Effect of Equal Channel Angular Pressing on the Microstructure and Mechanical Properties of Al6061-SiC\textsubscript{p} Composites

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Abstract. In the present study, Aluminium metal matrix composite with Al6061 matrix and SiC (10-30\textmu m) particulate reinforcement of varying composition (2-10wt. \%) were prepared by stir casting technique. Significant improvement in tensile strength and hardness was noticed as the wt. \% of SiC\textsubscript{p} increases in as cast Al6061- SiC\textsubscript{p} composites. The cast composites have been subjected to annealing treatment at a temperature of 400\degree C for 4 hours to homogenize the microstructure. The specimens have been prepared from these composites for Equal Channel Angular Pressing (ECAP). The ECAP process was carried out at room temperature using a die with channel angle of 120\degree and Bc route was adopted for successive passes. The effect of ECAP on the microstructure and mechanical properties of Al6061 -SiC\textsubscript{p} composite is evaluated. After ECAP process, the size and distribution of the reinforcement particles are not changed but there is a significant reduction in the grain size of the matrix alloy was observed. The hardness and tension tests were conducted at room temperature as per ASTM standards. The results were compared with the base Al6061 material and as cast Al6061-SiC\textsubscript{p} composites. There is a significant improvement in the hardness and the Ultimate tensile strength of ECAP processed composites.

Key words: -Mechanical properties, stir casting, Metal matrix composites, ECAP, Al6061, Silicon carbide.

1. Introduction

In recent years, the global need for light weight, high performance and low cost materials has caused a shift in research from monolithic materials to composite materials. Amongst different kinds of the recently developed composites, in particular Aluminium base particle reinforced MMC’s are gaining wide spread acceptance due to their light weight, high specific strength, high specific stiffness and superior wear resistance, hence finds applications in strategic sectors aerospace to general engineering industries [1-4]. Among the series of aluminium alloys, Al6061 is better choice as matrix material owing to its excellent mechanical properties combined with good formability and option for modification of the strength of composites by heat treatment [5]. Reinforcement of aluminium alloy with SiC particulates has generally been observed to improve the yield strength, ultimate tensile strength and wear behavior of the composite material [6-8]. Number of processing methods have been developed for the manufacture of particulate/whisker/short fiber reinforced composites. Stir casting method is found to be easier to handle during fabrication and the low cost production method, particularly with discontinuous reinforcements [9-11]. Severe plastic deformation (SPD) is a family of metal forming techniques used to refine the grain size to submicron or even nanometer size [12]. Amongst various SPD process, Equal Channel Angular Pressing (ECAP) is a novel technique for producing ultra fine grain structure in materials by imposing intense shear strain into the materials by pressing the sample through a special die without incurring any reduction in the cross sectional area of the work piece [13-15]. These materials have great impact in biomedical, electronics, military, aerospace, and automotive industries. Most investigations on ECAP have concentrated on pure metals and metallic alloys [16-19]. Horita et.al[16] showed that ECAP of several Aluminium alloys (1100, 2024, 3004, 5083, 6061, 7075) results in significant improvement in 0.2\% proof stress, hardness and ultimate tensile strength along with substantial grain refinement. Unlike in case of metals and alloys, not much work has been carried out in the case of MMC’s. Li and Langdon [20] reported that ECAP of
Al6061-10vol% Al$_2$O$_3$ composites increases the microhardness and ductility. It was increased to ~235% subjected to a strain of ~5, the grain size obtained was ~1 µm. Ma et al [21] investigated ECAP of SiC$_p$ reinforced Aluminium based composites and found that there was a decrease in average length of whiskers from 42 µm to 4.2µm and also homogeneous distribution of SiC$_w$ was observed. Saravanan et al [22] concluded that ECAP of Aluminium-Graphite composites results in two fold increase in hardness and tensile strength of the composite than the as cast composite. In addition, the grain size achieved was 300nm at room temperature. Ramu and Bauri [23] found that ECAP of Al-SiC$_p$ composite, refines the grain size of the matrix material and there is no change in the particle distribution after ECAP. The hardness and compressive strength increases significantly by ECAP.

A meger work has been carried out on ECAP of composites. The main objective of the present investigation is to prepare Al-SiC composite by stir casting technique and the prepared samples were subjected to ECAP. The effect of ECAP on the microstructure and mechanical properties of Al-SiC composite will be evaluated. The results are compared with the base Al6061 material and as cast Al-SiC composites.

2. Materials and Methods

2.1 Materials Selection

The matrix material for the research study selected was Aluminium 6061. The reinforcing material selected was SiC particulates of 10 to 30µm size. The chemical composition of Al6061 alloy by weight percent Si = 0.72, Mg = 0.89, Cu = 0.21, Fe = 0.23, Cr = 0.22, Zn = 0.10, Ti = 0.01 and Al = Balance. The amount of SiC particulates in the matrix were varied from 2 to 10wt. %.

2.2 Preparation of Composites

Stir casting technique has been used to prepare the Al6061-SiC composite (figure 1). The precast Al6061 alloy ingots were charged and melted to a temperature of 750°C using electric resistance furnace. The molten metal was degassed by passing pure nitrogen for 3-5 minutes. The SiC particles of 10-30µm size were preheated at 600°C for a soaking period of 2 hours to improve wettability. The extent of incorporation of SiC in the matrix alloy was varied from 2-10wt% in steps of 2wt%. The SiC particles were introduced into the degassed vortex of the molten matrix material Al6061 alloy maintained at 730°C. The reinforcements were uniformly distributed in the matrix by stirring using zirconium coated steel impeller at a speed of 300-400 rpm for 10 minutes. The molten composite was poured into the steel mould and castings were obtained in the form of round rods of 15 mm diameter x 150 mm length.

2.3 Equal Channel Angular Pressing

The samples of diameter 10mm and length 80 mm were machined from the cast composite billets for ECAP. Prior to ECAP, the surface of the samples were polished using SiC abrasive papers with grades of 600, 800 and 1000 to reduce the friction between the sample and the die wall. The samples are annealed at temperature of 400°C for 4 hours to homogenize the microstructure. The annealed composite samples were subjected to ECAP using a die with two channels having an equal circular cross section with diameter of 10 mm. The two channels of the die intersect at angle of $\phi = 120^\circ$ and $\psi = 0^\circ$ (figure 2). The theoretical effective strain according to the die geometry is given by the equation as formulated by Iwahashi et al [19].

$$\epsilon_N = \sqrt{3}\left[2\cot\left(\frac{\phi}{2}\right) + \psi\csc\left(\frac{\phi}{2}\right)\right]$$  \hspace{1cm} (1)

These die angles introduce a strain of ~ 0.7 on each pass of sample through the die. In order to minimize the friction between the die walls and the sample, the MoS$_2$ lubricant was used. B$_C$ route was adopted in the present work where the sample is rotated by 90° in one direction either clockwise or anticlockwise after each successive pass. ECAP process was carried out at room temperature. The ECAP experiments were repeated three times to verify the reproducibility of the results. The samples were taken for characterization after each pass of ECAP process (figure 3).
3. Characterization of composites

3.1 Microstructure
The samples of 10mm diameter and 10mm thick were cut from the specimens. These samples were polished with SiC abrasive papers up to 1000 grit size followed by polishing with Al₂O₃ paste on velvet cloth. Finally, the samples were polished with diamond paste and etched by Keller’s etchant. The polished samples were examined using metallurgical microscope to obtain micro photographs.

3.2 Hardness
The micro hardness of as cast and the ECAP processed composites are measured using Vickers’s hardness test with a load of 1kg for 15 seconds on polished samples of 10mm diameter and 10mm thick. An average of six indentations was considered on each sample for data presentation.

3.3 Tension Test
The tensile test was conducted as per ASTM E8M standard using computerized Tensometer. For each composite, three tensile test specimens were tested and an average value ultimate tensile strength and ductility (% elongation) were measured.
4. Results and Discussion

4.1 Microstructural Studies of as cast and ECAP processed composites

Figure 4 presents the microphotograph of as cast Al6061 alloy and their composites. From the microstructure photographs, it can be observed that, the distributions of reinforcements in the matrix are uniform. Figure 5 presents SEM micrographs of ECAP processed Al6061 alloy and their composites. The average grain size of as cast Al6061 alloy was observed to be 54µm. After first ECAP pass the grain size was reduced to 21µm (figure 5a). After second ECAP pass the grain size was reduced to 3µm (4 pass). Figure 5(b), (c) and (d) presents the microstructure of Al6061-2wt.%SiC, Al6061-4wt.%SiC and Al6061-8wt.%SiC composites, after 1\textsuperscript{st} ECAP pass. The grain size achieved was observed to be 18µm. There was no change in the size and distribution of reinforcement particles after ECAP process. After 3\textsuperscript{rd} ECAP pass, the grain size of Al6061-2wt. %SiC composite was reducing to 6µm. The grain size achieved for Al6061-4wt. %SiC after 2 ECAP passes was 9µm. Similar observations were made for Al6061-6wt. %SiC composites. For Al6061-8wt. %SiC and Al6061-10wt. %SiC composites only one ECAP passes was possible, the grain size achieved was 18µm. Further ECAP pass resulted in the formation of cracks (figure 3c) on top surface of the specimen (inner radius). This is due to higher strain rate sensitivity of composite material than the base metal due to ECAP process and due to non-uniform stress distribution across the sample [24, 25]. In addition, as the weight percentage of SiC particles increases, the hardness of the material increases which results in the loss of ductility that leads to the formation of cracks on samples. Similar observations are reported by other researcher [23].
4.2 EDAX Analysis

The interfacial bonding between SiC and the matrix was also observed. There were no interfacial reactions between the matrix and reinforcements and the bonding between them is good. The figure 6 shows the EDAX taken on the interface of matrix and reinforcements confirms this.
4.3 Hardness of as cast and ECAP processed Al6061 and their composites

The Vickers hardness of the cast base material Al6061 and their composites containing 2-10wt % SiC reinforced Al6061 composite were evaluated in the as cast and after ECAP process (figure 7). The hardness of the studied composites is higher than that of the base material in the as cast condition. The hardness of the composites increases with the increasing SiC content into Al6061-SiC composites. This improvement in the hardness of the composites is mainly attributed to the high hardness of the SiC particles. Further, the hardness of the composites was improved after ECAP process, compared to as cast composites. This is due to the fact that, the ECAP results in the formation of fine grain structure of the matrix alloy in the composites, also the material become strain hardened due to ECAP process. The as cast Alloy Al6061 exhibits hardness of 45. After the material was subjected to first pass of the ECAP, hardness value was increased to 69 with an increase of 51%. Further ECAP passes increases the hardness to 1.74 times the as cast alloy after 4 ECAP passes. The hardness of Al6061-2wt% SiC composite increases to 1.71 times as cast composite after 3 passes of ECAP. Only three ECAP passes are possible for this composite. Further ECAP passes results in the formation of cracks in the specimen. Similar observations were obtained for Al6061-4wt% SiC composites. The highest value of the hardness (90) was obtained for 6wt. % of SiC in the studied composite, but restricted the number of passes to only two. This is due to the hard nature of the composites. Nevertheless, further addition of SiC (10wt. %) in to the composites, the hardness of the composites almost remained constant and restricted the pass to only one. This may be due to the clustering of SiC particles at some regions in the composites results in the formation of cracks with further ECAP process.

Figure 7: Effect of reinforcements and ECAP process on Vickers Micro hardness of Al6061 and Al6061-(2 -10wt. %) SiC composites.
4.3 Tensile behaviour of as cast and ECAP processed Al6061 and their composites

The Ultimate Tensile Strength (UTS) of base material Al6061 and Al6061-SiC composites in as cast condition and after ECAP pass were studied and plotted as in figure 8 (a). In as cast condition, the composites exhibit higher strength than the base material this is due to the presence of hard SiC particles. These particles acting as barriers to dislocations, thereby providing enhanced resistance to tensile stresses. In addition, increased amount of reinforcements in the matrix alloy leads to increase in dislocation density during solidification due to thermal mismatch of the matrix alloy and reinforcements [26]. The UTS of the composites increases considerably after each ECAP pass. It was well known that ECAP process had profound effect in refining the grain size of the composites, which in turn leads to significant increase in the strength of the composites [27]. It is in accordance with the Hall-petch equation. The ultimate tensile strength of matrix alloy Al6061 in as cast condition was 120 N/mm². After the first ECAP pass, it is increased to 186 N/mm² with 55% increase. Further increase in number of passes, increased the UTS of the matrix alloy to 1.93 times the as cast alloy. Addition of 2wt. %SiC particles into Al6061 matrix, the strength increases to 1.18 times compare to base alloy in as cast condition. After ECAP the UTS increases by 1.66 times after 3 ECAP passes. The highest value of UTS is obtained for 6wt. %SiC in the studied composites with 1.35 times increase in the UTS compare to as cast condition. Further addition of SiC particles into Al6061- composites, decreases the strength of the composites. This is attributed to increase in hardness of composite due to the addition of SiC particles into the matrix [23].

Figure 8(b) gives the effect of reinforcement content on the ductility of composites in terms of percentage elongation in as cast and after ECAP process. The ductility of as cast matrix alloy Al6061 is 12.6%, after the first ECAP pass, the ductility decreases by 8%. Further ECAP passes decreases the ductility of the base material but after 3 passes it almost remains same, similar observations are reported by other researchers [11]. The ductility of Al6061-SiC composites decreases with the increase in addition of SiC particulates into the matrix in the as cast condition. After ECAP process the loss in ductility of the composites is less due to formation of equiaxed grains, also grain boundaries evolve into arrays of high angle boundaries resulting in better ductility of the composite than the heat-treated composites [14, 28].
4.4 Fractography

**Figure 9**: SEM photographs of fractured surfaces of ECAP processed Al6061 and their composites:

- a) Al6061 matrix alloy
- b) Al6061-2wt.%SiC
- c) Al6061-6wt.%SiC
- d) Al6061-10wt.%SiC.

The fracture surface of the samples after tensile testing was observed under SEM. From SEM picture 9(a), it was observed that larger number of small dimples is associated with ductile failure of Al6061 alloy after ECAP process. Particle reinforced composites fail by a mixed mode and gives rise to a bimodal distribution of dimples. As it can be seen from figure 9(b), it was observed that larger dimples are associated with SiC particles, whereas smaller dimples are associated with ductile failure of the matrix. The fracture surface of Al6061 – 6wt. % SiC after three pass of the ECAP process, the cracks are initiated between the matrix and the reinforcement which are clearly visible in the figure 9(c). Figure 9(d) exhibits that particle de-bonding has taken place at matrix- particle interface after second ECAP passes, this may be due to high shear strains generated during ECAP process and also the higher wt.% of SiC (10wt.%) Particles causes strain hardening during plastic deformation, which results in the formation of cracks [29].
Conclusions

The present investigation exhibits that:

1. Al6061-(2-10wt. %) SiCp composites were successfully produced by stir casting route. Castings were free from porosity and clustering with fairly uniform distribution of SiC particulates in the matrix.

2. The effect of the SiC reinforcement on Al6061-SiC composites is appreciable in terms of the ultimate tensile strength and the hardness in the as cast condition.

3. ECAP of Al6061-2wt. % of SiC composite was successfully carried out up to three passes. Where as in case of Al6061- 4wt. % of SiC and Al6061-6wt. % of SiC composites only two ECAP passes are possible. Further addition of SiC particles, reduces the number of passes due to increase in the hardness of the composites results in the formation of cracks at the surface.

4. After ECAP process, the size and distribution of the reinforcement particles are not changed but significant reduction in the grain size of the matrix alloy was observed.

5. The maximum value of the ultimate tensile strength and Hardness through ECAP Process was exhibited by 6wt. % of SiC among the studied composites.

6. There is no significant change in the ductility of the studied composites after ECAP process.

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