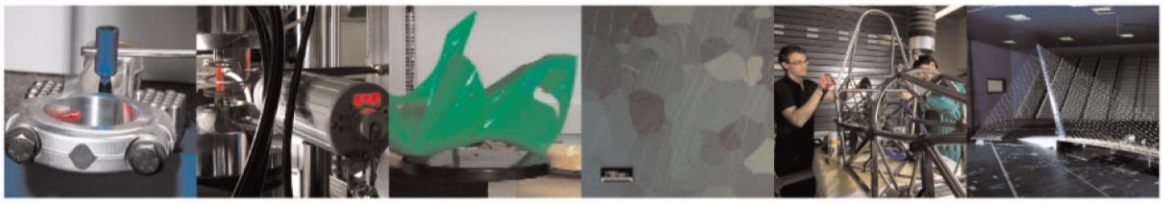

Manufacturing of B4C particle reinforced A360 aluminium cellular composite materials by the integration of stir casting and space holder methods

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Manufacturing of B_4C particle reinforced A360 aluminium cellular composite materials by the integration of stir casting and space holder methods.

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Abstract

Stir casting method has become prominent for fabrication of metal matrix composites in recent years. This method can be adjusted for casting around space holding particles to obtain cellular composite materials. In this study, a specific method which is a combination of stir casting and space holder techniques were used to produce open-celled A360 aluminium-B_4C composite foams with regular sized and distributed pores. Weight ratios of reinforcement particles determined as 0.5, 1, 1.5 and 2 %. The influences of particle reinforcement on the microstructure and the mechanical behaviour of composite foams were investigated. Microstructures were analysed with optical microscope (OM), scanning electron microscope (SEM). Compression and hardness tests were carried out to observe the effects of reinforcement on mechanical properties. Compression strength properties and hardness of composites increased with the ceramic reinforcement, however the plastic strength of the composite foams showed worsening trend after a certain reinforcement ratio (0.5 wt.%). Energy absorption properties of the composite foams showed parallel trends with compressive strength properties.

Keywords: composites; reinforcement; cellular materials; mechanical properties; Stir-casting.

1. Introduction

Materials with porous or cellular structure have interesting properties like low weight, high stiffness and impact resistance [1–3]. There are several advanced manufacturing techniques such as gas injection, powder metallurgy [3] or additive manufacturing [4] to produce these specific materials. Another technique that can be used to produce cellular materials and recently has increased its importance due to its versatility is infiltration technique [5]. These techniques are commonly used to produce cellular metals and metallic foams. However very wide range of cellular metals can be produced successfully by these techniques, advanced manufacturing methods demand high production costs such as special processing technologies, machine and equipment prices [6]. The low-cost manufacturing methods can make these advanced materials more attractive for the industry where specific functional features are desired. The casting of metals and alloys around space holder material (space-holder method) is very simple way to obtain metallic foams or cellular parts [4, 7-8]. Moreover, space holder particles directly affect the structure parameters of metallic foams such as porosity, pore shape, pore size and homogeneity of pore distribution, etc. [9-10]. In the meantime, within the space-holder method, powder metallurgy technique also can be preferred instead of casting [11]. Stir casting route is known as producing metal matrix composites in an economic way [6, 12-13]. Producing aluminium matrix composites via stir casting is also in great demand lately [12, 14]. A method which is a combination of these two methods mentioned above can be very useful and controllable way to produce particle reinforced composite foams.

Addition of reinforcement particles have direct effects on mechanical properties such as hardness, tensile and compressive strength. Al-Si-Mg (3xx.x) alloys are reinforced with ceramic particles such as Al_2O_3 and B_4C with different reinforcement particles ratio. The studies on alloy A356 reinforced with B_4C and Al_2O_3 particles showed that ceramic particle addition enhanced hardness and tensile strength of the materials [15, 16]. This improvements on mechanical properties are interpreted as grain refinements due to the addition of hard ceramic particles [16]. The reinforcement with B_4C particles of wrought aluminium alloys such as 7075, 6061 showed also similar results. Hardness and tensile strength raised with the increment of B_4C volume content [17, 18]. Al/ Al_2O_3 (0-10 vol. %) composite foams which has different porosities (50-70 %) have been produced by powder metallurgy method using carbamide space holders. It is concluded that decreasing porosity results with a raise on compressive strength and energy absorption capacity. It is also stated that composites with 2 % volume content of Al_2O_3 showed superior properties [19]. Stir casting method was used to produce ZA27/SiC particle (10 wt.%) reinforced composite foam using CaH_2 (0.4-0.6 wt.%) foaming agent. It is reported that resulting ZA27/SiC composite foam showed greater strength than that of ZA12 or ZA22 foam of same relative density [20]. High strength composite foams also studied. Low carbon steel and stainless-steel composite foams were produced with using steel hollow spheres covered and surrounded by steel powders. Composite steel foams showed a superior energy absorbing capability and a higher strength [21].

Previous studies with metal foams with regular pore shape and distribution have reported several results and applications of lattice or porous structures made with different production methods [22–24], whereas there is a small number of research on composite foams with standard shape and regular distribution of pores in the literature [25-26]. The

objectives of the present work are to produce A360- B_4C open-celled aluminium matrix composite foam with regular pore shape and distribution material and to investigate the effects of different weight ratios of the reinforcement particles on microstructure and mechanical properties. In pursuit of this purpose, the samples were examined experimentally using optical microscopy (OM), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS). Mechanical properties were also studied by applying compression and hardness tests.

2. Materials and method

A360 (AlSi10Mg) aluminium material with the commercial name of Etial-171 was obtained from Eti Aluminium Inc. company and was used as main material. Boron carbide (B_4C) ceramic powders with 16µm average particle size were used as reinforcement particles for cellular composite foams. The chemical composition of the A360 alloy is reported in Table 1. Shell moulding sand with of 3.5 % of resin ratio and 65-75 AFS number was obtained from Marmara Metal Products Corp. and was used as space holder materials to produce A360 (AlSi10Mg) aluminium- B_4C composite foam.

Table 1. Chemical composition of A360 cast alloy.

Element	Si	Fe	Cu	Mn	Mg	Zn	Ni	Ti	Al
Wt. [%]	9.86	0.42	0.08	0.41	0.38	0.09	0.1	0.15	Bal.

Shell core sands were pressed and heated at the temperature of 175 ^{0}C in a metal mould and thus space holder laminates were obtained. The pore geometry, pore size and porosity of composite foams can be controlled since the dimensions of space holder geometries were fixed. The pore geometrical shape and dimensions was determined as 10 mm x10

mm and cubic variation with 18 faces and 48 edges. Detailed view of the pore shape and resulting cellular cell structure are shown in Fig.1.

[insert Figure 1.]

Stir casting system consists of a resistance furnace, an electric motor for stirring and a control unit. That main components were set up over a steel construction frame and details were given at Fig.2. Melting process was taken place on a 304L stainless steel crucible which has 6 mm thickness. The crucible has maximum 2.4 kg capacity for proper melting and stirring. The resistance heating of furnace was provided by A1 type Kanthal wire with a thickness 2 mm and total length of 30 m. Wrapped resistance wire was covered inside the Hycast95 isolated outer refractory which length is 40 cm with 25 cm diameter and 4 cm wall thickness. The manufacturing system uses approx. 3.3kW heating power. For controlling the temperature during melting and stirring, GEMO-dt106 PID temperature controller with K-type thermocouple was used. Stirring was carried out with 4-blade stainless-steel propeller and rod powered by a A.C. electrical motor with Delta VFD-EL A.C. motor drive. The motor was mounted as vertically movable with the mechanical stirrer.

[insert Figure 2.]

A360 ingot sliced into small pieces and replaced into the crucible. A pre-heat treatment, keeping at 165^0C for 2 hours was implemented to B_4C particles to obtain better wetting conditions [27]. B_4C powders were weighed and wrapped with thin aluminium foil and then placed into the crucible. The reinforcement weight ratios were determined as 0.5, 1, 1.5 and 2 %. Besides, A360 alloy metallic foam without particle reinforcement was produced for comparison with composite foams. The furnace cap was closed and tightened with lock nuts. Melting was occurred in an argon atmosphere created by a gas

flow-meter which supplies continuous low-pressure gas flow into the furnace. Mechanical stirring with the speed of 300 rpm was applied at the temperature of 680^{0}C for 3 minutes. The stirring speed are very important to get successful results. Hashim at all. Reported that the stirring speed should not be very high, additionally it should continue for a while before pouring of liquid metal [28]. It is also reported that the best distribution of Al2O3 particles in composite microstructure obtained at stirring speed of 300 rpm [29]. After stirring process, graphite stopper was pulled horizontally to pour liquid metal into the shell mould cavities than left for solidification in laboratory environment. Last process to obtain metallic composite foam was to remove the space holder particles. Space holder particles were prepared by shell core sand which consists approx. %3.5 resin inside. After casting process, the resin burned with the effect of over-temperature. The binding between sand particles diminished due to this effect. Space holder particles were removed from the resulting samples as mechanically (with a simple skewer tool) and cellular composite materials was obtained. Fig.3. illustrates the process steps of production route.

[insert Figure 3.]

Traditional metallographic sample preparation procedures as cutting, grinding, polishing and etching were applied to all samples for microstructure examinations. Keller reagent (95 mL distilled water, 2.5 mL nitric acid, 1.5 mL hydrochloric acid, 0.5 mL hydrofluoric acid [30]) was used for etching of polished surfaces. Carl Zeiss Ultra Plus Gemini scanning electron microscope (SEM) equipped with Bruker X Flash 6/10 energy dispersive spectroscopy (EDS) were used for characterization studies. Hardness values of the composite samples were measured on Shimadzu microhardness tester as applying 4,903 N test load for 20 seconds. The measurements were repeated 3 times for each

sample and the average values were calculated. Compression tests were carried out using Zwick/Roell 600kN mechanical tester with the compression speed of 0.005 s^{-1}. The specimens shown in Fig.4. were processed delicately by turning and cutting to obtain flat and parallel faces. Tests were repeated 3 times for each sample to obtain mean values. The resulting compression test samples had 80mm length and 50mm diameter.

[insert Figure 4.]

A specific test method which is titled as mechanical testing of metals -ductility testing-compression test for porous and cellular metals ISO13314 was followed for compression tests. This standard has some terms such as first maximum compression stress, plateau stress, quasi-elastic gradient to evaluate the compression test results [31]. According to ISO 13314, plateau stress is a term which is arithmetical mean value of the compression stress data between the strain of 30 and 40 %. First maximum compressive stress can be expressed as the stress point where the compression stress starts decreasing for the first time in the stress-strain curve. Another term to evaluate test results is quasi-elastic gradient. Quasi-elastic gradient represents the porosity dependent rigidity and calculated as the slope of the linear elastic region of stress-strain curve [31]. Additionally, energy absorption capacities were obtained by calculating the area under the stress-strain curves up to %50 strain.

3. Results and discussions

A360-B_4C composite cellular materials with regular pore shape and distribution were produced successfully. The fabricated composite samples are also shown in Fig.3. Porosities of metallic foams were calculated as a fraction of weights in porous state and dense state [32, 33]. The weight of ceramic particles also considered throughout

calculating the porosities. Each porosity result value was extracted from three different measurements as an average value. Fig.5 indicates the porosities of the sample produced by stir casting method.

[insert Figure 5.]

When the porosity results evaluated, approximately 60 % porosity obtained from the samples. The porosity levels for metallic foams which produced with Al, Zn, Pb and Cu main materials by casting around space holders were reported as lower than 65 % [7, 34, 35]. These results confirm that composite foams samples produced in this experimental study showed consistence with these reference works. The adjacent porosity results can be pointed from that the space holder geometries have constant shape and distribution. It is also clear that B_4C addition has less considerable effect on porosity values. However, a small decrease on porosity approx. %2 can be seen in Figure 5, this level of decrease is not significant statically because the weighted average standard deviation of the group is 2.21. This small decrease can be occurred because of the small dissimilarities of liquid metal temperature. In liquid state-produced composites, the properties can be affected by melt temperature and the interfacial reaction between molten matrix and the reinforcement particles [36]. Soltani at all. [37] reported that Al and SiC mainly react with each other just after the melting of aluminium and large release of heat is seen. The increase of particle amount will enrich these effects and alter the melt temperature. They also reported that, higher viscosity of the melt increases the porosity level by preventing gas escape in lower temperatures. Accordingly, it is possible that the porosity reduces at a higher melt temperature. Therefore, the stirring and casting at higher temperature might lead the lower porosities.

3.1. Microstructural characterization

Aluminium 3xx series and specially A360 alloy consists Al crystals, eutectic (Al+Si), Mg_2Si and some intermetallic phases such as AlFeSi and AlFeMnSi [38-40]. Fig.6. shows the microstructure of A360 alloy which is in the state of casting (without modification). As shown in Fig.7., AlFeSiMn and Mg_2Si phases were formed in the microstructure of A360. The light grey phase which was tagged with number 1 in the Fig.6. is known as AlFeSiMn and called Chinese Script in the literature [41]. Black (dark grey) phase which is labelled with number 4 was shown as Mg_2Si phase.

[insert Figure 6.]

[insert Figure 7.]

The SEM images of A360/ B_4C samples reinforced different weight ratios and the sample without reinforcement were obtained. The images in Fig.7. indicate matrix regions of A360 alloy and the samples reinforced with B_4C particles. It can be seen from the Fig.7. that the SEM microstructure of the samples showed accordance with the microstructure of alloy A360 which is shown Fig. 6. In Figure 7, 2% B_4C SEM images show that, AlFeSiMn phases formed in larger sizes when compared the other images. That can be due to the temperature rise with the effect of interfacial reaction between matrix and the particles. Therefore, the B_4C particles can be localized around of AlFeSiMn phases because of that reasons. EDS analysis also showed that there are B_4C particles in the microstructure. The spectrums that is thought to be taken from B_4C particles, are shown in Fig.8. In addition, B_4C particles analysed with using the elemental mapping feature of EDS. By considering the results shown in Fig.8., B_4C particles are existed and well wetted into the matrix material.

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[insert Figure 8.]

3.2. Hardness measurements

As for the hardness measurements, Fig.9.a. shows the micro-hardness results of composite foam samples. The hardness value of the A360 aluminum alloy was measured as approximately 70 HV, and the hardness values of the samples reinforced with 0.5, 1, 1.5 and 2 wt. % B_4C, are determined as 75.4, 82.8, 84.8 and 83.4 HV respectively. Additional micro-hardness measurements were carried out from the vicinity of B_4C particles and illustrated in Fig.9.b.

[insert Figure 9.]

It is concluded that the addition of B_4C causes significant increment on hardness values. The positive effects of the addition of hard ceramic particles were reported before in some reference works [29, 42-44]. These hard-ceramic particles prevent dislocation movement and resists the deformation, so hardness and strength of the material increases [37, 45]. The increment in hardness may be derived from the rise of strain energy the collapse of dislocations around ceramic particles distributed in matrix phase [37, 43, 45]. Some studies reported that Orowan mechanism which is defined as occurrence of dislocation circles around reinforcement particles, has an effective role on the increase of hardness and strength [29,46]. It is possible that the increase of strength can be because of the load transportation from matrix to reinforcement materials [29, 43]. Due to the difference of coefficient of thermal expansion properties of aluminium and ceramic particle, inherent stress and condensation of dislocations can be accrued and this can enhance the strength and hardness of the materials [29,43,47,48]. Several studies conducted on aluminium matrix composites reinforced with ceramic particles such as B_4C, SiC and Al_2O_3 [42-45, 47-52]. In this regard, the rise in hardness showed an accordance with the literature.

However, B4C addition seems to improve the hardness of the composites, the hardness of sample 2% B4C measured lower than the 1.5% B4C. In Figure 7 the microstructure of 2% B4C has significantly greater amount of AlFeSiMn secondary phase area. The size and distribution of these intermetallic phases affects the properties. The excess amounts of this secondary phases decrease the mechanical properties of casting aluminium alloys [53]. The small hardness drop of 2% B4C sample can be interpreted by this phenomena.

3.3. Compression test results.

Stress-strain curves generated through calculating the average the resulting curves of three compression test. Fig.10. shows the compressive stress-strain curves of the samples produced through this study.

[insert Figure 10.]

In the compression tests of cellular materials, generally three phases of deformation were seen. The first region is related with the elastic deformation of the cell walls. The second region is called as plateau region. Plateau region is related with the plastic deformation of cell walls and the stress data fluctuates in that region. The final phase is densification phase which the compression stress increases due to densification. In this experimental study only the first and second deformation phase were focused. The aluminium composite foam reinforced with 0.5 wt.% B_4C showed greater compressive properties with the first maximum stress of 58.164 MPa and the plateau stress of 32.502 MPa. Higher reinforcement ratios than the 0.5 wt.% B_4C effected compressive properties negatively. It was reported that, after a significant level of ceramic content, the higher reinforcement ratio has negative effects on mechanical properties of metallic foams produced via space holder particles [54,55]. This can be interpreted as the ceramic

particles becoming more fragile due to agglomeration and this leads a lower compression strength. The quasi-elastic gradient values represent the elastic properties of the composite samples and are reported in Fig.11.

[insert Figure 11.]

The composite foam with 2 wt.% B_4C which is the greatest amount of reinforcement in this study showed the highest quasi-elastic gradient value. Higher quasi-elastic gradient value indicates that the material is more brittle. Ceramic particle addition provides higher hardness, this causes the material to behave more brittle. In contrast to bulk aluminium composites, cellular composite materials deform more easily because of the smaller surface area caused by cellular structure. On the other hand, micro-porosities and agglomeration of particles occurred inside the materials by the nature of stirring process and micro-porosities effects hardness and strength negatively [44]. It is reported that plateau stress value is decreased with increasing of porosity [56]. A rise of 10 % in porosity results a 30 % decline in plateau stress [54].

Energy absorption values were calculated as the area under the stress-strain curves up to %50 strain. Fig. 12 shows the mean energy absorption values of the samples.

[insert Figure 12.]

Shape geometries has effects on the energy absorption properties of different samples [23]. On the other hand, it is also reported that the energy absorbing capacity of the cellular metals is dependent on its relative density [57]. Another work reveals that the trend of energy absorption is consistent with compressive strength and plateau stress for aluminium matrix syntactic foams prepared by stir casting [58]. It is also reported that the mean plateau strength has a deep effect on the energy absorption capacity [59]. Composite foams absorbed greater amount of energy than the A360 alloy foam. A study conducted

on the production of open cell Al-Al2O3 composite foams having different volume fractions of Al2O3 (0-10vol.%) supports the similar trend as the energy absorption capacity of the composite foams was more than that for Al foams [19]. The sample reinforced with 0.5% wt. B4C showed the best energy absorption capacity. After 0.5% B4C content the energy absorption values decrease gradually. It is also reported that the energy absorption properties of Al/Al2O3 composites with different reinforcement ratios follows the trend like the plateau stress [19]. In this study energy absorption capacities were showed similar tendency with the compression strength of the composite foams.

4. Conclusions

This study consists of the manufacturing of open cell aluminium foam with the reinforcement of B_4C particles. Beside OM, SEM-EDX mapping, also mechanical analysis methods such as hardness and compression tests were applied to fabricated composite foams. The samples are open celled and regularly distributed in the structure. All samples showed similar porosity levels around 60 % due to the standard design of space holder geometry. SEM images demonstrated that the composite foam has B_4C particles inside the matrix. The mechanical behaviours of the samples were evaluated with hardness and compression tests. The size of secondary phases plays an important role on hardness and compressive properties of composites. Hardness of the samples were raised by the increment of B_4C reinforcement. Results show that, the metal foam with 0.5 wt.% B_4C reinforcement has the greatest first maximum compressive stress, plateau stress and energy absorption capacity. The composite foam which was reinforced with 2 wt.% B_4C showed the highest quasi-elastic gradient value. This indicates that the sample reinforced with the greatest amount of ceramic reinforcement is the sample which is the

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most brittle. On contrary, addition of 2 wt.% B_4C increases the hardness but decreases the compressive strength of the foam.

Acknowledgments

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