	78	20	2

The batches were prepared through mixing the powder compositions determined in Table 1 using Spex (Mixer mill–8000D). In the next step, the bar shape samples with the dimension of 25×5×5 mm^3 were compacted by uniaxial pressing at 250 MPa. Then, microwave sintering was performed at temperatures of 650°, 750°, 850° and 950°C in graphite bed for 1 minute. Also, the sintering process was done at 550° and 600°C for D composites. A pyrometer was used in order to depict the temperature. Samples were also sintered in a conventional furnace for 1 hour. In sample coding "M" and "C" symbols are referred to microwave and conventional heating, respectively. Numbers of 1, 2, 3 and 4 are also denoted the 650°, 750°, 850° and 950°C temperatures, respectively (Table 2).

Table 2: Sample coding after sintering.

Sintering condition	A	B	C	D
650˚C-microwave	AM-1	BM-1	CM-1	DM-1
750˚C-microwave	AM-2	BM-2	CM-2	DM-2
850˚C-microwave	AM-3	BM-3	CM-3	DM-3
950˚C-microwave	AM-4	BM-4	CM-4	DM-4
550˚C-microwave	-	-	-	DM-550
600˚C-microwave	-	-	-	DM-600
650˚C-conventional	-	-	-	DC-650

The bulk density of sintered samples was measured using the Archimedes' Principle. The bending strength of samples was examined with Santam-STm20. It was a three-point bending test followed ASTM C1161 and the rate of loading was 1 mm/min. X-ray diffraction (XRD, Philips X'Pert System) analysis was carried out to determine the phases formed in the composite samples. The microstructure of sintered samples was investigated using a scanning electron microscope (SEM, VEGA//SCAN).

3. Results and discussion

3.1. Density

Figure 1a shows the density of aluminum matrix composites sintered at temperatures 650˚, 750˚, 850˚ and 950˚C under

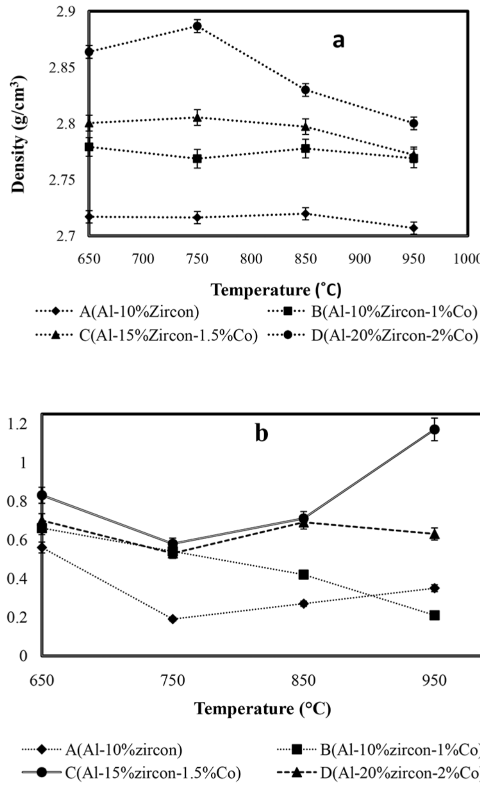

Figure 1: (a) Density and (b) porosity of Al composites sintered at temperatures 650˚, 750˚, 850˚ and 950˚C with microwave processing.

microwave irradiation. The comparison between composites A and B shows the effect of cobalt additive on density. As can be seen from figure 1a, using 1 wt.% cobalt increases the density of aluminum composites at all sintering temperatures. Furthermore, the density of composites increases with increasing the amount of zircon reinforcement from composites B to D. This is because zircon has a higher density compared to aluminum. Furthermore, by considering the error bars it seems that the density of composites (A, B and C) has been partially changed up to 850°C and decreased at the 950°C. For D composite, the density increases up to 750°C and decreases with further increase in sintering temperature up to 950°C. The density of composite can be influenced by the amount of component that will be changed at different sintering temperatures due to decomposition process and formation of new products. Consequently, it seems that the decrease of density at temperature of 750° for D and 850°C for A and C composites can be attributed to the formation and decomposition of some components in these composites. The maximum density (2.89 g/cm^3) was achieved when 20 wt.% zircon and 2 wt.% cobalt were used in aluminum matrix sintered at 750˚C. Figure 1 shows the low amount of porosity in all aluminum composites reinforced with zircon and similar results were found by Chen et al[24].

The porosity of composites was all increased by addition of zircon particles. C composites have the higher porosity than A and B composites and increasing temperature leads to the decrease of porosity in latter composites. It can be seen that the porosity decreases with the increase in sintering temperature for B composite and also with the increase in zircon content, the sintering temperature of composites increases in order to reduce porosity.

The density of aluminum based composites prepared in the present work is comparable with density reported in other works (2.83 g/cm^3 [19], 2.9 g/cm^3 [16]).

Figure 2 shows the effect of microwave and conventional heating on the density of D composites (20 wt. % zircon and 2 wt. % cobalt) sintered at different temperatures. It has been reported that[21] the initiation of the liquid phase during sintering and densification of aluminum body can be started about 570-590˚C and its amount will be increased at higher temperatures. With regard to above mentioned, the liquid phase sintering is expect more in composites prepared in our work and leads to the successful sintering of aluminum alloys[1]. The aluminum composites sintered by microwave and conventional furnaces showed comparable values in density. However, at low sintering temperatures, microwave process was found to yield higher densities rather than conventional process. DM-550, DM-600 and DM-650 composites have remarkable higher densities than DC-650 composite.

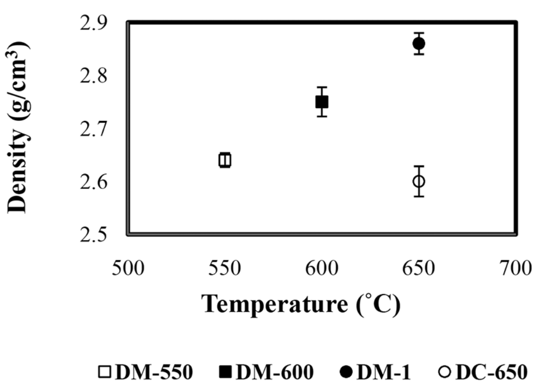

Figure 2: Density of Al composites sintered at temperatures 550˚, 600˚, and 650˚C using microwave heating and conventional furnace.

3.2. XRD

Figures 3 and 4 show the XRD patterns of aluminum based composites sintered in microwave (composites DM) and conventional furnaces (composite DC-650). In order to depict zircon peaks, DM composites that contain the high percentage of zircon were chosen. XRD patterns of DM-550 (figure 3) and DC-650 (figure 4) show aluminum and zircon as the main phases. Zircon was always identified by XRD analysis of aluminum-zircon composites sintered through conventional furnace in another study[21]. XRD of DM-series composites in the present work identified alumina, quartz and an intermetallic phase of aluminum-zirconium-silicon (m) as well as aluminum and zircon phases. Referring to

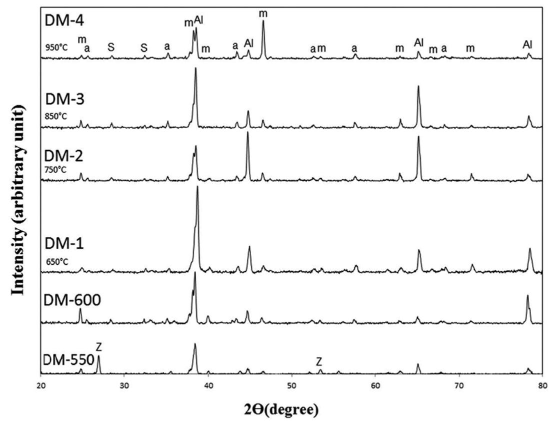

Figure 3: XRD patterns of aluminum composites containing 20 wt.% zircon and 2 wt.% cobalt sintered in microwave at different temperatures. Al: aluminum, a: alumina, z: zircon, s: quartz and m: aluminum-zirconium-silicon intermetallic phase.

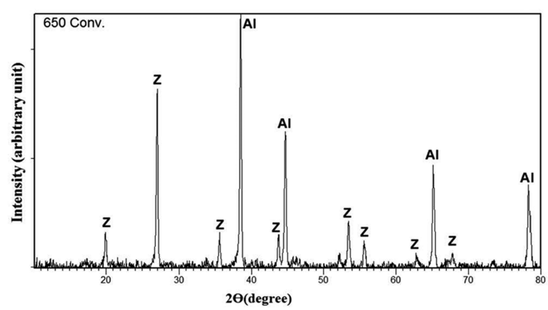

Figure 4: XRD of DC-650 composite sintered conventionally (Al: aluminum and Z: zircon).

JCPDS Card Number 00-014-0625, intermetallic phase of aluminum-zirconium-silicon was denoted as $Al_{0.45}Si_{0.217}Zr_{0.33}$ phase. The formation of a intermetallic phase in aluminum matrix composites was reported by Banerji et al[27]. They reported the presence of a reaction zone including some concentrations of magnesium and silicon at the interface of zircon and matrix particles. The decomposition temperature of zircon is higher than 1600°C [28], however the intrinsic impurities of natural zircon can lower its decomposition temperature and play an important role on the mechanisms of its reactions[29]. The small amounts of impurities effect on the decomposition temperature of zircon and its kinetics and also the formation of low melting liquid phases resulting in the favored dissociation of solid silica immediately after releasing from zircon structure[30]. The formation of a liquid phase reaction sintering at the boundaries of the reaction grains in aluminum alloy matrix composites at 615°C in vacuum for 20 min was reported, leading to the formation of different aluminum compounds or intermetallic phases[20]. These intermetallic phases have been known to be the responsible of good wear properties, high abrasion resistance and good heat resistance.

XRD results revealed that the dissociation of zircon in aluminum matrix occurs during microwave heating even at a low heating temperature of 600°C without soaking (Fig. 3), while it was not observed during conventional heating even at higher temperature of 650°C after 2h of holding (Fig.4). This dissociation occurs because the increase in the local temperature around the zircon particles, leading to the formation of intermetallic phase. It is believed that in the thermal decomposition reaction process at solid–liquid interface of molten aluminum and zircon particles, Al matrix reacts with oxygen to give Al_2O_3 and also reacts with zirconium (Zr) and silicon (Si) to form an intermetallic compound of Al-Zr-Si.

3.3. Microstructure

SEM micrographs of aluminum composites and elemental analysis of A, B and C regions of figure 5b are shown in figures 5 and 6, respectively. Micrographs show uniform distribution of reinforcement phases in Al metal matrix. SEM images show a clear phase contrast of different phases. High difference between the density of aluminum and two other phases can cause the adequate contrast to *distinguish between three* distinct *phases.* It is expected that the well dispersion of the phase particles in the aluminum matrix may lead to an improvement of mechanical properties of final composites. It is observed that the increase in sintering temperature from 550˚C to 750˚C and 950˚C results in the increase of the grain size of the products (Figs.5(a) to 5(c)).

Elemental analysis of A, B and C regions were done and given in figure 6 and table 3. High amounts of aluminum and zirconium in point A indicate the formation of an intermetallic phase. It is supposed that zircon particles are

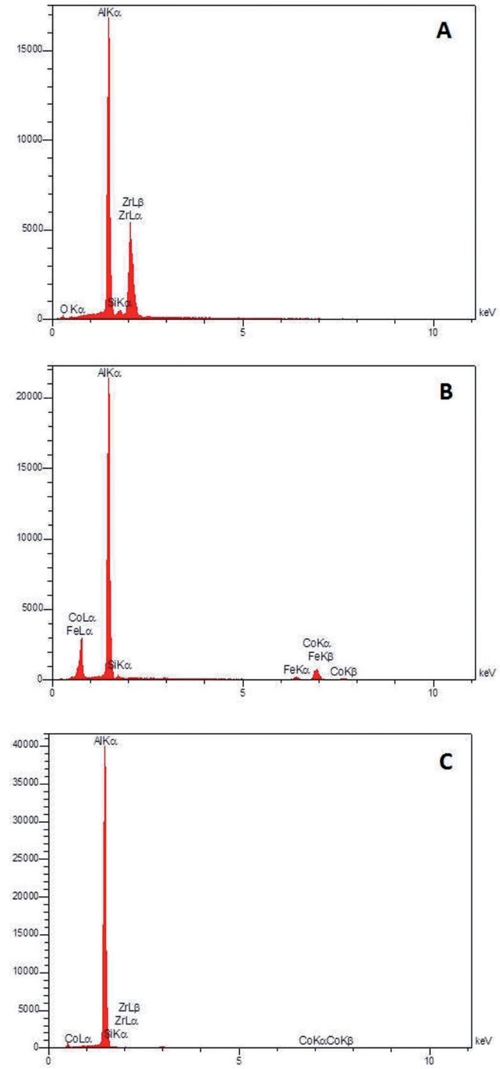

Figure 6: EDX analysis of points A, B and C in figure 5b

Table 3: Elemental compositions of points A, B and C of figure 5

Batch	A	B	C
O	1.68	-	-
Al	51.51	71.49	98.02
Si	1.08	1.28	1.08
Zr	45.73	-	0.66
Fe	-	3.94	-
Co	-	23.29	0.24

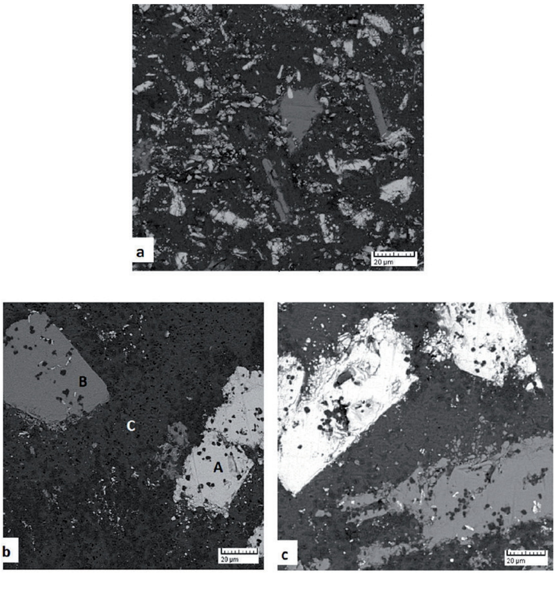

Figure 5: SEM photograph of aluminum-zircon composite (a) DM-550, (b) DM-2, (c) DM-4

completely decomposed while Al particles diffuse through them. Furthermore, B point indicates the presence of Al, Si and Co elements that form an intermetallic phase with another composition. The formation of intermetallic phases can be attributed to the high sintering temperature and fast sintering rate in microwave heating. In microwave process the rapid heating rates through skin effect via Eddy currents and suceptor lead to enhance diffusion kinetics as a result of local high temperatures[22]. C point indicates the aluminum matrix.

3.4. Mechanical strength

The mechanical strength of aluminum based composites is presented in table 4 with tolerance limits about ±10MPa. As can be seen, Zircon reinforcement and cobalt additive cause the increase of the mechanical strength of composites from series A to D. The strength values obtained by composites having the reinforcement combination of 10 wt.% Zircon and 1 wt% cobalt sintered at 750° and 950°C were 263.9±10 and 266.1±10 MPa, respectively. It can be seen that the presence of zircon particles in the aluminum matrix results in the increase in strength. The observed increase in strength of these composites can be attributed to the intrinsic strength and hardness of zircon. On the other hand, the liquid phase sintering can lead the successful densification and mechanical properties of aluminum alloys[1].

Table 4: Mechanical strength of aluminum based composites.

Temperature (°C)	650	750	850	950
A (Al-10%Zircon)	213.1	210.8	231.5	225.1
B (Al-10%Zircon-1%Co)	221.7	263.9	249.7	266.1
C (Al-15%Zircon-1.5%Co)	206.6	250.2	255.4	216.1
D (Al-20%Zircon-2%Co)	232.1	259.9	223.9	239.2

The formation of intermetallic compounds in these composites may be the other possible reason for increasing the mechanical strength. It has been reported that a chemical reaction between reinforcement phase and aluminum, and wetting of the reinforcement by aluminum and formation of an intermetallic phase are the major issues encountered in the processing of the aluminum based composites[32].

The relationship between the mechanical strength of composites with sintering method is presented in table 5. A remarkable increase of mechanical strength is observed with increasing sintering temperature. As table 5 reveals, even at lower heating temperatures, composites sintered in microwave obtained higher mechanical strength compared to those sintered in conventional furnace.

4. Conclusions

Aluminum based composites reinforced with zircon particles and cobalt additive were prepared successfully using microwave and conventional heating. The aluminum-Zircon composites were fabricated through powder metallurgy technique at temperatures higher than the melting point of aluminum as novel technique. The results showed that the addition of zircon particles and cobalt additive improve the densification and mechanical properties of aluminum matrix. Microwave heating led to the decomposition of zircon reinforcements and formation of intermetallic compounds. Density and mechanical strength of composites sintered in microwave (2.86 g/cm^3 and 266 MPa, respectively) were remarkably higher than those of composites sintered in conventional furnace (2.60 g/cm^3 and 156 MPa, respectively). The microstructure of composites showed a good dispersion of the second phase particles containing an intermetallic phase in aluminum matrix. The best mechanical results were achieved for composites sintered in microwave at 750°C, containing 10 wt.% zircon and 1 wt.% cobalt.

Table 5: Mechanical strength of aluminum composite sintered by two different methods

Sintering temperature	Composite	strength(MPa)
Microwave sintering-550°C	DM-550	219±7
Microwave sintering-600°C	DM-600	217±3
Microwave sintering-650°C	DM-1	232±9
Conventional sintering-650°C	DC-650	156±7

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