

## The Effect of ECAP on the Al6061 Matrix and Al-SiC<sub>p</sub> at Elevated Temperature

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**Abstract:** In the present study, stir casting method (SCM) was used to produce metal matrix composites (MMC). Al6061 and silicon carbide particles (F500≈15 μm) were selected as matrix and reinforcement materials respectively. Matrix, Al-5%SiC<sub>p</sub> and Al-7.5%SiC<sub>p</sub> were subjected to equal channel angular pressing (ECAP) to 8, 6, and 4 passes, respectively, of a Bc route, at temperature 300°C. Optical microscope was used to determine the SiC particles distribution in the Al matrix of the composites (as-cast and after the ECAP process). Improvement in SiC distribution for Al-5%SiC and Al-7.5%SiC, and agglomeration decreased by 80.5% after 6<sup>th</sup> pass and 80.2% after 4<sup>th</sup> pass, respectively. The densities of the composites, as-cast and after the ECAP process, were also measured. The porosity percentage for Al-5%SiC and Al-7.5%SiC has decreased by 63% after 6<sup>th</sup> pass, and 48.6% after 4<sup>th</sup> pass, respectively, compared to the as cast samples. Tensile and hardness tests was used to evaluate the mechanical properties of the composites. Results show that, the peak values of ultimate tensile strength (UTS) and hardness are obtained after the 2<sup>nd</sup> pass. Where, the UTS of Al6061 matrix, Al-5%SiC and Al-7.5%SiC has increased by 51.03%, 49.13%, and 168.4%, respectively, compared to the as cast samples.

### 1. Introduction

Metal matrix composites have been an attracting interest of many researches due to their improved mechanical properties. This makes it suitable to be used in many applications, such as, aerospace, automotive, and sports equipment industries[1–3]. Stir casting, powder metallurgy, metal injection molding and squeeze casting etc. have been developed for the manufacture of particle, whisker, and short fiber-reinforced composites[4 – 6]. Stir casting technique is currently one of the simplest and most economical fabrication routes for manufacturing particle-reinforced metal matrix composites[4, 7]. However, the casting process has two major limitations: first, reinforcing particles tend to sink or float, depending on their density relative to the liquid metal, hence, the dispersion of ceramic particles is non-uniform; Second, the ceramic particles are generally poorly wetted by the liquid metal matrix resulting in a poor bonding force between the matrix and reinforcement[8, 9]. The previously mentioned casting problems appear significantly in the processing of fine reinforcement particles[10]. Kumar et al. [11], studied the mechanical properties of Al6061-Glass particulate composite, with particle size 65μm. It was found that, up to 9% Glass particulate, the UTS increase. Rahman et al.[12] studied the mechanical properties of Al-SiC composite. The SiC particles content and size was varying from 0% to 20% and from 53 to 74 μm, respectively. It was found that, up to 10%SiC by weight, the UTS increases, beyond this weight percentage the UTS decrease. They reported that the decrease of UTS is due to the effect of porosity and agglomeration of SiC particles. Balamurugan et al. [13] studied the mechanical behavior of

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percentage Al-SiC composites prepared by SCM, using Al2024 and SiC (320-grit) with weight 10% and 15%SiC, and found that the strength decreases from 270.9 MPa in case of 10%wt Al-SiC to 228.6MPa in case of 15%wt Al-SiC, and the hardness increases from 48 to 54 in case of 10% and 15% respectively.

Severe plastic deformation (SPD) is a useful processing tool to refine the grain size to the submicron or even nanometer size. Various techniques have been developed to obtain ultra-fine grain structure materials such as high pressure torsion (HPT), accumulated rolling (AR), and equal channel angular pressing (ECAP)[14, 15]. Among all SPD methods, the ECAP process has proved to be attractive to many researchers, because the procedure may be applied repeatedly to billets without causing any change in its cross sectional geometry[4, 16–18]. Limited number of reports are available on the application of ECAP at elevated temperatures to metal matrix composites. Sabirov et al.[19] studied the effect of ECAP at 370°C on Al6061-20%Al<sub>2</sub>O<sub>3</sub> composite prepared by the powder metallurgy technique. The results showed that, the UTS increased after the 4<sup>th</sup> pass compared to the as-cast composite from 225 to 315 MPa, while it has decreased after the 7<sup>th</sup> pass to reach 306 MPa. Kim et al. [20] studied the hardness of Al6061 using the equal-channel angular pressing, at different temperatures. It was found that the hardness, after the 8<sup>th</sup> pass at 553K, decreases relative to the as received specimen. This was attributed to the dynamic recovery accelerated in ultrafine grained microstructure.

The aim of the present work is to investigate the effect of ECAP process, at different passes, on the homogeneity of SiC particle distribution and mechanical properties of fabricated Al6061-SiC<sub>p</sub> composite. Stir casting technique is used to disperse the reinforcement material through the matrix molten metal.

## 2. Materials and Experimental Work

### 2.1 Material

Al 6061, of chemical composition shown in **Table 1**, was used as the matrix of the composite material prepared. While, a SiC powder of size F500 (5 to 25 micron), and a theoretical density of 3.1 g/cm<sup>3</sup>, was used as the reinforcement. The Al-SiC composites were prepared with 5% and 7.5% SiC volume fraction.

**Table 1 Chemical Composition of Al6061**

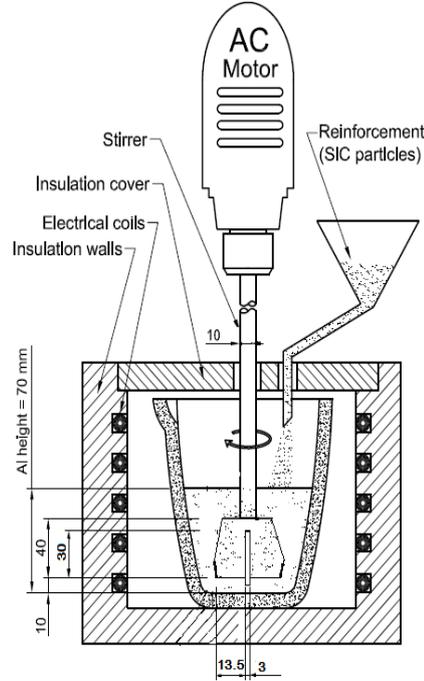
Element	Si	Fe	Mn	Mg	Cu	Cr	Zn	Ti	Al
Weight (%)	0.52	0.025	0.005	0.871	0.259	0.2	0.008	0.04	98.08
Standard values % [21]	0.4-0.8	Max. 0.7	Max. 0.15	0.8-1.2	0.15-0.4	0.04-0.35	Max. 0.25	Max. 0.15	Balance

### 2.2 Experimental work

#### 2.2.1 Stir casting process

The SiC was preheated at 800°C for two hours in the resistance furnace to improve the wetness properties by removing absorbed hydroxide and other gases[3, 22]. Al alloy in the form of extruded sections was melted at 750°C in graphite crucible placed inside the resistance furnace as shown in Fig. 1. The molten metal was stirred using a stirrer, composed of a steel rod coated with clay of water glass and graphite. The stirrer rotated at a constant angular velocity of 200 rpm to smoothly stir the Al matrix. When the vortex appears, the preheated SiC particles were added to the melted Al. The SiC particles are added uniformly with a rate of 6 g/min to the molten matrix[3, 23]. The stirring process continued 5 minutes after adding the particles, in order to ensure uniform distribution of the reinforcement

particles [23]. During the stirring process, a 1wt% of Mg was added to the melt to improve wettability, reduce porosity and develop adequate bonding between Al and SiC<sub>p</sub>[12, 24]. After stirring, the composite slurry was then reheated to a fully liquid state and poured into a preheated steel mold of five cavities each of dimension 16 mm diameter and ≈120 mm long. The mold was left to cool, and castings were then ejected and prepared to the ECAP process.



**Fig. 1 the dimensions of crucible and stirrer used in Al-SiC composites**

### 2.2.2 The ECAP Process

Specimens from the matrix, Al-5%SiC and Al-7.5%SiC materials were machined to diameter Ø15mm and length 85mm. Figure 2 shows the ECAP die designed for the present investigation. The die blocks are made of hot work tool steel X40CrMoV5-1, which has excellent wear resistance and hot toughness.

The ECAP die was designed with two channels, the angle between the channels was  $\phi = 90^\circ$  and the outer arc of curvature was  $\psi = 90^\circ$ . The billets were processed at a pressing rate of 5 mm/sec, at 300°C using a ram attached to a hydraulic press of 100 ton capacity. The die and sample were heated using an induction furnace, as shown in Fig. 2(c). The temperature was controlled using a thermocouple placed near the channel wall with accuracy of  $\pm 5^\circ\text{C}$ . All the pressing passes were conducted using a Bc route where the billet is rotated after each pass by  $90^\circ$  in the same direction. The billets were greased with *MOLYKOTE* as a lubricant to minimize the friction between the billets and the die walls.

The equivalent strain  $\varepsilon_N$ , after N passes, can be expressed by following equation:[25, 26]

$$\varepsilon_N = \frac{N}{\sqrt{3}} \left[ 2 \cot \left( \frac{\phi}{2} + \frac{\psi}{2} \right) + \psi \operatorname{cosec} \left( \frac{\phi}{2} + \frac{\psi}{2} \right) \right] \quad \text{Equation 1}$$

At  $\phi = 90^\circ$  and  $\psi = 90^\circ$  the strain value equal 1.15 after first pass.

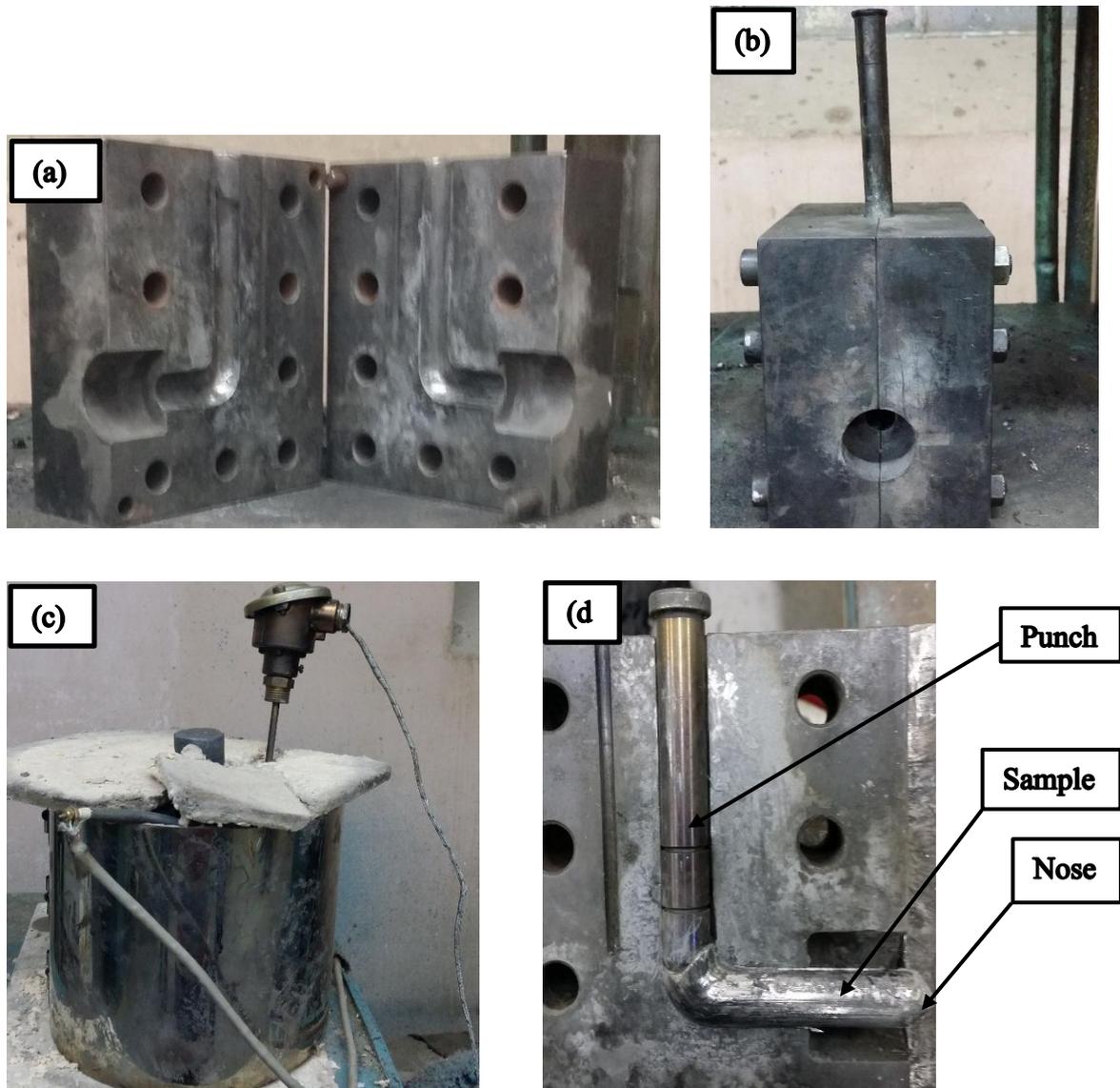
### 2.3 Density Measurement

Samples from the as-cast and ECAPed composites were cut for the density measurements. The actual density ( $\rho_{act}$ ) of the composite were measured using Archimedes method by weighing samples in air and in distilled water. On the other hand, theoretical density ( $\rho_{theo}$ ) of the composite samples was calculated based on the simple mixture rule as follows[4, 27–29]:

$$\rho_{composite} = v\rho_{(matrix)} + v\rho_{(reinforcement)} \quad \text{Equation 2}$$

where  $\rho$ , and  $v$  are the density and the volume fraction of each constituent, respectively. The actual density of Al6061 and SiC powders were 2.7 and 3.2g/cm<sup>3</sup>, respectively. The porosity was evaluated by comparing the density with that of the theoretical density given by Eq.3.

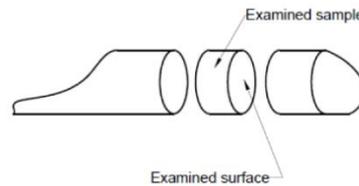
$$porosity\% = \frac{\rho_{theo} - \rho_{act}}{\rho_{theo}} * 100 \quad \text{Equation 3}$$



**Fig. 2. The die used in the present investigation: (a) Opened, (b) closed, (c) The ECAP die during heating inside the furnace, (d) shape of the sample after the ECAP process.**

## 2.4 Microstructure

Sliced samples of composites, as shown in Figure 3, were grinded with emery paper up to 2000 grit size, followed by polishing with  $\text{Al}_2\text{O}_3$  suspension on velvet cloth. An optical microscope was used to observe the microstructure, and the particle distribution.



**Figure 3 Drawing of examined specimen for microstructure characterization**

## 2.5 Mechanical Properties

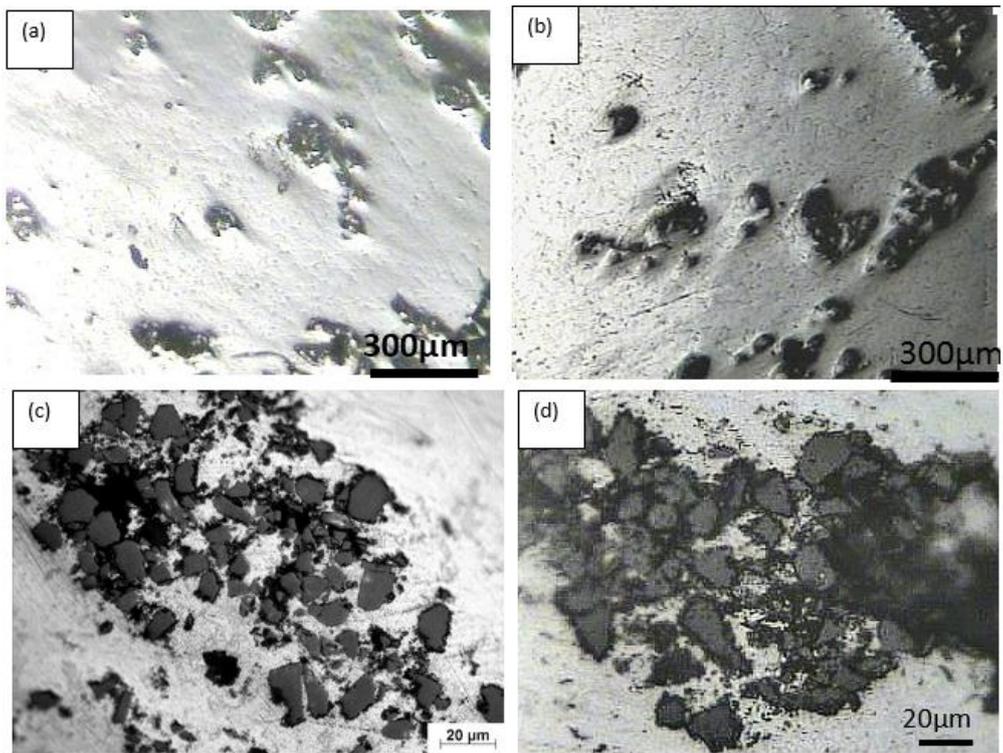
The mechanical properties were assessed by tensile and hardness tests results. The tensile test samples were prepared according to ASTM E8M, where the overall length is  $\approx 65\text{mm}$ , tension specimen gauge length is 20 mm and gauge diameter 4 mm, using a tensile testing machine, at room temperature with strain rate of  $5 \times 10^{-4} \text{ s}^{-1}$ , using a Lloyd-LR300 universal testing machine with capacity 30 ton. While the hardness test was carried out for the matrix and composite samples, before and after ECAP, it was evaluated using a micro-Vickers hardness tester model Mitutoyo. Prior to testing, the surface of the samples were grinded and polished to obtain a flat and smooth surface finish. A direct load of 1kg was then applied on the specimens for 10 seconds.

## 3. Results and Discussions

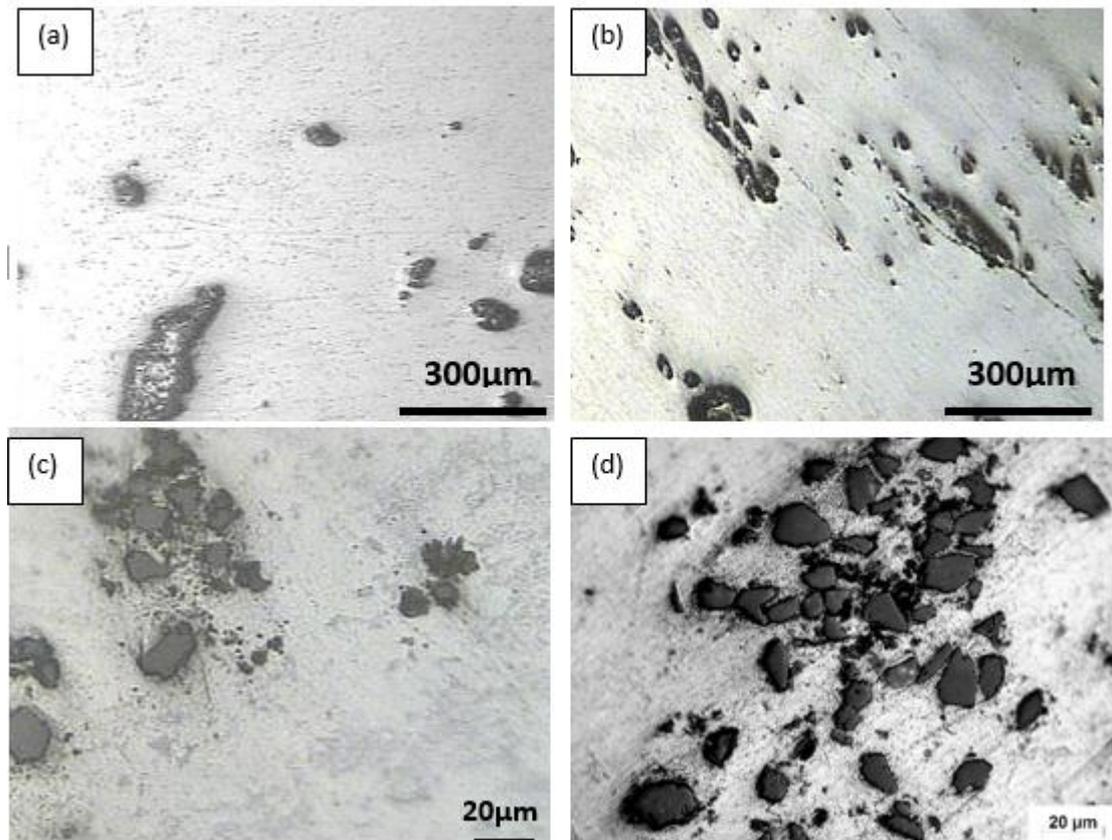
### 3.1 Microstructure

The optical micrographs of Al-5%SiC and Al-7.5%SiC as-cast composites. Show agglomerations, non-uniform particle distribution, and porosity in both composites. Figure 4 Agglomeration can be as a result of interaction between  $\text{SiC}_p$  which leads to clustering of the particles[30], which is accumulated due the presence of the fine particles. These particles tend to agglomerate more as their size decrease[24]. The porosity results from sucking of the air bubbles into the melt via the vortex created during the stirring process which is then entrapped in the particles agglomeration cites as previously observed [2–4, 24] Particles agglomeration and porosity were appearing to be more pronounced in the composite containing 7.5% SiC.

Figure 5(a, c) shows microstructure for Al-5% $\text{SiC}_p$  (a, c) and Al-7.5% $\text{SiC}_p$  (b, d) composites after the fourth pass, where the distribution of reinforcements in the matrix alloy indicate homogeneity. The porosity and agglomeration are overcome with the increase of ECAP passes, which is obvious in the microstructure of Figure 4. The samples seem to have a more uniform distribution of the SiC reinforcement than the samples of as-cast composites. These porosities and agglomeration are decreased by increasing the ECAP passes. Applying ECAP makes the particles in the agglomerations areas convert from circles to elliptical shapes which are coaxial with axis of extrusion. The most of particles in agglomerated area were broken. This means that as the concentration of reinforcement particles increases in a specific area. This mean that the agglomeration decreased with increase the ECAP pass.

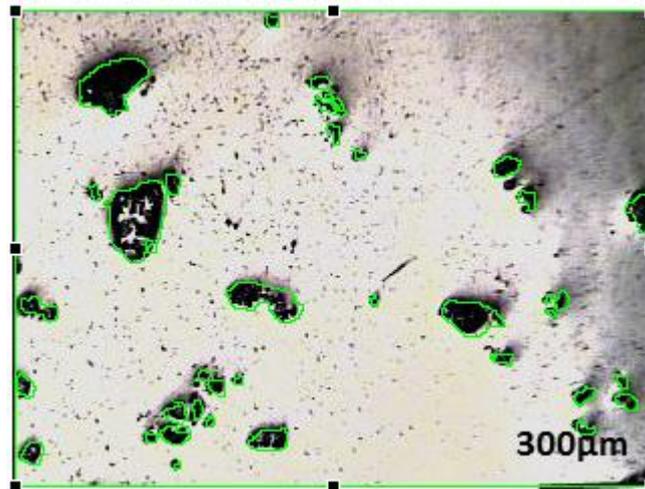


**Figure 4 (a, b) general view illustrating the distribution of SiC particles (X40) for as-cast 5%SiC and 7.5%SiC, respectively.(c, d) agglomeration detail in (X500)**

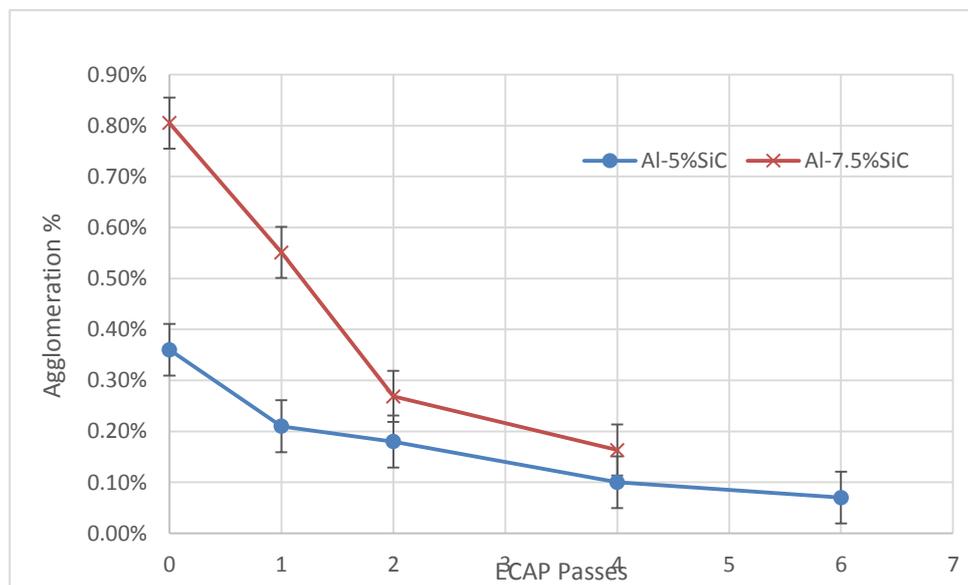


**Figure 5 Microstructure of composites Al-5%SiC (a, c) and Al-7.5%SiC (b, d) after 4<sup>th</sup> pass.**

The agglomerations percentage after each pass was assessed by area fraction from the microstructure micrographs by JMicrovision software as shown in Figure 6 , and the data are plotted in Figure 7 . The figure shows the decrease of agglomeration by increasing the ECAP passes. The agglomeration percentage for as-cast Al-7.5%SiC<sub>p</sub> was very high compared to that of Al-5%SiC<sub>p</sub>. The agglomeration percentage decreased after the fourth pass by 72.2% and 80.2% for Al-5%SiC<sub>p</sub>, and Al-7.5%SiC<sub>p</sub> respectively.



**Figure 6** analyzed picture by JMicrovision for Al-5%SiC after the 1<sup>st</sup> pass



**Figure 7** the agglomeration percentage Vs the ECAP passes for Al-5%SiC<sub>p</sub> and Al-7.5%SiC<sub>p</sub> composites

### 3.2 Density and porosity measurements

Table 2 lists theoretical and actual density values, as well as, the porosity percentage, calculated using Equation 3, of Al-5%SiC, Al-7.5%SiC, and Al6061 matrix. Table 2 indicate that actual densities values are lower than the theoretical density for all compositions, which can be attributed to the presence of porosities. The porosity ranging between 2.218% and 3.927%. It is noticed that the presence of SiC<sub>p</sub> increases the porosity in the composite. The

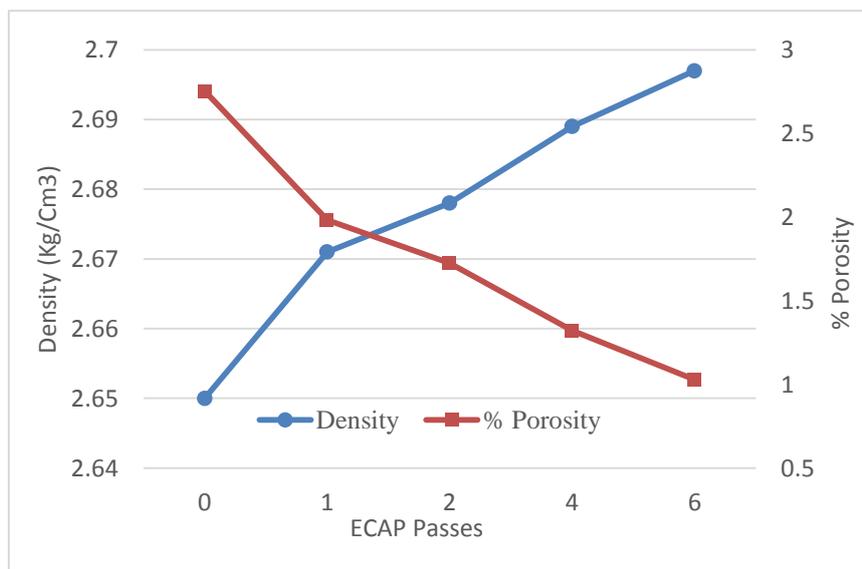
porosity in Al-5%SiC<sub>p</sub> is less compared to the Al-7.5SiC<sub>p</sub> composite. This can be attributed to the higher SiC content in Al-7.5SiC<sub>p</sub> composite which in turn increased the stirring time during the composite preparation, allowing more air bubbles to enter the melt, as reported by many researchers[3, 30], 31]. Furthermore, the maximum value of porosity is 3.927% at Al-7.5% SiC composite. This value lies in the acceptable range, from 2% to 4%, of porosity percentage [3, 31–33].

**Table 2 Comparison of the theoretical and the experimental densities**

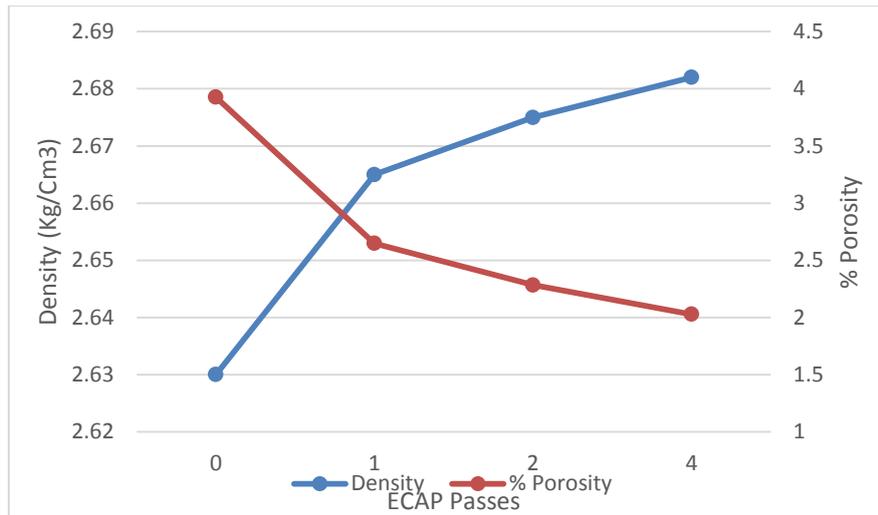
	Theoretical density (g/cc)	Actual density (g/cc)	Porosity (%)
Al6061	2.7	2.641	2.218
Al-5%SiC <sub>p</sub>	2.725	2.65	2.752
Al-7.5%SiC <sub>p</sub>	2.7375	2.63	3.927

After ECAP the densities and porosity of ECAPed two composites are different. So that, by increasing the number of ECAP passes, the density increases and the porosity decreases, Figure 8, and Figure 9. The maximum density, and minimum porosity values were 2.697g/cm<sup>3</sup> and 1.02%, respectively, after 6 passes in Al-5% SiC composite, while for Al-7.5%SiC<sub>p</sub> the maximum density, and minimum porosity values were obtained 2.682 g/cm<sup>3</sup>, and 2.027%, after 4<sup>th</sup> pass, respectively.

The maximum increasing rate of density and the maximum decreasing rate of porosity were achieved after the first pass. This is attributed by many researchers to the higher decrease in the pores size after the first pass, over the decrease in pores size for further passes of as-cast composite, which results in higher mechanical properties, as previously mentioned[3, 4, 34]. For further ECAP passes, the extensive nucleation of voids limits the decrease in porosity percentage as suggested by Shehata et al.[3].



**Figure 8 Density and porosity % of Al-5%SiC<sub>p</sub> at different passes of ECAP**

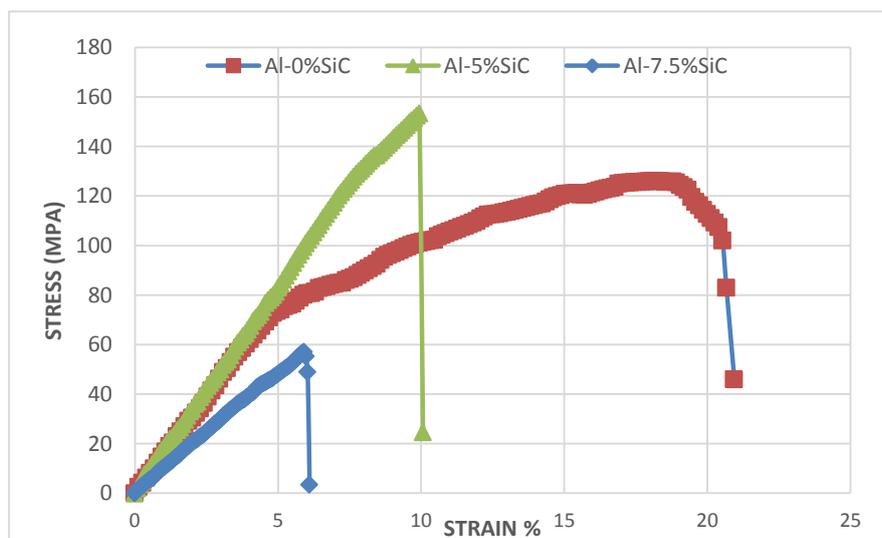


**Figure 9 Density and porosity % of Al-7.5%SiCp at different passes of ECAP**

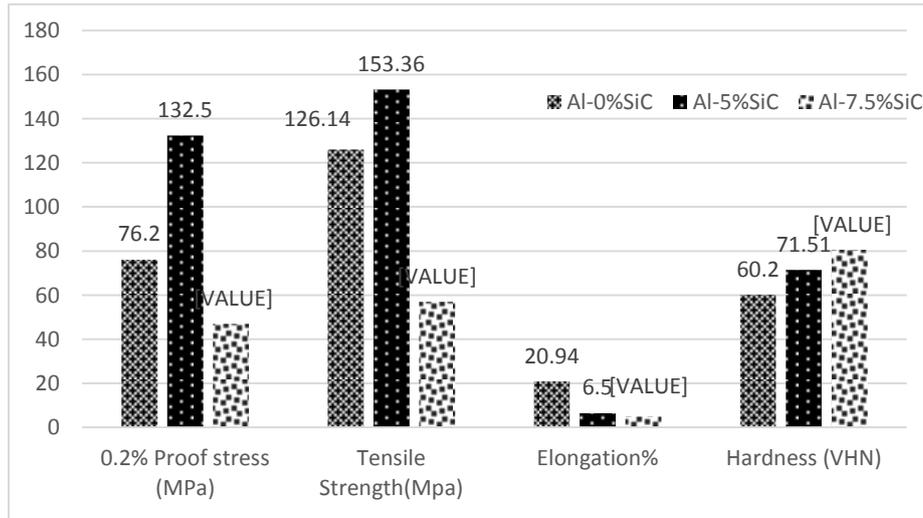
## 4. Mechanical Properties

### 4.1 Tensile Test

Figure 10 shows the tensile engineering stress-strain curves for as cast Al-5% SiC, and Al-7.5%SiC composites compared to Al6061 matrix material. The mechanical properties are summarized in Figure 11. Considering the UTS and the proof stress, the results have showed that, The addition of 5%SiC to the Al6061 have increased them by 21.57% and 73.9% respectively, While, the addition of 7.5%SiC has decreased the UTS and the proof stress by 54.7% and 38.3% respectively. The improvement in strengths of MMC is due to the effective dispersion of the SiC particles manufactured by SCM as previously [3, 11, 12], while observed the deterioration of the strength is due to the increase in the porosity and the agglomerations percentage as discussed above. Such strength behavior was also reported in many investigations[11, 12].Where the porosity level and distribution in MMC usually plays an important role in controlling the mechanical properties[3, 31].



**Figure 10 Engineering stress–strain curves from tension tests of as cast composites**



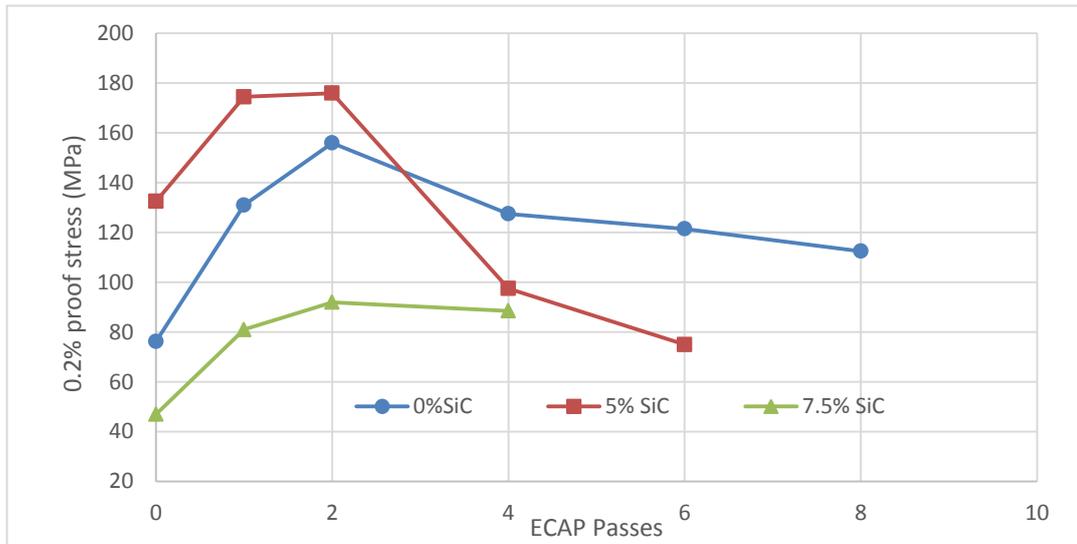
**Figure 11 Comparison of various mechanical properties for as cast composites with different percentages of SiC**

As for the hardness, the results show that, increasing the reinforcement particles have increased the hardness by 18.7% and 34% for Al-5%SiC and Al-7.5% SiC composites, respectively, as compared to matrix. A similar hardness trend was also reported by Subramani et al.[13].

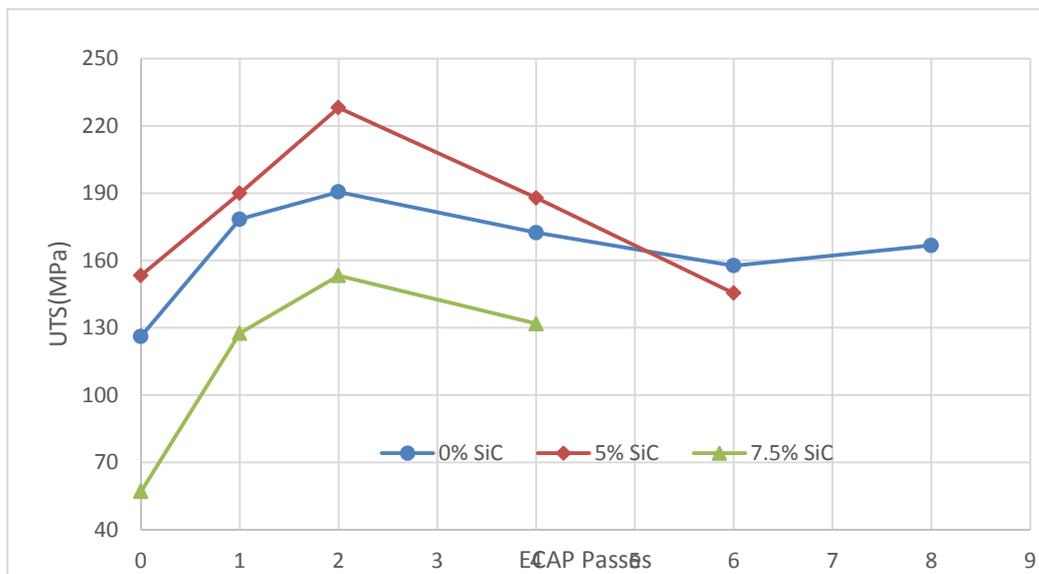
After ECAP, the UTS and the proof stress of matrix and composites containing 5% and 7.5% SiC have tremendously increased as the number of ECAP passes increase, up to the second pass, Figure 12, Figure 13. For further passes, they had a decreasing trend for all compositions. After the first pass, the proof stress has almost twice the value of the as cast matrix and Al-7.5%SiC composite, while it has just increased by 31.7% in case of Al-5%SiC composite. After the second pass, the proof stress of the matrix, Al-5%SiC, and Al-7.5%SiC composites has increased by 104.7%, 32.8%, and 95.7%, respectively, as compared to the as cast. The proof stress superiority of the Al-5%SiC composite was also maintained over the matrix and the Al-7.5%SiC composite. After the first pass, the UTS increased by 41.43%, 23.85% and 123.4% for the matrix, Al-5%SiC and Al-7.5%SiC composites, respectively. After the second pass, the UTS of the matrix, Al-5%SiC, and Al-7.5%SiC composites has increased by 51%, 48.7%, and 168.4%, respectively, as compared to the as cast. After the second pass, upon all samples of different SiC contents, the Al-5%SiC composite had the maximum UTS and proof stress of 228.1 MPa, and 176 MPa, respectively, at the second pass.

The reasons behind the increasing of strength are the decreasing of porosity and agglomerations percentage, because the Porosity level and distribution in MMC usually play an important role in controlling the mechanical properties. These percentages were decreased by increasing the ECAP passes.

The decrease in the strength beyond the second pass, can be attributed to the severe plastic deformation at elevated temperature which in turn has resulted in a ultrafine grain structure which has an accelerated rate of dynamic recovery of the materials as suggested by many researchers[20, 35–38]. The formation of the new fine-grained structure is significantly affected by temperature at higher strains repeated ECAP deformation, which can give enough time for dislocation rearrangement taking place in various strain-induced boundaries, leading to both increase of grain boundary and decrease of dislocation density in sub-grain interiors as previously[35, 36].

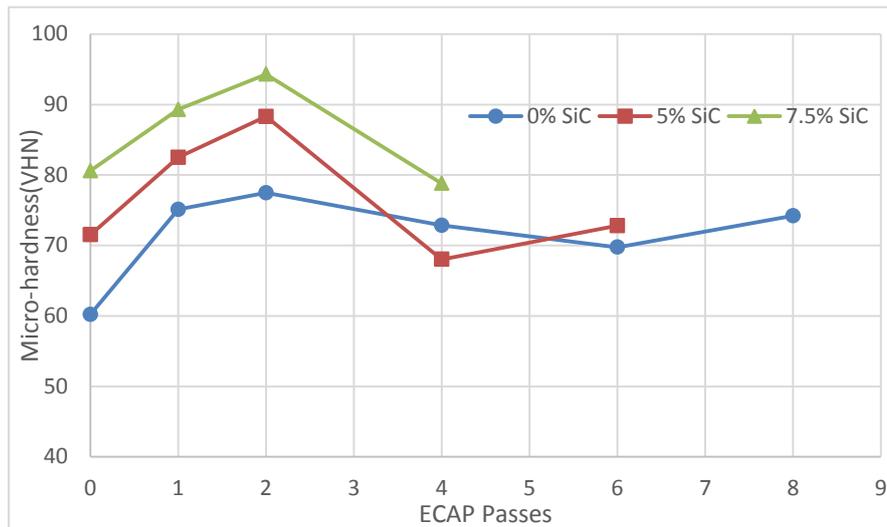


**Figure 12** The 0.2% proof stress of the Al6061 and composites in as cast and after ECAP pass against the number of ECAP passes.



**Figure 13** the UTS of the Al6061 and composites in as cast and after ECAP pass against the number of ECAP passes.

**Figure 14** shows the effect increasing number of ECAP passes on average hardness values for matrix materials and composites containing 5% and 7.5% SiC particles. The hardness increase by increasing the ECAP passes in all cases. After the first pass the hardness increase by 24.7%, 11% and 10.8% in case of Al6061, Al-5%SiC and Al-7.5%SiC, respectively. Upon all samples of different SiC content the Al-7.5%SiC composite had the maximum hardness 94.3VHN, at the second pass.



**Figure 14** the hardness of the Al6061 and composites in as cast and after ECAP pass against the number of ECAP passes.

Similar behavior is observed in hardness variation with ECAP passes. The hardness increased for all materials up to the second pass beyond, which the hardness falls again. On the other hand the hardness of the matrix increased with addition of the SiC so that the hardness of 7.5%SiCp reached the highest value. This is due to the present of the SiC hard particles is respective of the ductility which affect the tensile behavior.

## 5. Conclusion

Al6061 matrix with SiC reinforcement can be successfully fabricated using conventional low cost method of stir casting, with 5% and 7.5%SiC<sub>p</sub>, then ECAPed at 300°C up to 8<sup>th</sup> passes.

In the cast composite the percentage of porosity increases with the amount of SiC addition and the distribution of SiC particles becomes less uniform with formation of agglomerations. Increasing the SiC content up to 5% in the matrix increase the strength. while, the hardness continue to increase by increasing the SiC content.

Applying ECAP technique on as cast composites has resulted in high reduction of the porosity level. The ECAP fourth pass has decreased the porosity level from 2.75%, and 3.9% in the as cast composite to 1.3% and 2.02% for Al-5%SiC and Al-7.5%SiC respectively. While the maximum reduction of porosity level was 1.03%, obtained after the 6<sup>th</sup> pass for Al-5%SiC.

Proof stress, UTS, and hardness increased with the ECAP passes, up to the second pass, beyond which, they had a decreasing trend for all compositions.

The maximum values obtained after the second pass of proof stress are 156, 176 and 92 MPa, and for UTS are 190.5, 228.1, and 153.3 MPa, while for hardness are 77.5, 88.3, and 94.3 HVN, for matrix, Al-5%SiC and Al-7.5%SiC respectively.

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