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Analysis of Microstructures and Mechanical Properties of Particle Reinforced AlSi7Mg2 Matrix Composite Materials

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The present study examined the microstructures and mechanical properties (tensile and impact strength, hardness) of selected metal matrix composite materials. SiCp reinforced AlSi7Mg2 matrix composites were produced using gravity and squeeze casting methods and subsequently T6 heat treated. Some of the squeeze casted composites were shaped by extrusion. The extrusion generated an equiaxed matrix structure and SiCp caused a homogeneous distribution. The quasi absence of porosity in the squeeze casted composites led to improved mechanical properties. Whereas an increase in the SiCp ratio resulted in an increase of the tensile strength, it led to a decrease of the impact strength values. The enhancement of the mechanical properties following an applied heat treatment was better for materials shaped by extrusion.

Keywords casting, extrusion, mechanical testing, metal matrix composites (MMCs)

1. Introduction

Continuously developing technologies contribute to improvements in materials science. Composite materials have now replaced traditional engineering materials. Metal matrix composite materials (MMCs) are especially preferred for high temperature applications in many areas such as the automotive, space, and aviation industries as well as sport applications due to their light weight, high mechanical properties, excellent wear, and corrosion resistance. The production of composite materials and the development of subsequent shaping processes have become very relevant for technological applications.

MMCs cannot be easily fabricated using conventional methods. The fabrication techniques can vary considerably according to the choice of the matrix and reinforcement materials and to the type of reinforcement. In general, there are two types of fabrication methods, namely, the solid phase and liquid phase fabrication methods (Ref 1). In the liquid phase fabrication method, relevant factors include high strength and elasticity modules, low density, high melting temperature, thermal stability, and reinforcement of the material form and size. The compatibility of the ceramic reinforcement with the matrix alloy must be considered. Since many ceramic particles cannot be wetted by liquid matrices, it is necessary to add a material that improves the wettability with the liquid or coat the particles before mixing (Ref 2). Magnesium and silicon in liquid aluminum increased the wettability of the matrix and assisted to the particle incorporation (Ref 3, 4).

High casting and forming capabilities are required to produce good materials. Both the formability and the mechanical properties of the materials are directly related to the material microstructure. Improvements in the microstructure of the composite materials have been observed using the squeeze casting method. The application of high squeeze pressures provides positive effects such as a decrease in porosity or improvements of the tensile strength and gravity (Ref 5).

The squeeze casting process allows us to forcibly charge a liquid metal into a preheated ceramic fiber or any preformed reinforcement that is inserted in a metallic die. Thus, it enables the molten metal to solidify by applying high pressures, and thereby squeezing the liquid metal. The preform of the ceramic fiber is pre-heated to below the melting temperature of the matrix and then inserted into a metallic die. Al or Mg alloys are heated below their melting temperatures and squeezed into the fiber using a hydraulic press to form the fiber and molten metal mixture (Ref 1). Whiskers or particulates can also be mixed with molten metals before squeeze casting. Cast aluminum alloy composite materials containing SiC, Al_2O_3 powders, and silicon nitride whiskers can be fabricated using this method (Ref 6). As reported by Ghomashchi and Vikhrov, the squeeze casting process was first described by Chernov in 1878 who applied steam pressures to molten metals that underwent solidification processes. Despite of its 100-year-old invention, the commercialization of squeeze casting has only been achieved very recently. This method is used to fabricate high integrity engineering components with or without reinforcement (Ref 7).

As described by Dutta et al. cast metal matrix composites ingots were shaped to obtain a homogeneous distribution, low porosity, and finer grain structures using the extrusion method (Ref 8). The values of the ultimate tensile strength and the elongation of the extruded composites were greater than those of hot pressed composites. The elongation of the composites doubled after extrusion (Ref 9). Extrusion is a common primary

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shaping method used for all wrought MMCs. In a study conducted by Jeffrey et al. who analyzed the extrusion of the particulate reinforced aluminum composites produced following a casting route, it was stated that hot extrusion is a viable process for the high volume production of shaped composites without incurring any fracture of the reinforcement or the formation of a strong anisotropy (Ref 10). The extrudability of the composites exhibits several similarities with unreinforced aluminum with respect to the speed of extrusion, the surface finish, and the extrusion ratios. Brusethaug et al. studied the extrudability of a type A357 discontinuous cast aluminum alloy reinforced with 15% SiC (20 μm) particulates. The grain structure of the reinforced material after extrusion was equiaxed, recrystallized and more independent on the billet temperature (Ref 10).

In this study, many earlier articles on particle reinforced composite materials were investigated and a new composite material was studied. This work analyzes the microstructure, the tensile and impact strengths, and the hardness values of AlSi7Mg2 matrix composite materials containing SiC particle reinforcements in 5, 10, and 15 wt.%, which were produced using gravity and squeeze casting and shaped using hot extrusion methods. The results show considerable improvements in mechanical properties due to extrusion and T6 heat treatment of the metal matrix composites (Ref 11).

2. Experimental Procedures

2.1 Material

AlSi7Mg2 was selected as the matrix material. 99.7% pure Al was added to AlSi12. Therefore, the Si ratio was dropped approximately to 7%. Then, 2% Mg was supplemented. Consequently, the AlSi7Mg2 matrix material, see chemical composition in Table 1, was produced. SiC particles with a density of 3.18 g/cm^3, 400 mesh (11-44 μm) according to FEPA (Federation of European Producers of Abrasives) standards with an average diameter of 23 μm were selected as the reinforcement material.

2.2 Production of MMCs

Squeeze and gravity casting methods were selected for the production of the composite material. The AlSi7Mg2 matrix material was put in the crucible placed in an induction furnace and was subsequently melted. SiC particles in 5, 10, and 15% weight ratios were mixed with a graphite rod. A fraction of this mixture was then poured into a preheated mold at 100 °C made of hot work tool steel (W.Nr. 1.2714). The pouring temperature of the mixture was 740 °C. The molten material was compressed with squeeze pressures of 100 MPa using a hydraulic press. A squeeze pressure of 100 MPa was shown to be sufficient to guarantee maximum mechanical properties (Ref 12). Thus, cylindrical experimental blocks having a diameter and length of 90 mm were produced using the squeeze casting method. Some of the mixture was die-cast (gravity) and cylindrical specimens of 35 mm in diameter and 200 mm in length were also obtained.

Table 1 Chemical composition of AlSi7Mg2 matrix material (%)

Al	Si	Mg	Cu	Fe	Zn	Mn
Balance	7.48	2.47	0.05	0.357	0.043	0.047

2.3 Hot Extrusion of MMCs

A fraction of the MMC materials produced using the squeeze casting method were shaped at 450 °C, using a 23:1 extrusion ratio for the hot extrusion method so that bars of 25-mm diameter were obtained. The extrusion process was carried out in a horizontal extrusion press and an extrusion pressure of 150 MPa was recorded. The extrusion speed was 0.5 m/min.

A T6 heat treatment was applied after the casting for some specimens (solution heat treatment at 520 °C for 6 h, water quenched at 20 °C). An artificial aging was carried out at 170 °C for 5 h and finally air cooled to the environment temperature.

The energy-dispersive x-ray spectroscopy (EDX) analysis of the composite material containing 5% SiC was and produced using the squeeze casting method was done using a Jeol brand JSM5410LV model scanning electron microscope (SEM).

Specimens taken from composite materials obtained using squeeze and gravity casting and shaped by extrusion with and without application of a T6 heat treatment were subjected to a metallographic examination. Before the examination, the specimens were ground with emery papers having a 320, 400, 600, 800, and 1000 mesh size. They were first polished using chromium oxide powder and then successively 6 and 1 μm diamond pastes. The etching process was conducted at room temperature for 5-25 s using a distilled water mixture containing 0.5% HF, 1.5% HCl, and 2.5% HNO_3. Photographs of the composite materials were taken using the SEM Jeol brand JSM5410LV. The microstructure pictures represent back scattered electron images (BSEI).

The densities of the composite materials were calculated theoretically and experimentally and then compared to each other. A scale with a measurement precision of 0.0001 g was used for the density measurements and Archimedes principle was applied. The porosity of the composite materials was determined by using the theoretical and experimental densities, according to the equation:

$$\text{Porosity }\% = \frac{d_t - d_e}{d_t},$$

where d_t and d_e represent the theoretical and experimental densities, respectively.

Tensile and impact tests were carried out to determine the mechanical properties of the composite materials produced using squeeze and gravity casting methods and shaped by extrusion. The hardness values were also measured.

For the tensile tests, the specimens conformed to EN 10002 standards were prepared from composite materials made with and without T6 heat treatment. The tensile tests were made using a Shimadzu Autograph AG-250kNG model tensile test machine (Kyoto firm) at a deformation speed of 1 mm/min.

The specimens conformed to EN 10045 standards were prepared for Charpy impact tests. The tests were carried out at room temperature using 150 J in a Frank device.

The surface roughness was obtained using emery papers of 320, 400, 600, 800, and 1000 mesh for specimens taken from each material group for the hardness tests. The hardness values were measured using a Riechter hardness measurement device and the Brinell hardness (HB) format (EN ISO 6506). For the hardness measurement, a 625 N load and a ball of 2.5 mm in diameter were used.

3. Results

3.1 Energy-Dispersive X-ray Spectroscopy Analysis

Microstructural images of the composite materials containing 5% SiC and produced using the squeeze casting method are shown in Fig. 1. As a result of the EDX analysis, 100% SiC was intensively determined in Point 1. Point 2 was taken from the matrix/particle interface. It is a Mg_2Si precipitate and contains Mg, Si, Al, and O_2. Mg can easily migrate to the matrix/particle interface, which is a thermodynamically unstable region. Due to the high reactivity characteristics of Mg, intermetallics such as Mg_2Si have a tendency to precipitate near the particles (Ref 13). For Point 3 taken from the small dark regions in the microstructure, the density decrease is related to the existence of a spinel phase characterized by Mg_2Si, which was generated from Al, Si, Mg, and O_2. Point 4 consists of Al and Si forming the matrix alloy. The white parts shown on Point 5 represent a β-AlFeSi intermetallic phase rich in iron.

3.2 Microstructure Analysis

SiC particles are pushed toward the eutectic regions. They constitute the regions that cool the last during the solidification process because of the slow cooling of the composite materials made using gravity casting methods. This leads to the formation of particle clusters and to a non-homogeneous distribution in the matrix (Fig. 2). The homogeneity of the distribution of SiC particles was found to improve with increasing the SiC volume fraction from 5 to 15%. The same result was reported by Bindumadhavan et al. for aluminum alloy A356-SiCp metal matrix composites (Ref 14). The micrograph of AA7010-10 wt.% SiC reinforced composite clearly shows the uniform distribution of SiC particle in 7010 alloy matrix and the good interface bonding between SiC and matrix (Ref 15). However, the formation of porosities increases with by increasing the reinforcement ratio from 5 to 15%. As a result of the gravity casting method, the formation of porosities between the reinforcement and the matrix increases due to the weakness of the matrix/reinforcement interface bond. This behavior was especially observed in the particle clusters sections.

Applying a T6 heat treatment to the composite materials produced using a gravity casting method led to a spherical Mg_2Si precipitation in the microstructure due to diffusion. The plate and pin form a β-AlFeSi intermetallic compound, which negatively affects the mechanical properties of the composite materials negatively and remains in the structure after the heat treatment. However, these negative effects decrease when the sharp corners are rounded.

When the microstructures of the matrix alloy and composite materials produced using a squeeze casting method were studied, it was seen that the microstructure was composed of primary Al dendrites with SiC containing eutectic Si (Fig. 3). Attia and Lee et al. reported that a high cooling rate should be imposed on the melt to produce a refined network of primary aluminum dendrites. Thus, it becomes possible to obtain a more homogeneous distribution of SiC (Ref 16, 17). Addition of SiC particulates to the Al-Si alloy resulted in a morphology and distribution of eutectic Si phase that formed secondary phases, which changed the volume fraction of the intermetallic phase

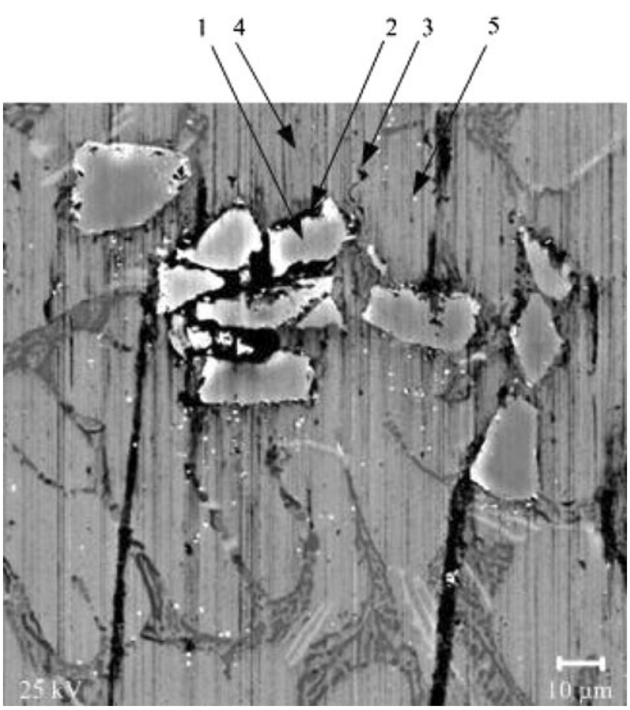

Fig. 1 SEM photograph of AlSi7Mg2-5 wt.% SiCp composite materials produced using a squeeze casting method (back scattered electron images)

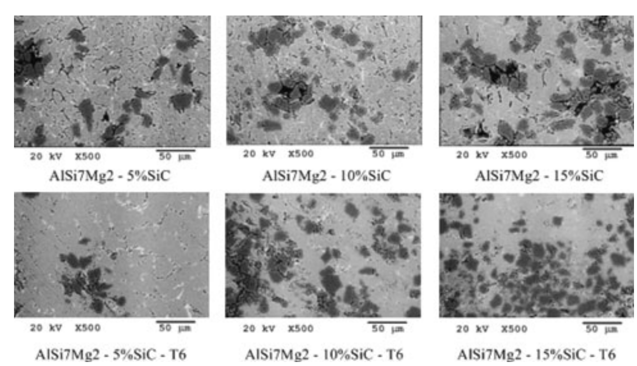

Fig. 2 SEM photographs of composite materials produced using a gravity casting method (back scattered electron images)

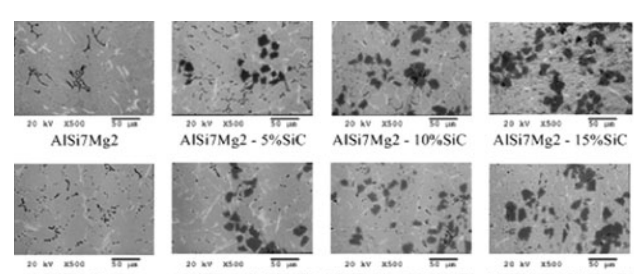

Fig. 3 SEM photographs of the composite materials produced using a squeeze casting method (back scattered electron images)

rich in iron (Ref 18). The porosity level was significantly reduced in the squeeze cast composite due to high squeeze pressure effects during solidification. This result is in agreement with previous work (Ref 17). This way, the low porosity levels lead to an increase in the tensile strength, the impact strength, and the hardness in contrast to composite materials produced using a gravity casting method. Despite the fact that Mg_2Si precipitates show regional clusters in the matrix alloy structure, they exhibit a regular distribution in the form of network in the composite materials. The increase in the SiC ratio from 5 to 15% causes an increase in the number of neighboring SiC particles. This leads to cracks in the SiC particles and even to fractures in some regions.

Mg_2Si precipitates without heat treatment locally clustered in the microstructure get rid of the sharp corners by diffusion and become spherical due to the heat treatment applied on the matrix material produced using a squeeze casting method.

The microstructure of the composites in the as-extruded conditions consists of equiaxed, recrystallized grains and of a relatively high fraction of precipitates, which are located both at the grain boundaries and in the grain interior as previously reported by Spigarelli et al. (Ref 19). It was shown in Fig. 4. The hot extrusion process leads to a redistribution of the particles in the matrix and favors the subsequent globularization of the structure in the semisolid state (Ref 20). Secondary shaping processes such as the hot extrusion bring the particle distribution into a more homogeneous state and improve the mechanical properties (Ref 20-22).

In the studies by Nguyen et al. and Tokaji et al. it was observed that particles were oriented along the extrusion direction at the end of the hot extrusion process (Ref 20, 23). When microstructures parallel to the extrusion direction were considered in this study, it was seen that the particles were oriented along the axis of the produced bar as a result of the extrusion process. In addition, due to differences in the dimensions that occurred because of particle fractures, different dimensions of the particles in the composite material led to the formation of bands; and the materials showed anisotropic properties (Fig. 5). Some SiC particles were broken during their movement from the die flat surface to the entrance edge of the die bearing. The broken SiC particles adhere to the product or die friction surfaces and scratch both deeply (Ref 24).

3.3 Density and Porosity

The density of the AlSi7Mg2 matrix alloy was calculated as 2.67 g/cm^3 using the rule of mixtures. In this study, we calculated the equivalences as the volume fraction of the SiC weight ratios in the composite materials as well as the theoretical densities according to these ratios. As a SiC content increases the relative density increases for the same compacting pressure (Ref 25). The composite material densities and the changes in porosity percentages are shown in Fig. 6.

The microstructure examination of the as-cast composites generally revealed that SiC particles were not distributed evenly in the matrix, regional clusters of particles exist and some pores are resolvable in the as-cast samples (Ref 26). In the composite materials produced using a gravity casting method, the porosity percentages increase with increasing the SiC ratio due to weakness of the bond at the matrix/reinforcement interface; the highest porosity percentage was determined to be 10.71 in the specimen containing 15% of SiC (Fig. 7). The applied heat treatment did not caused significant changes in the porosity.

The porosity level was significantly reduced in the squeeze cast composite because of high squeeze pressures. The formation of porosities between the SiC particles clustered together increased the porosity percentages as the SiC ratio increased, which is consistent with studies examined in the literature.

The porosity percentage in the composite materials shaped by an extrusion process increased in contrast to the squeeze cast composite materials. The change in the SiC ratio from 5 to 15%

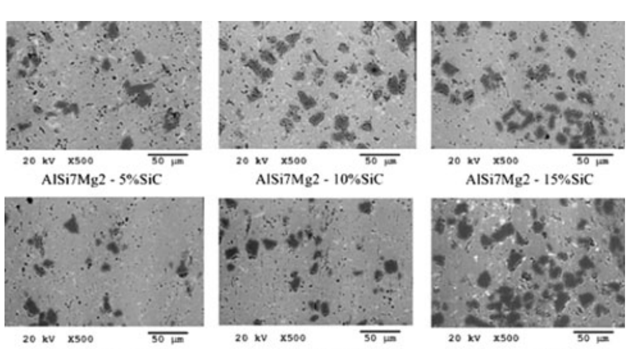

Fig. 4 SEM photographs of composite materials shaped by an extrusion process (back scattered electron images)

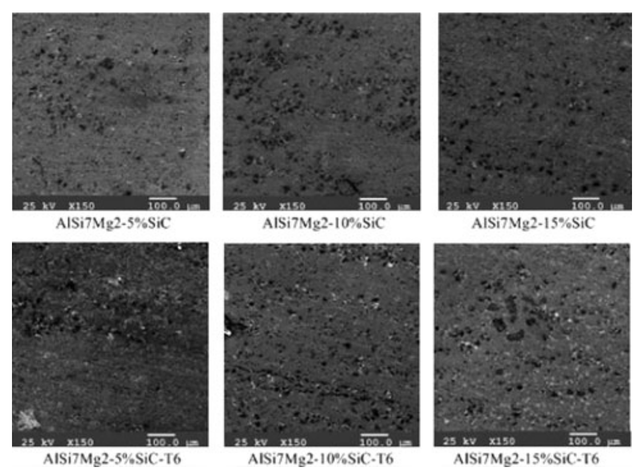

Fig. 5 SEM photographs of composite materials shaped by an extrusion process in sections parallel to the extrusion direction (back scattered electron images)

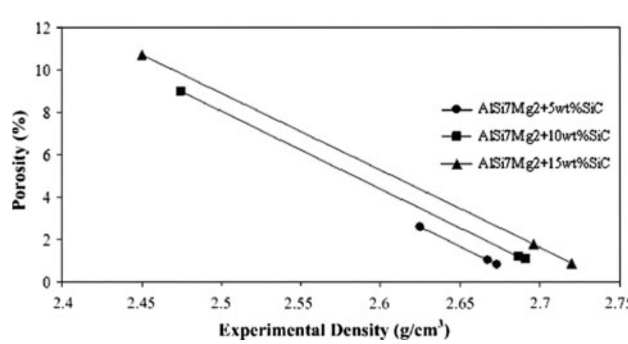

Fig. 6 Changes in porosity percentages and experimental density values

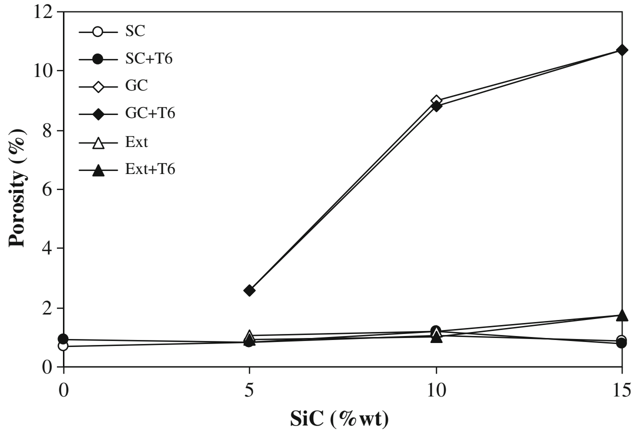

Fig. 7 Empirical relationship between the SiCp reinforcement and porosity percentages (*SC* squeeze casting, *GC* gravity casting, and *Ext* extrusion)

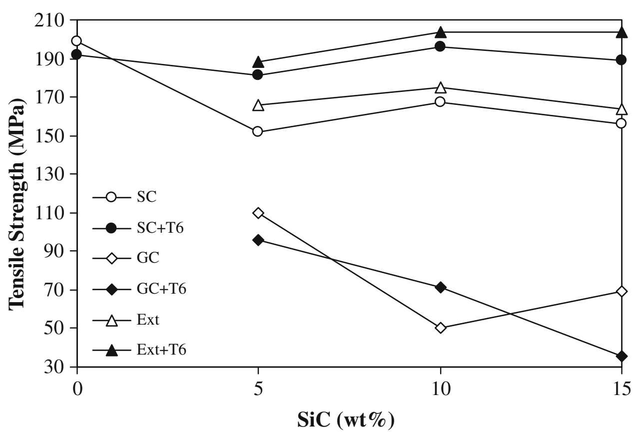

Fig. 8 Empirical relationship between the SiCp reinforcement and the tensile strength (*SC* squeeze casting, *GC* gravity casting, and *Ext* Extrusion)

caused a 65% increase in the structure porosity percentage. The application of a T6 heat treatment did not lead to a significant change in the porosity percentages.

3.4 Mechanical Properties

The tensile strength of the composite materials showed some variations as a function of the SiC particle ratio added to the matrix, the production methods, the hot extrusion process, and the T6 heat treatment. The effects of SiC reinforcement on the tensile strength are shown in Fig. 8.

The tensile strength of gravity cast specimens was low due to high porosity. The tensile strength of composite materials containing 5% of SiC decreased up to 55% when the SiC ratio was increased to 10% and further increased to 15%. The reason why the tensile strength was low for a 10% SiC ratio is assumed to be the high porosity in the region where the specimens were taken from.

The tensile strength of all composite materials produced using squeeze casting had a tendency to decrease compared to matrix alloys produced using the same method. This variation is due to the formation of inner defects, i.e., cluster, porosity,

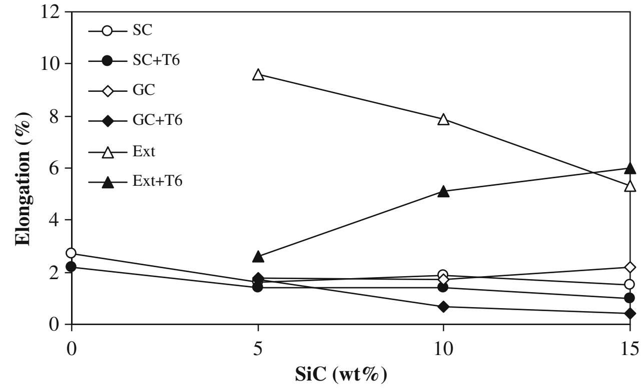

Fig. 9 Empirical relationship between the SiCp reinforcement and the elongation percentage (*SC* squeeze casting, *GC* gravity casting, and *Ext* extrusion)

and shrinkage, as reported by Seo and Kang (Ref 27). While the tensile strength of the matrix alloys was of 199 MPa, the tensile strength of the composite materials containing 5% of SiC became 24% lower and was equal to 152 MPa. This drop was of 16 and 22% in the composite materials containing 10 and 15% of SiC, respectively. In comparison to the composite materials produced by squeeze casting, it was observed that the tensile strength increased when the SiC ratio increased from 5 to 10%. When the ratio increased to 15%, the tensile strength decreased again due to particle fractures. Although there was a 4% decrease in the tensile strength of the heat treated matrix material, an increase was observed in the tensile strength of the composite materials. The reason is that intermetallic compounds become spherical after heat treatment. This pattern appeared in the tensile tests conducted on composite materials with SiC reinforced Al alloy by the Duralcan company (Ref 28). In this study, after applying a T6 heat treatment, the highest increase in tensile strength was of 21% and this occurred for the composite materials containing 15% of SiC.

The tensile strength in the extrusion direction has increased due to the particles orientation after the application of the extrusion process. Maximum tensile strength values up to ~175 MPa were recorded in the extrusion composite materials containing 10% of SiC. The composite material containing 15% of SiC had the lowest tensile strength due to particle fractures. An increase in the tensile strength of the heat treated specimens was observed. A tensile strength of 204 MPa was recorded in the composite materials with 10 and 15% SiC ratios.

An increase of the SiC ratio and the application of a heat treatment caused the elongation percentage of the specimens to decrease for the composite materials produced using a gravity casting method (Fig. 9).

An elongation percentage of 2.7% was observed in the matrix material produced by squeeze casting. When the SiC ratio increases from 5 to 10%, the elongation percentage value increases and then decreases at 15% of SiC due to particle fractures. A T6 heat treatment caused a decrease in the elongation percentage values.

In the specimens prepared from composite materials shaped by extrusion, higher values of the elongation percentage were obtained because of the particle orientation and the heat treatment processes applied before the extrusion. The elongation percentage of squeeze cast composite materials containing

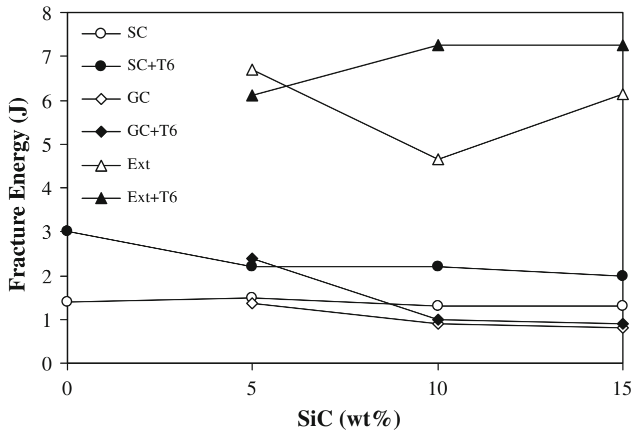

Fig. 10 Empirical relationship between the SiCp reinforcement and the fracture energy (*SC* squeeze casting, *GC* gravity casting, and *Ext* extrusion)

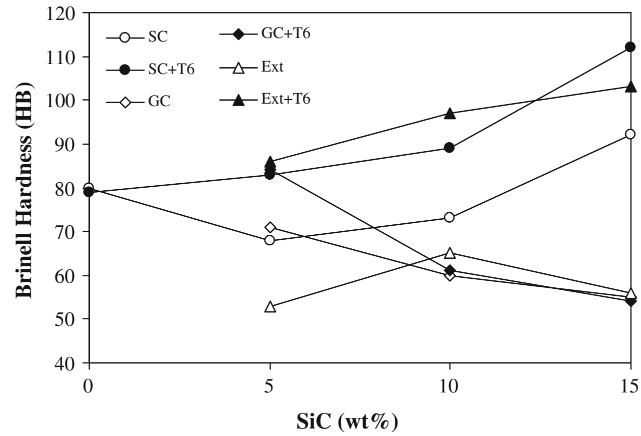

Fig. 11 Empirical relationship between the SiCp reinforcement and the hardness values (*SC* squeeze casting, *GC* gravity casting, and *Ext* extrusion)

5% of SiC shows an increase of up to 500%. For the extrusion specimens, a decrease in the elongation values is observed with increasing SiC ratios. Applying a T6 heat treatment leads to a decrease in the elongation percentages for 5 and 10% SiC and to an increase for a 15% SiC ratio in contrast to specimens without heat treatment, because the heat treatment was applied above the recrystallization temperature of the material.

The impact behavior of composites was affected by clustering of particles, particle cracking, and weak matrix-reinforcement bonding (Ref 29). Due to the high porosity of gravity cast specimens, the impact strength values are low (Fig. 10). The presence of hard SiC particles as a reinforcement in the ductile matrix of the aluminum alloys diminishes the impact toughness of the metal matrix composites (Ref 29). The lowest fracture energy of all composite materials was measured for the gravity composite materials containing 15% of SiC. As a result of the heat treatment, the fracture energy values increased. This increase was of 78% for the composite materials containing 5% of SiC.

The fracture energy of the matrix alloy produced by squeeze casting increased approximately of 7% in the composite materials with 5% of SiC reinforcement. However, as the SiC ratio was further increased, the impact strength value dropped again. The fracture energy values increased because intermetallic compounds became spherical after heat treatment.

The highest impact strength was reached by extrusion due to the homogeneous particle distribution and the recrystallized particle structure. The fracture energies were 6.7 and 4.65 J for the composite materials containing 5 and 10% of SiC, respectively. The fracture energy increased again to 6.15 J for 15% SiC composite materials. The impact strength of the 5% SiC composite materials dropped approximately of 9% after heat treatment. The impact strength of the 10 and 15% SiC composite materials reached a maximum value of 7.25 J.

In gravity cast specimens, the hardness dropped as the SiC ratio increased (Fig. 11). While the hardness of 5% SiC specimens was 71 HB, this value dropped by 15% to 60 HB in 10% SiC specimens. In specimens containing 15% of SiC, this value was 55 HB, which represents a decrease of 23%. After heat treatment, the measured hardness increased up to 18% for the 5% SiC composite materials, up to 2% for the 10% SiC composite materials and dropped approximately by 2% for the 15% SiC composite materials.

As a result of the hardness measurements of the squeeze cast specimens, the hardness of the matrix alloy was 80 HB. The hardness of the materials produced by addition of 5 and 10% of SiC were 68 and 73 HB, respectively. These hardness is lowered in comparison to the matrix material. However, the hardness of the 15% SiC composite materials increased by 15% compared to the matrix alloy and reached 92 HB. As the SiC ratio increased in the composite materials, the hardness increased (Ref 30, 31). As a result of the heat treatment, a 1% drop in the matrix alloy and an increase in the composite materials were recorded due to the spherical state of the intermetallic compounds, contrary to specimens without heat treatment.

If we consider the hardness of specimens shaped by extrusion, a decrease in the hardness is observed contrary to the case of squeeze cast specimens. When extrusion specimens were compared to each other and when SiC was increased from 5 to 10%, the hardness increased from 53 to 65 HB, which approximately represents an increase of 23%. However, for 15% SiC specimens, there was a decrease in the hardness due to particle fractures down to 56 HB. After applying a type T6 heat treatment, the hardness increased with increasing SiC ratios.

4. Conclusions

The following conclusions can be obtained from this work:

1. Due to the slow cooling rate in gravity casting methods, a non-homogeneous particle distribution increases in the structure. It was determined that the formation of porosity increased the matrix/reinforcement interface, especially in the sections where particles were clustered. The pressure applied during the squeeze casting method was an effective way to reduce the porosity. After the hot extrusion process, the particle structure became equiaxed; homogeneous and oriented along the extrusion direction.
2. The tensile strength decreased with increasing SiC ratios in the specimens prepared using a gravity casting method. The tensile strength of the composite materials produced by squeeze casting had a tendency to drop in contrast to

the matrix alloy. Increases in the SiC ratios caused changes in the tensile strength especially for low ratios. As a result of the hot extrusion process, the tensile strength increased. Hot extruded and squeezed cast specimens had similar pattern. The T6 heat treatment provided an increase of about 30-40 MPa in all SiC particles in the experimental specimens.

3. As the SiC ratio increased, a drop was recorded in the elongation percentages of the specimens prepared by gravity casting. It increased for squeeze cast specimens. The heat treatment reduced the elongation percentages and the extrusion process led to an increase.
4. The impact strength of the composite materials produced by squeeze casting decreased as the SiC ratio increased. The highest values were reached for the extrusion process. After applying a T6 heat treatment, the impact strength was increased.
5. In specimens shaped by extrusion, the hardness increased when SiC increased from 5 to 10%. However, the hardness of the 15% SiC specimens decreased again due to particle fractures. The hardness increased as a result of the T6 heat treatment.

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