

Modification of As-Cast Al-Mg/B₄C Composite by Addition of Zr

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Abstract

Zirconium was used in Al-Mg/B₄C composite to improve compocasting efficiency by increasing particle incorporation. Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) results revealed that by addition of zirconium a reaction layer containing Zr, Al, B and C is formed on the interface of B₄C-matrix. X-ray diffraction (XRD) analysis of extracted particles unveiled that the ZrB₂ phase is the main constituent of this layer. Formation of ZrB₂ is an exothermic reaction which can rise temperature locally around particles and agglomerates. Rising temperature around agglomerates in conjunction with turbulent flow of melt facilitates agglomerates wetting and dissolving into molten aluminum. As the result, final product contains more uniformly distributed B₄C particles. Besides enhancing compocasting efficiency, addition of Zr and formation of reaction layer by improving particle matrix bonding quality, led to increase in ultimate tensile strength and elongation of the composite around 8% and 30%, respectively. SEM observations of the fracture surfaces confirmed that a proper bonding presents at the interface of particles and matrix in presence of Zr.

Keywords: Metal-matrix composites; Liquid-solid reactions; Mechanical properties; Scanning electron microscopy; X-ray diffraction.

1. Introduction

The particle reinforced aluminum matrix composites (AMC) are widely used in transportation-related industries because of their desirable properties including high value of specific strength, improved elastic behavior, and high wear resistance [1-3]. Generally, the fabrication process of AMCs can be divided to casting and powder metallurgy routes [1]. Among various casting methods, stir casting has been known as an inexpensive process that is applicable for a wide range of materials [1, 2].

Boron carbide (B_4C) is known as an attractive reinforcement for AMCs due to its lower density compared with aluminum, high hardness, and low thermal expansion in comparison with other common reinforcements such as SiC and Al_2O_3 [4]. However, poor wettability of B_4C with molten aluminum at temperatures below 1100 °C is a drawback of liquid state fabrication methods [5, 6]. Several researches have been conducted so far to improve the interaction and incorporation of B_4C in molten Al, including preheating particles to form boron oxide, surface preparation techniques, coating with Ti, and applying Ti-containing flux [6-11]. As a result of the interfacial reaction between the reinforcement and matrix, an interface forms, which plays an important role in mechanical properties of composites [12-14], and hence, lots of research has been conducted on this subject [2,3,12,13].

Zirconium has been widely used in aluminum alloys as a grain refiner in casting or microstructure stabilizer at high temperature [15]. It has been also proved that according to Eq. 1, Zr can react with boron carbide and produces Zirconium Carbide and Zirconium Boride [16], even when Zr is in the Al matrix as an alloying element [17].



Based on the facts that this reaction is exothermic [18] and also interaction in metal-ceramic system is rarely desirable [19], it seems that addition of Zr can improve the particle incorporation and mechanical properties. However, there is not an available comprehensive

study on the influence of Zr on the B₄C incorporation in the Al matrix. Therefore, the aim of current work is to investigate the effect of Zr as an alloying element on the boron carbide incorporation into molten aluminum, particle-matrix interface, and mechanical properties of the compocast product. The stirring time and the casting temperature were considered as influential processing parameters and their impacts on the microstructure were investigated. Moreover, based on the Response Surface Methodology (RMS) [20], the mentioned parameters were optimized to reach the highest B₄C yield, which has gained a great technological importance.

2. Experimental details

2.1. Stir-casting

B₄C particles with the average particle size of 25 and 80 μm were used as the reinforcement material in commercially pure aluminum (99.81 %) matrix. In order to examine the effect of Zr on the wettability, three different ratios of (wt.% Zr)/(wt.% B₄C), namely 0, 0.05 and 0.1, were used by adding Al-15Zr master alloy into the molten aluminum. Particulate reinforced AMCs were produced by using 0.5 kg aluminum and adequate amount of master alloy which were melted and were held for 40 minutes in an alumina crucible. B₄C particles were added into the melt via vortex formed from string with 570 rpm. Stirring was applied for 5, 12.5 and 20 minutes at the stirring temperatures of 750, 825 and 900 °C under Argon atmosphere. Just before pouring into a cold cylindrical steel mold, 3 wt. % Mg was added to the melt, and there is not enough time for take parting Mg in reactions, or in other words, there is not so much probability for an excessive reaction of Mg with B₄C [7,21].

2.2. Design of experiment for investigating particle incorporation

Finding the effects of Zr and the processing related parameters such as stirring time and temperature, calls upon large numbers of experiments. In such experiments, one parameter is varying while the other factors are constant. A major demerit of this method is ignoring the interactions between parameters. Response surface methodology, which is based on the combination of mathematical and statistical techniques for designing experiments, provides a realistic optimization process by considering the effect of variable interactions. Reduction of the number of the experiments is the main merit of the RSM. In the current research, the central composite design (CCD) based on the Design-Expert® software (Ver. 8.0.1) [22] was utilized. CCD is an efficient way to provide a sufficient amount of information to test the fitness of a model. Considering three parameters (temperature, stirring time and amount of Zr) in three levels, the number of required experiments was obtained as 11 which are presented in Table 1.

Table 1: Casting conditions and obtained results for different samples.

Sample	Time (min)	Temperature (°C)	$D \times W_{Zr} / W_{B4C}$	B ₄ C incorporation %
1	5	750	0	16.41
2	12.5	750	1.25	26.12
3	20	750	2.5	32.87
4	5	825	1.25	17.13
5	12.5	825	0	8.06
6	12.5	825	1.25	18.08
7	12.5	825	2.5	20.34
8	20	825	1.25	24.00
9	5	900	2.5	5.29
10	12.5	900	1.25	7.00
11	20	900	0	2.00

To find the effect of parameters on particle incorporation (B₄C yield%), the image analysis technique was used. Some corresponding micrographs of the composites, from which the data were obtained, are presented in Fig. 1 as representative examples. For microstructural

observations and energy dispersive X-ray spectroscopy (EDS), a Thermal-Field-Emission Scanning Electron Microscope (Jeol 7001f -0.1-30 kV) was employed.

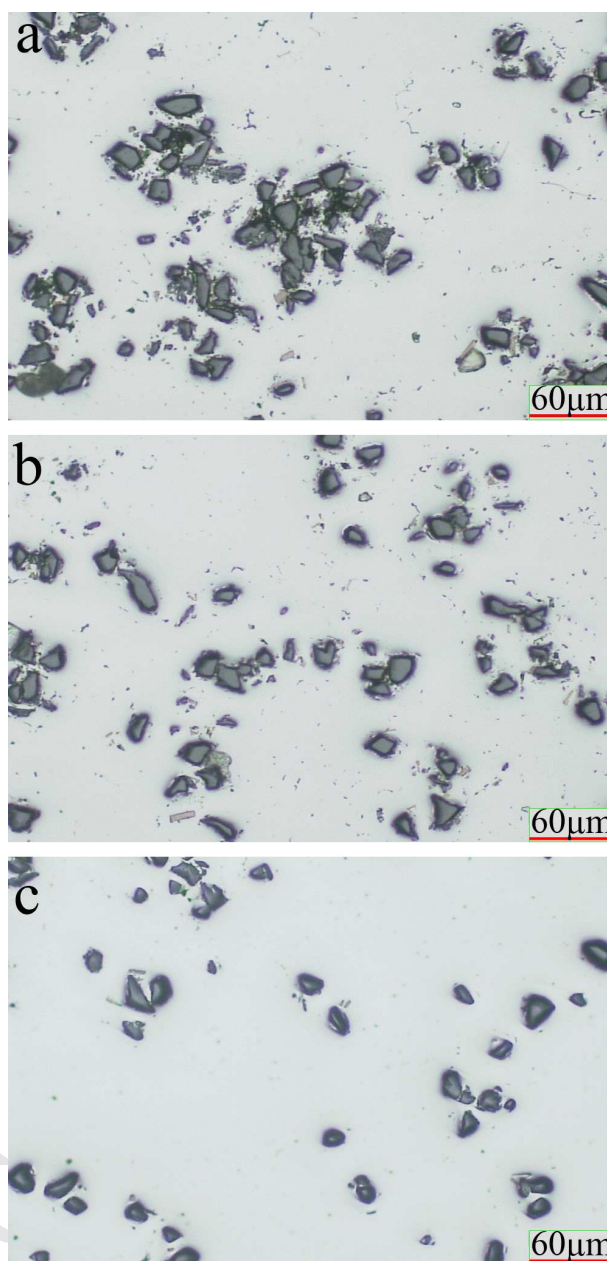


Fig. 1: Optical micrographs of Al/B₄C composites for (a) Sample 1, (b) Sample 6 and (c) Sample 10.

After resolving the aluminum matrix in the NaOH solution, the X-Ray Diffraction (XRD) analysis was used to identify the phases present in the particles. The XRD experiment was performed by a Rigaku Ultima IV using Cu-K α radiation.

It was proved by Kennedt [5] that the weight fraction of alloying elements (here Zr) required for interfacial reaction depends on the weight fraction of the reinforcement (e.g. W_{Zr}/W_{B_4C}) and the particle diameter (D).

The critical mass of alloying element needed for wetting particles (m_{Zr}) required to form a reaction layer is related to the surface of all of the particles (S), i.e. $m_{Zr} = cS$ [6]. The total surface of particles can be calculated by using Eq. 2;

$$S = n\pi D^2, \quad (2)$$

where n and D are the number and average diameter of particles, respectively. In the case of spherical particles, n can be evaluated by dividing total volume of particles to volume of one particle (Eq. 3).

$$n = \frac{m_{B_4C}/\rho_{B_4C}}{\pi D^3/6} \quad (3)$$

By combining abovementioned equations, the ratio of masses required for wetting can be expressed as:

$$D \frac{m_{Zr}}{m_{B_4C}} = \frac{6c}{\rho_{B_4C}} \quad (4)$$

Therefore, Dm_{Zr}/m_{B_4C} or DW_{Zr}/W_{B_4C} are independent of the particle size. In the other words, the effects of Zr, B_4C , and D were considered in a unique parameter presented as DW_{Zr}/W_{B_4C} .

Finally, the response (output of the model) was the B_4C yield percentage, i.e. fraction of particles incorporation into the molten Al.

2.3. Investigating the impact of interface on mechanical properties

Chemical interactions between the matrix and the reinforcement determine the interface adhesion, modify the characteristics of the composite components and affect its mechanical characteristics [13]. In other words, the interface quality has an influential impact on the load

transfer from matrix to particles. Therefore, the higher the strength of the interface, the higher the load that the composite can withstand. Moreover, it is expected that increasing the quality of interface results in enhanced tensile elongation. Indeed, a weak interface is a suitable site for formation of cavities because of low strength of this area in comparison with either of particle or matrix [14]. In order to investigate the impact of Zr-containing layer on the mechanical properties, two composites, one with addition of Zr ($COMP_{Zr}$) and another without Zr (COMP), were fabricated. The casting condition was optimized to produce a Al-3 wt.% Mg - 10 vol% B_4C composite (exact volume fraction of B_4C was measured from microstructure) and then the product was remelted to fabricate $COMP_{Zr}$ and COMP by adding Zr. The details are presented in Table 2. The corresponding microstructures of $COMP_{Zr}$ and COMP are shown in Fig. 2, which confirms that the particle size and volume fraction are the same for both composites. After homogenization at 580 °C for 2 hours and hot extrusion at 470 °C with the ratio of 14:1, samples were prepared via machining according to ASTM E8 standard (sub size). Universal tensile test machine (SANTAM 2000) with crosshead speed of 1 mm/min was used for performing three tensile tests for each condition.

Table 2: details of fabrication process for $COMP_{Zr}$ and COMP.

	Stirring temperature	Stirring time	Stirring temperature after remelting for adding Zr	Stirring time after remelting for adding Zr	Amount of added Zr
$COMP_{Zr}$	720 °C	10 min	800 °C	20 min	0.2 wt% ($D \times W_{Zr} / W_{B_4C} = 1.6$)
COMP			800 °C	20 min	0

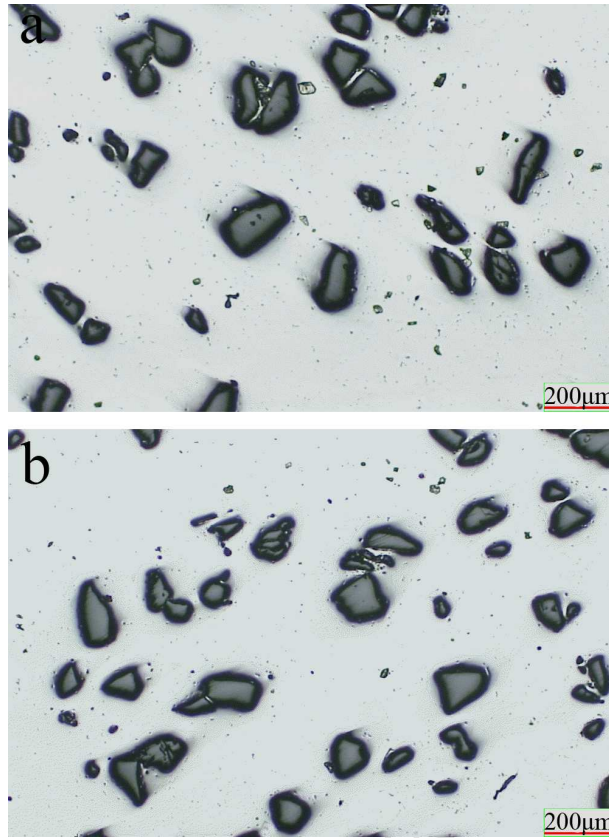


Fig. 2: Optical micrographs of (a) COMP_{Zr} and (b) COMP.

3. Results and discussion

3.1. Particle incorporation

To evaluate the effect of Zr, casting temperature, and stirring time variables on the percentage of B₄C yield in the product, experiments were performed on the basis of central composite design and the results are presented in Table 1. After the analyzing of the experimental results, Eq. 5 was obtained from Design-Expert software based on the response of different input variables:

$$B_4C \text{ Incorporation} = (-51.696 + 0.1526T + 0.565DW_{Zr} / W_{B_4C} - 1.0039 \times 10^{-4}T^2)^2 \quad (5)$$

While this model is based on regression analysis, some useful remarks can be deduced from it. For example, the model reveals that, in comparison with temperature and amount of Zr, the stirring time has negligible effect on the B₄C yield. Interestingly, the model also predicts no interaction between parameters. Based on Eq. 5, it can be seen that the effect of Zr is

significant. Fig. 3 shows the effect of Zr and stirring temperature on the particle's incorporation (yield) as predicted by Eq. 5. It can be concluded from this figure that Zr plays an influential role in wettability of the boron carbide particles and enhancement of particle's incorporation at a given stirring temperature.

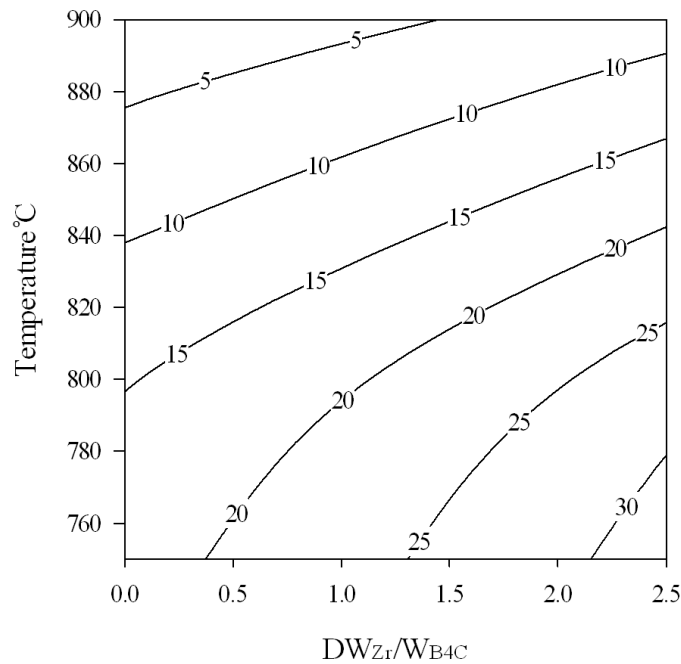


Fig. 3: Variation of particle incorporation versus stirring temperature and Zr based on Eq. 5.

3.2. The interaction layer

Fig. 4 shows a typical particle from sample 6 (stirring temperature of 820 °C, stirring time of 12.5 min and DW_{Zr}/W_{B4C} of 1.25) with a fine white layer at the interface of particle-matrix.

EDS map analysis from this area represents a layer which contains Zr surrounding particle.

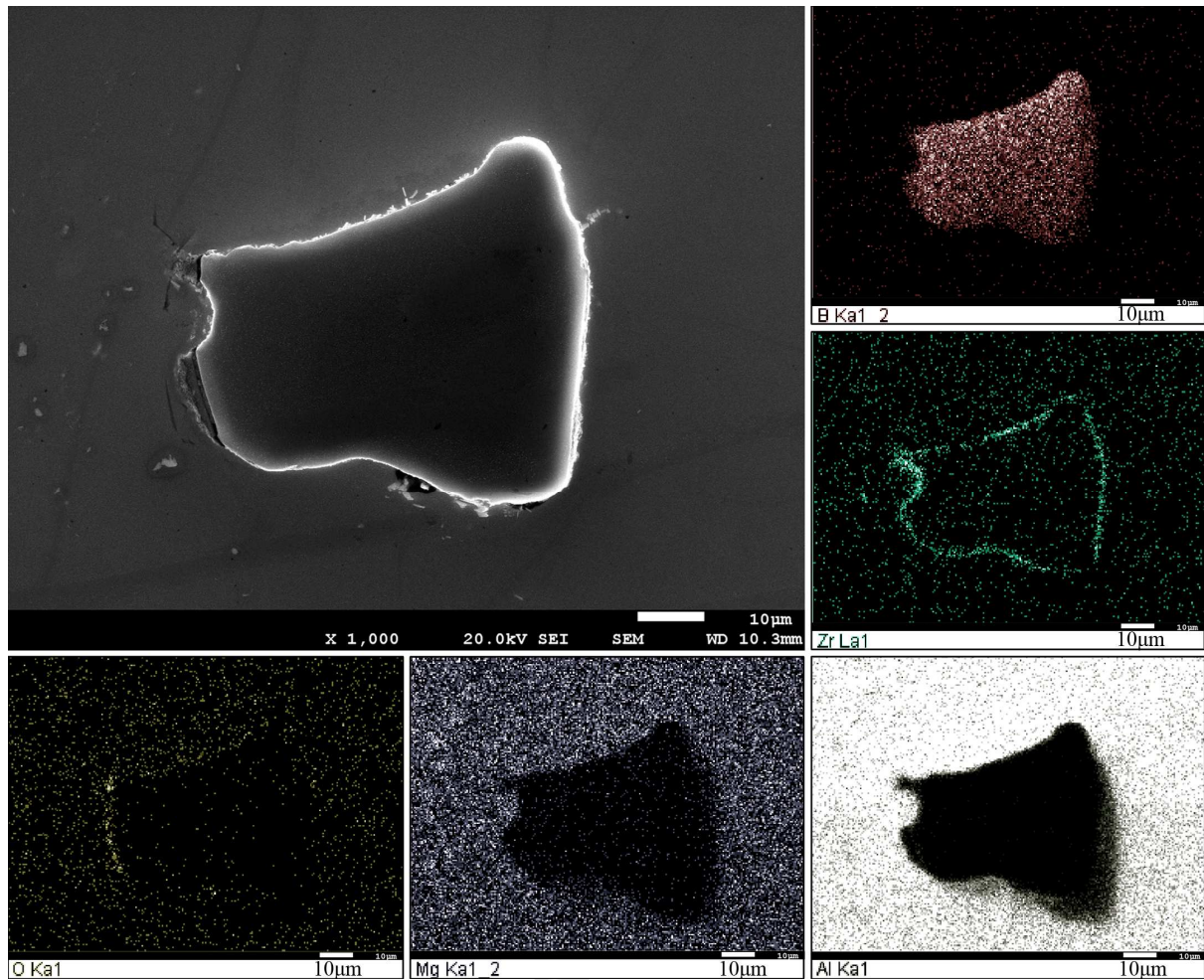


Fig. 4: EDS map for variation of Zr near the B_4C -matrix interface for sample 6 (stirring temperature of 820 °C, stirring time of 12.5 min and DW_{Zr}/W_{B_4C} of 1.25).

Higher magnification of interface layer is presented in Fig. 5 and it can be seen that the layer is formed by some fine and plate-like crystals grew toward the matrix. Thickness of this area varies in different locations of a particle but it is less than 1 μm .

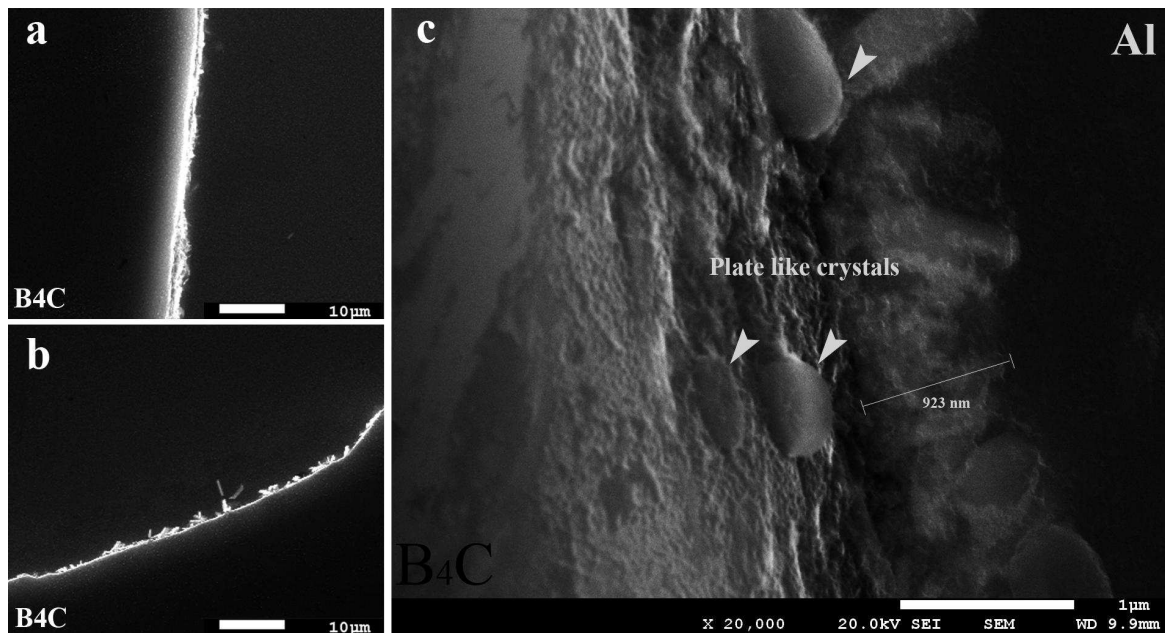


Fig. 5: The morphology of interface layer including crystals in different areas of a particle.

As it is shown in Fig. 6, EDS analysis was applied to identify the composition of the interface layer. According to the variation of elements from B_4C particle to Al matrix at the interface (Fig. 6b), the beginning of the crystal layer is enriched with Al, Zr, and C. Then the value of C and Al are decreasing while Zr and B are increasing, and finally, by reaching the matrix, the amounts of Zr, B and C reach to zero. Therefore, several intermetallic phases can be imagined to be present at the interface. It has been shown that in Al-Zr- B_4C system, the interaction layer contains Al_4C_3 , Al_3BC , $AlB_{12}C_2$, ZrB_2 and ZrC phases according to following reactions [17,23];



Considering the results of EDS analyses and the morphology of the interface, it seems that the initial part of interface consists of Al_3BC or $AlB_{12}C_2$. Moreover, large plate-like crystals are formed from ZrC , and ZrB_2 .

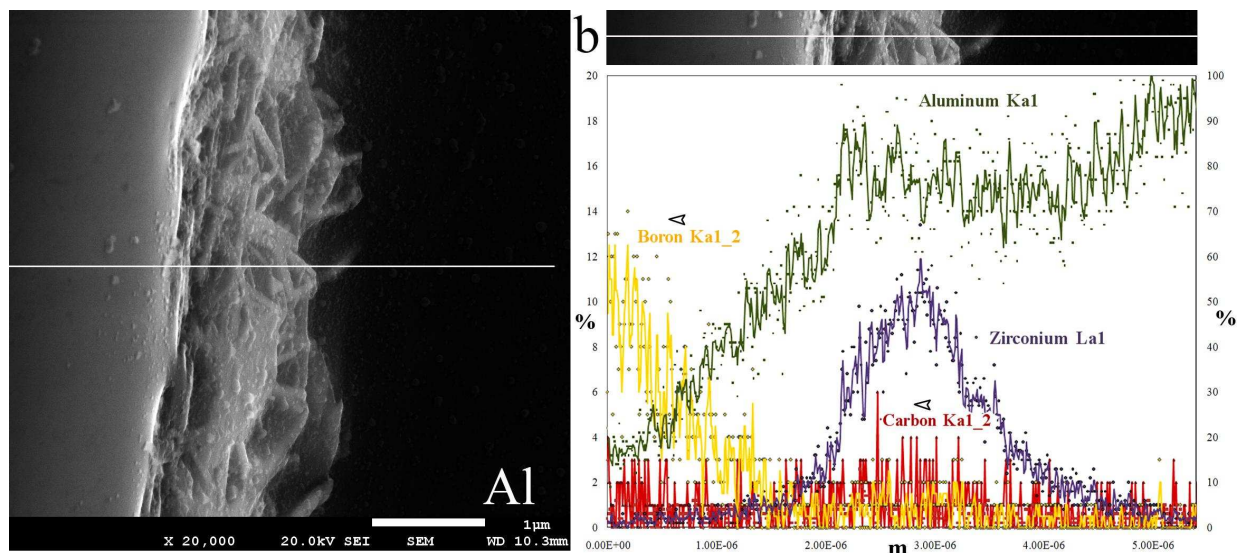


Fig.6: variation of elements through the interface of matrix –particle.

XRD analysis of particles extracted from composite is represented in Fig. 7, and reveals that the interaction layer is mainly formed by ZrB_2 . Based on the number of peaks and peak intensity, this layer also contains some amount of $AlB_{12}C_2$, which was predictable as the result of presence of Al and B and C elements in this area.

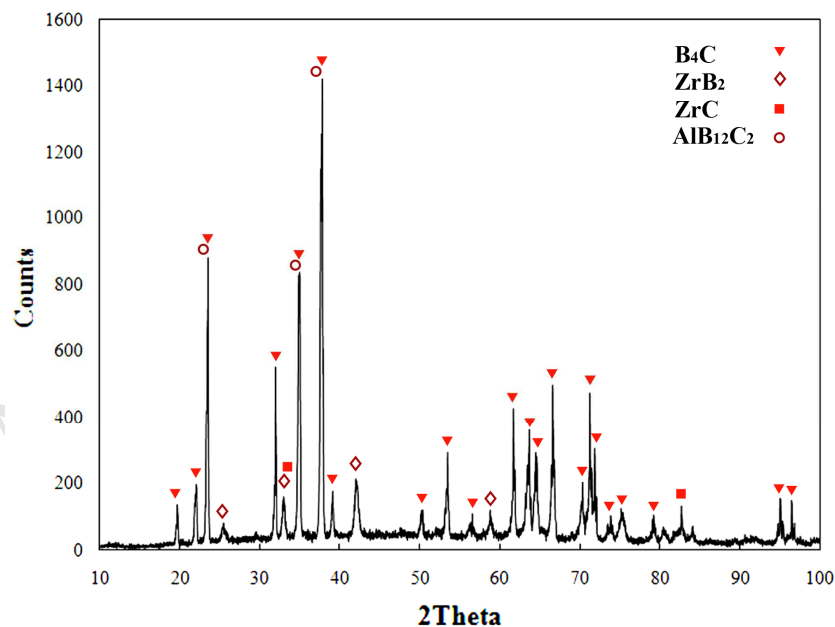


Fig. 7: XRD analysis of bulk extracted B_4C particles (sample #7- stirring temperature of 820 °C, stirring time of 12.5 min and DW_{Zr}/W_{B_4C} of 2.5). It should be noted that the main peaks of B_4C and $AlB_{12}C_2$ are near to each other.

It has been shown that Eq. 1 is an exothermic reaction [18], so heat of formation of ZrC and ZrB_2 can increase the temperature locally around particles [19], which in turn leads to reduction of the contact angle, or in other words, improving the particles wettability. During stirring, particles are floating on liquid surface and are drawn towards the center by the turbulent flow of melt. As a result, particles are entered to the melt and scattered in form of agglomerates [24]. Agglomerates which are not resolved, based on their lower density, will be on the surface of melt as the slag after stirring. Presence of Zr in molten aluminum by facilitating the reaction of Al and B_4C and increasing the temperature around particles, during the stirring, lead to dissolving the agglomerates into the molten aluminum. As a result, incorporation of B_4C particles in the aluminum is increased and the fabrication efficiency is improved. This fact practically can be deduced from comparing the number of agglomerates in composite micrographs (i.e. Fig.1a and b).

Fig. 8 indicates that the thickness of the interface depends on the stirring temperature. It should be noted that although the thickness of interface varies in different particles, overall investigation of samples reveals that casting at higher temperatures results in thicker interface layer. Enhancement of the melt temperature promotes the reaction shown in Eq. 1 due to reduction of Gibbs free energy [18] and also facilitating atomic diffusion. As a result, it is expected to have thicker interface layer and also higher B_4C yield. Besides, according to the solubility limit of Zr at lower temperatures, the ratio of Zr/ B_4C has an upper limitation and exceeding it will result in the formation of some large particles of Al_3Zr , which in turn can impair the mechanical properties of the product [25]. Therefore, working at elevated temperatures, moreover, provides this opportunity to have more Zr in the melt. As a result, formation of the reaction layer will be more promoted.

However, particles incorporation is diminished by increasing stirring temperature as presented in Fig. 3. In fact, casting at higher temperature intensifies the aluminum oxidation

which in turn inversely affects wettability by formation of oxide layer. In the other words, although rising temperature intensifies the interface reaction, the particle incorporation depends on the interaction between aluminum oxidation at surface and Zr reaction with B_4C , and that is why Eq. 5 shows an extremum for yield around 700 °C.

It is necessary to mention that formation of ZrC and ZrB_2 containing layer restricts the atomic diffusion [17], and hence, a very pronounced difference in thickness of interface layer is not expected by little variation of stirring time or stirring temperature. That is why the effect of stirring time on particles yield in this work is not pronounced.

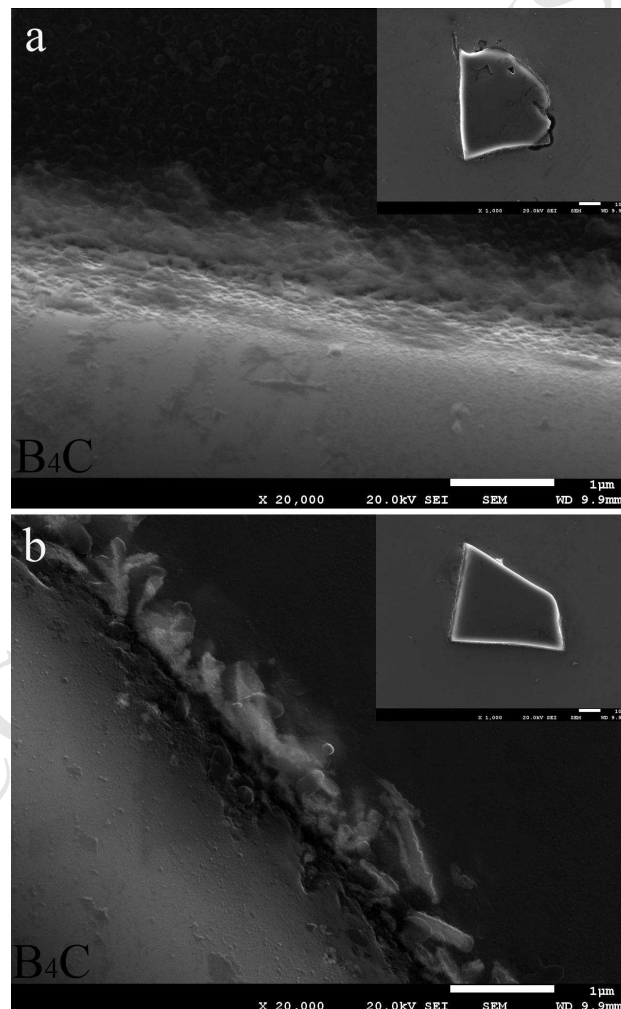


Fig. 8: Interface of B_4C -matrix at different casting conditions, (a) sample#3 (stirring temperature of 750 °C), (b) sample#10 (stirring temperature of 900 °C).

3.3. Mechanical properties and fractography

Fig. 9 shows the engineering strain-stress curves for COMP and COMP_{Zr} as defined in Table 2. As it can be seen, the effect of interface (by comparing COMP with COMP_{Zr}) is much more pronounced during plastic deformation. This might be ascribed to the fact that during plastic deformation, the restraining effect of particles [26] on the deformation of the surrounding matrix highly depends on the properties of the interface. As a result, COMP_{Zr} shows higher strength (8%) and elongation (30%) than COMP (Table 3). It has also been shown that particle–matrix interface acts as a sink for dislocations [27]. Therefore, dislocation pileup behind the particles in the composite materials becomes less pronounced and hardening rate declines. As a result, the interface can play an influential role in hardening behavior. This point can be deduced from Fig. 9, in which the hardening behavior of COMP is inferior due to the weak interface between matrix and reinforcement in this material.

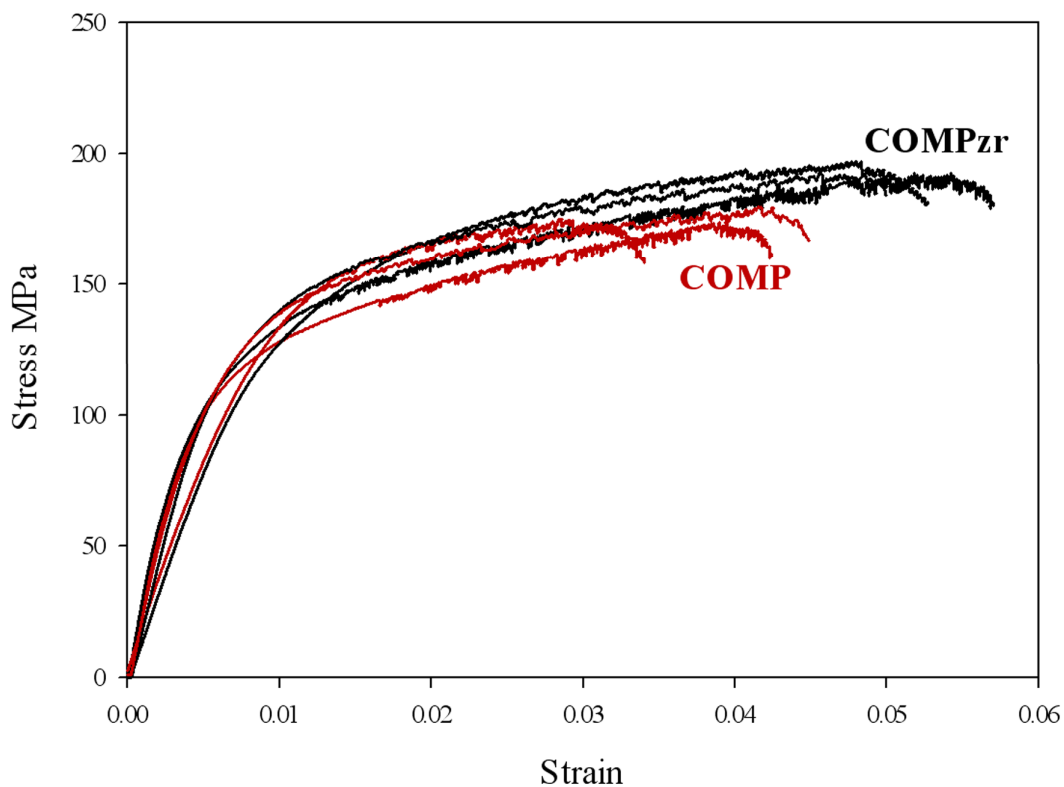
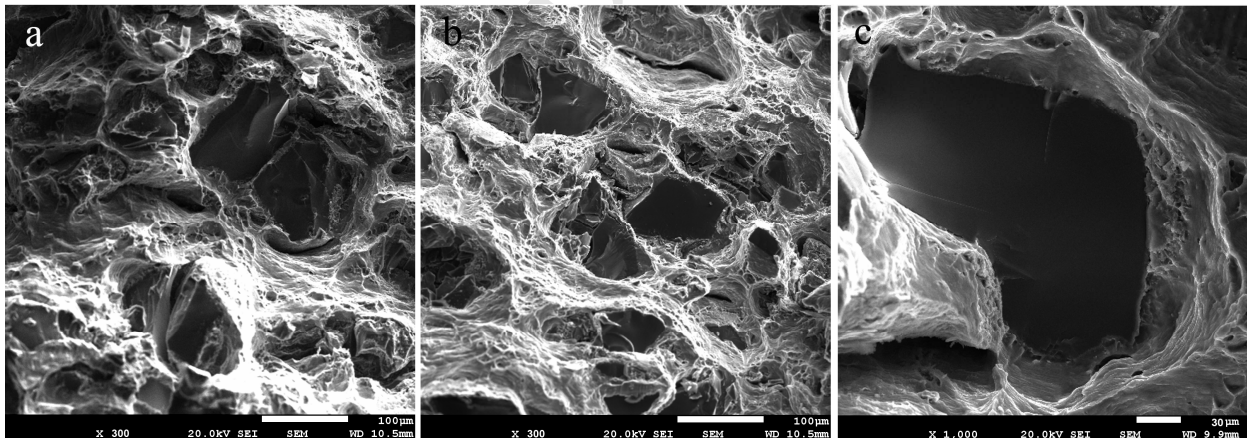


Fig. 9: Engineering stress-strain curves for COMP_{Zr} and COMP.

Table 3: Average mechanical properties of COMP and COMP_{Zr}

	Offset Yield Stress (MPa)	Ultimate Tensile Strength (MPa)	Elongation (%)
COMP	123 ± 6	176 ± 2	4.1±0.6
COMP _{Zr}	122 ± 5	192 ± 3	5.3±0.4

To investigate the effect of Zr-containing layer on the properties of the particle-matrix interface, the fracture surfaces were taken into account. Fig. 10 illustrates the fracture surface of composites subjected to tensile test. According to this figure, fracture proceeded in both specimens by ductile rupture mechanism in the matrix. Dimples are the result of microvoid coalescence [26]. The fracture surface of COMP shows some dimples containing non-fractured particles and the separation have occurred from the particle-matrix interfaces (Fig. 10a). However, Fig. 10b shows that the numbers of fractured particle are greater in COMP_{Zr}, which suggests that the particle-matrix interfaces have adequate strength. These results confirm that the addition of Zr can increase the quality of the interface.

Fig. 10: Fracture surface of a) COMP and b, c) COMP_{Zr}.

4. Conclusion

Zirconium as an alloying element was introduced to aluminum in order to improve the incorporation of boron carbide particles during stir casting. The influential parameters of

compo-casting process including stirring temperature, stirring time and also the amount of Zr were optimized by applying the response surface methodology. The results and corresponding analyses showed that the addition of Zr enhances the particle yield during fabrication process. This fact was attributed to the formation of a reaction layer at the interface of matrix and B₄C. EDS and XRD analyses revealed that this layer is mainly formed by ZrB₂. Formation of ZrB₂ by increasing the temperature around agglomerates during stirring, leads to dissolving them into the molten aluminum. It was found that although stirring at elevated temperature promotes the reaction and leads to form a thicker interface, the intensified oxidation of molten Al has an adverse effect on the casting efficiency. Moreover, addition of Zr led to 8% increase in the ultimate tensile strength and around 30 % increase in elongation of the composite. SEM observations of the fracture surfaces confirmed that a proper bonding presents at the interface of particles and matrix of composite containing Zr.

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Figure captions

Fig. 1: Optical micrographs of Al/B₄C composites for (a) Sample 1, (b) Sample 6 and (c) Sample 10.

Fig. 2: Optical micrographs of (a) COMP_{Zr} and (b) COMP.

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Fig. 10: Fracture surface of a) COMP and b, c) COMP_{Zr}.

Highlights

- Using Zr in Al-Mg/B₄C system improves the particles incorporation by promoting the particle matrix interfacial reaction.
- Based on SEM, EDS and XRD, the fine reaction layer mainly consists of ZrB₂.
- The reaction layer strengthens the particle matrix interface which in turn, improves the mechanical properties and fracture behavior of product.