# MICROSTRUCTURAL REFINEMENT AND ENHANCEMENT OF MECHANICAL PROPERTIES OF Al (2014) – SiC COMPOSITES BY EQUAL CHANNEL ANGULAR PRESSING

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**ABSTRACT**

The major aim of this work is to prepare a metal matrix composite that combines the high tensile properties of material like 2014 Aluminium alloy (base metal) with the high strength material like Silicon Carbide (reinforcement) within an optimum cost limit. There is a need to refine and enhance the properties of composites fabricated by primary processing method such as Stir Casting. Equal Channel Angular pressing (ECAP) is a proven secondary processing method for grain refinement. ECAP refines the grain size of the composites, leading to higher strength as grain is refined down to sub-micron level. Initially SiC is coated with Nickel in order to increase its wettability. The Aluminium composites are fabricated by Stir Casting process with 5 %, 10 % and 15 % SiC respectively. Then the composites are subjected to ECAP using die with $120^o$ angular channel. The Stir Cast and ECAPed samples are tested and compared for tensile, flexural, impact and hardness properties. Results reveal that the mechanical properties are improved when the composites are subjected to ECAP. The composite samples with Al (2014) - 15 % Nickel coated SiC have higher Tensile Strength, Flexural Break Load, Impact Strength and Hardness followed by Al (2014) - 10 % Nickel coated SiC, Al (2014) - 5 % Nickel coated SiC and Al (2014). Also it is observed that the percentage elongation of the Stir Cast samples reduces when subjected to ECAP as the yield strength of ECAPed sample is higher than Stir Cast samples. The microstructure is analysed using Optical Microscope and grain size is found to be reduced in ECAPed samples.

**Keywords:** 2014 Aluminium alloy, Silicon Carbide, ECAP, Mechanical Properties, Microstructure

## 1 INTRODUCTION

A Metal Matrix Composite (MMC) is a composite material with at least two constituent parts, one being a metal. The other material may be a different metal or another material, such as a ceramic or organic compound. The widespread adoption of particulate metal matrix composites (MMCs) for engineering applications had been hindered by the high cost of producing components of even minimally complex shape. In the case of particle reinforced metal matrix composites, the distribution of the reinforcement particles in the matrix alloy was influenced by several factors during casting [7]. The properties of 2014 Aluminium alloy-based powder metallurgy composites containing silicon carbide particles/fibres was studied and was concluded that the addition of reinforcement imparted improved densification and work hardening rates [13]. High silicon content aluminium alloy–silicon carbide metal matrix composite material was synthesized, with 10% SiC using different stirring speeds and stirring times. The brinell hardness test was performed on the composite specimens from base of the cast to top. The results revealed that stirring speed and stirring time influenced the microstructure and the hardness of composite [9]. Improving wettability and optimizing interfacial structure were prerequisites for successful fabrication and further enhancement of thermal properties [16]. The effect of electroless Ni coating of SiC particles on the corrosion behaviour of A356 based squeeze cast composite was studied and it was observed that electroless Ni coating can be used to increase wettability of SiC [2]. The strain localization patterns under equal channel angular pressing were studied [12]. The displacement of the shear band spectrum was found towards lower wave numbers due to decreased shear strain rate. The effect of equal channel angular pressing and torsion on SiC-particle distribution of SiC-Al composites was studied and it was found that the distribution greatly increases from the compaction stage to the angular pressing stage during ECAPT [8]. The sample size effect was investigated on the deformation heterogeneity and texture development during equal channel angular pressing [5]. For each sample, the simulated effective plastic strain rate is close to the theoretical value. The minimum grain size obtainable by equal channel angular pressing was investigated and it was demonstrated that minimum grain size obtainable by ECAP agrees with dislocation model developed [6]. In this work Stir casting is the primary processing method used for composite production. The Composites subjected to Stir Casting are not consistent and show variation in properties. So there is a need to bring consistency and refine the final properties of composites obtained by Stir Casting. Equal Channel Angular Pressing (ECAP) is a proven Secondary processing method to refine the properties of materials. Therefore the fabricated composites are subjected to ECAP.

## 2 SELECTION OF MATERIALS

### 2.1 Aluminium Alloy (2014)

It is among the strongest available aluminium alloys, as well as has high hardness. The Chemical composition of 2014 Aluminium alloy was verified using Optical Emission Spectroscopy. Then the chemical composition of the alloy was determined. Table 1 shows the comparison of theoretical and test values of 2014 aluminium alloy.

| **Chemicals** | **Theoretical Composition (%)** | **Test Results (%)** |
|---|---|---|
| Chromium | 0.1% max | 0.081 % |
| Copper | 3.9% - 5% | 5 % |
| Iron | 0.7% max | 0.179 % |
| Magnesium | 0.2% - 0.8% | 0.417 % |
| Manganese | 0.4 - 1.2% | 1.2 % |
| Silicon | 0.5% - 1.2% | 1.2 % |
| Titanium | 0.15% max | 0.001 % |
| Zinc | 0.25% max | 0.246 % |
| Aluminium | Remainder | Remainder |

Table 1: Comparison of Theoretical and Test values

### 2.2 Silicon Carbide

Silicon carbide (SiC) is a compound of silicon and carbon having high temperature resistance and high hardness. It is also highly inert chemically. Silicon Carbide with average particle size 25 µm is used as the reinforcement of composites.

## 3 ELECTROLESS NICKEL COATING OF SILICON CARBIDE

Electroless Nickel coating was carried out for improving the binding properties and wettability of silicon carbide. First the SiC particles were cleaned in acetone for 15 minutes. After cleaning, particles were senstized for another 15 minutes in a solution containing 10 g/L $SnCl_2$ (Tin (II) Chloride) and 30 ml HCl (Hydrochloric Acid). The sensitized particles were immersed in a solution containing 0.25 g/L $PdCl_2$ (Palladium (II) Chloride) and 3ml/L HCl for 15 minutes in order to activate the non-metal SiC particle surface.


Figure 1: Arrangement for Nickel Deposition

Rinsed and dried SiC particles were gently dispersed in an electroless nickel bath containing 45 g $NiCl_2$ (Nickel (II) Chloride), 8 g $NaH_2PO_2.H_2O$ (Sodium Hypophosphite), 100 g $Na_3C_6H_5O_7$ (Tri Sodium Citrate) and 50 g $NH_4Cl$ (Ammonium Chloride) and was dissolved in 1 L distilled water. Deposition was carried out at 353 to 363 K and 8 to 9 pH. Figure 1 shows the arrangement for Nickel deposition.

## 4 FABRICATION BY STIR CASTING

Here the stir-casting method is adopted for the preparation of metal-matrix Al-SiC composites. Stir casting set up is shown in figure 2. Initially the die was cleaned using emery sheet and silicone spray was sprayed over die to avoid sticking of mould in die walls. The melting of the aluminium alloy ingot pieces is carried out in the graphite crucible inside the furnace.

Figure 2: Stir Casting Set Up

First 150 g Silicon Carbide powder was preheated at 773 K. Next the crucible with aluminium alloy is heated to about 1273 K. Then degasser (which removes all the trapped gases from the mixture in the crucible) and coverall (which ensures that the temperature of the mixture in the crucible does not get transferred easily to the atmosphere) was added to the mixture. Then the preheated powder is mechanically mixed with each other. The furnace, at the said temperature completely melts the pieces of aluminium alloy and the inserted powder of silicon carbide, remain scattered. The stirring mechanism is lowered into the crucible inside the furnace and set at the required depth. The drive arrangement of stirrer is switched on and the vigorous automatic stirring of the material takes place for five minutes with normal 300 rpm of stirring rate, thereby uniformly dispersing the additive powders in the aluminium alloy matrix. After completion of this process, the mixture has been taken out of the furnace and within thirty seconds, poured into the dies and allowed to solidify. Al (2014) and 5 %, 10 % and 15 % SiC (coated) was fabricated using Stir Casting method. Also to determine the variation in properties due to addition of reinforcements, only Al (2014) was fabricated by stir casting.

## 5 EQUAL CHANNEL ANGUALR PRESSING (ECAP)

ECAP, as secondary processing method, is able to refine the microstructure of metals and alloys, thereby improving their strength. First the 2014 Aluminium alloy – Silicon Carbide composite is fabricated by stir casting method in rod shape. The sample, in the form of a rod, is machined to fit within the channel and the die is placed in some form of press so that the sample can be pressed through the die using a plunger. The die has two equally sized channels connected at a finite angle. The nature of the imposed deformation is simple shear which occurs as the sample passes through the die.

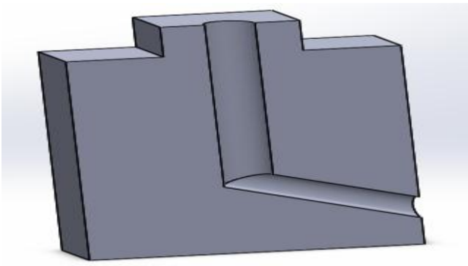


Figure 3: Planar View of ECAP Die

Despite the introduction of a very intense strain as the sample passes through the shear plane, the sample ultimately emerges from the die without experiencing any change in the cross-sectional dimensions. Planar View of ECAP Die is shown in figure 3. The retention of the same cross-sectional area when being processed by ECAP, despite the introduction of very large strains, is the important characteristic of this Severe Plastic Deformation processing. ECAP improves Mechanical properties of metals and alloys, leading to high strength or Super Plasticity. This is because grain is refined down to sub-micron level. ECAP Die has $120^{o}$ angular channel and its clamping set has also been fabricated. Die and punches are made up of D2 Tool Steel. There are three punches namely stepped bottom punch, middle punch and top punch. The clamping set consists of two top clamps with tea nuts and six side blocks for holding the die. There is a screw jack for adjusting the height of the top clamp. Molybdenum Disulphide ($MoS_2$) is used as the lubricant for ECAP process.

## 6 RESULTS AND DISCUSSION

Sample 1 contains 95 % Al (2014) – 5 % SiC (coated), Sample 2 contains 90 % Al (2014) – 10 % SiC (coated), Sample 3 contains 85 % Al (2014) – 15 % SiC (coated) and Sample 4 contains 2014 Aluminium alloy only. For each test, five samples were used.

### 6.1 Tensile Test

The capacity of a material to withstand a static load can be determined by testing that material in tension.

| Specimen | Yield Strength [MPa] | |
|---|---|---|
| | **Stir Cast Sample** | **ECAP (120$^o$) Sample** |
| Aluminium alloy - 95%<br>Silicon Carbide - 5 % (Coated) | 130.00 | 209.75 |
| Aluminium alloy - 90%<br>Silicon Carbide - 10 % (Coated) | 143.09 | 228.96 |
| Aluminium alloy - 85%<br>Silicon Carbide - 15 % (Coated) | 151.01 | 242.62 |
| Aluminium alloy | 123.01 | 196.82 |

Table 1: Results of Tensile test – Yield Strength

| Specimen | Tensile Strength [MPa] | |
|---|---|---|
| | **Stir Cast Sample** | **ECAP (120$^o$) Sample** |
| Aluminium alloy - 95%<br>Silicon Carbide - 5 % (Coated) | 140.62 | 225.13 |
| Aluminium alloy - 90%<br>Silicon Carbide - 10 % (Coated) | 151.31 | 243.16 |
| Aluminium alloy - 85%<br>Silicon Carbide - 15 % (Coated) | 160.65 | 257.14 |
| Aluminium alloy | 132.30 | 211.68 |

Table 2: Results of Tensile test –Tensile Strength

| Specimen | Elongation [%] | |
|---|---|---|
| | **Stir Cast Sample** | **ECAP (120$^o$) Sample** |
| Aluminium alloy - 95%<br>Silicon Carbide - 5 % (Coated) | 3.11 | 2.96 |
| Aluminium alloy - 90%<br>Silicon Carbide - 10 % (Coated) | 3.43 | 3.20 |
| Aluminium alloy - 85%<br>Silicon Carbide - 15 % (Coated) | 3.78 | 3.54 |
| Aluminium alloy | 2.44 | 2.23 |

Table 3: Results of Tensile test – Percentage Elongation

The specimen for tensile test was prepared as per ASTM B557M standard and tested. The results of tensile test are tabulated in tables 1, 2 and 3. From the tables 1 and 2, it may be concluded that composite samples with Al (2014) - 15 % Nickel coated SiC have higher tensile strength and yield strength followed by Al (2014) - 10 % Nickel coated SiC, Al (2014) - 5 % Nickel coated SiC and Al (2014). The Tensile Strength and Yield Strength is higher for samples subjected to ECAP (120$^o$) compared to Stir Cast samples. Also from table 3, it is observed that the percentage elongation of the Stir Cast samples reduces when subjected to ECAP (120$^o$). Figure 4 shows the tensile test specimen.

Figure 4: Tensile test specimen

### 6.1.1 Comparison of Tensile Properties

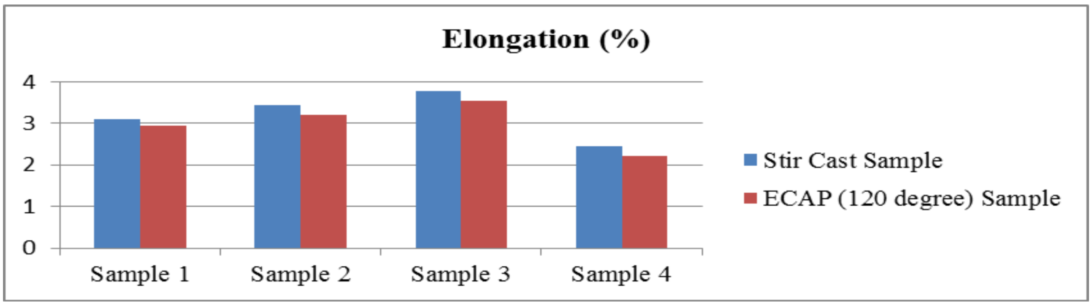



Figure 5: Variation of Percentage Elongation

Figure 5 shows the variation of percentage elongation. The reason can be co-related with the help of following formula.

$$E = \text{stress / strain}$$

$$= \sigma / \varepsilon$$

Where, E – Young's Modulus in N/mm$^2$,

$\sigma$ – Yield Strength in N/mm$^2$

$\varepsilon$ – Strain

For a material, Young's Modulus is constant. So when yield strength increases, strain has to decrease as per the above relation. Therefore percentage elongation of Stir Cast sample decreases when subjected to ECAP (120$^o$) as the yield strength of Stir Cast sample increases when subjected to ECAP (120$^o$).

## 6.2 Flexural Test

This mechanical testing method measures the behaviour of materials subjected to simple bending loads. The three point flexure fixture produces its peak stress at the specimen mid-point with reduced stress elsewhere.

| Specimen | Flexural Break Load [kN] | |
|---|---|---|
| | Stir Cast Sample | ECAP (120$^o$) Sample |
| Aluminium alloy - 95% Silicon Carbide - 5 % (Coated) | 15.25 | 18.12 |
| Aluminium alloy - 90% Silicon Carbide - 10 % (Coated) | 17.85 | 20.23 |
| Aluminium alloy - 85% Silicon Carbide - 15 % (Coated) | 20.06 | 22.12 |
| Aluminium alloy | 13.47 | 15.36 |

Table 4 Flexural test results

The specimen for flexural test (figure 6) is prepared as per ASTM B557M standard and tested in three point flexural testing machine (figure 4.4). The results of flexural test are tabulated in table 4 and from the table, it can be seen that the Flexural Break Load is higher for samples subjected to ECAP (120$^o$) compared to Stir Cast samples.

Figure 6: Flexural test specimen

### 6.2.1 Comparison of Flexural Properties

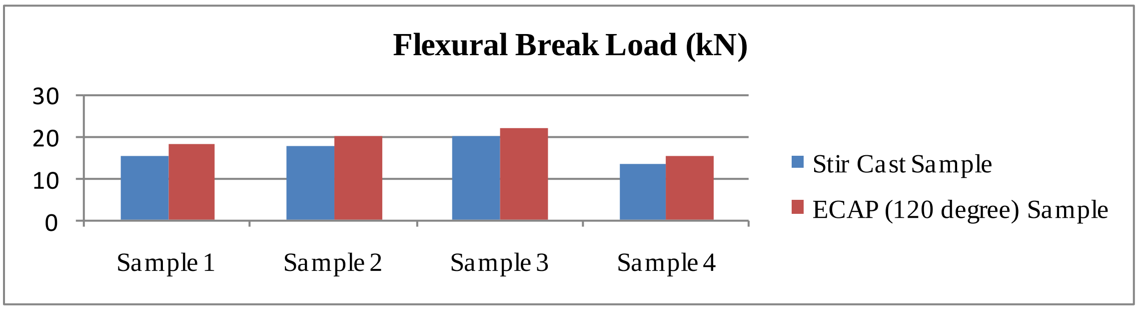



**Figure 7: Variation of Flexural Load**

Figure 7 shows the variation of flexural load. After both the processes i.e. Stir Casting and ECAP ($120^o$), flexural load is higher for Al (2014) - 15 % SiC, followed by Al (2014) - 10 % SiC and Al (2014) - 5 % SiC. Al (2014) has the least flexural load in all three processes.

## 6.3 Impact Test

Impact Testing involves the sudden and dynamic application of the load. Impact test is defined as the resistance of a material to rapidly sudden applied loads. Charpy impact test was carried out on the fabricated samples.

| Specimen | Energy absorbed [J] | |
|---|---|---|
| | **Stir Cast Sample** | **ECAP ($120^o$) Sample** |
| Aluminium alloy - 95% Silicon Carbide - 5 % (Coated) | 4 | 6 |
| Aluminium alloy - 90% Silicon Carbide - 10 % (Coated) | 6 | 8 |
| Aluminium alloy - 85% Silicon Carbide - 15 % (Coated) | 8 | 10 |
| Aluminium alloy | 2 | 4 |

Table 5: Impact test results

Figure 8: Impact test specimen

The specimen for charpy impact test is prepared as per ASTM B557M standard and tested. The results are tabulated in table 5. It is evident that Stir Cast samples have the lowest values of impact strength in both the processes. The impact strength is higher for samples subjected to ECAP (120°) compared to Stir Cast process. Figure 8 shows the impact test specimen.

### 6.3.1 Comparison of Impact Properties

Figure 9 shows the impact energy absorbed by the samples and it is clear that impact strength is higher for Al (2014) - 15 % Nickel coated SiC, followed by Al (2014) - 10 % Nickel coated SiC and Al (2014) - 5 % Nickel coated SiC and Al (2014) has the least impact strength in both the processes.

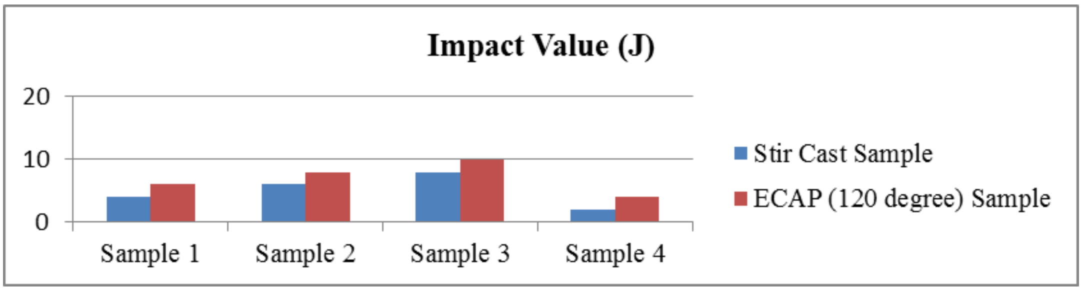



Figure 9: Impact energy absorbed by Samples

## 6.4 Hardness Test

The Brinell test is frequently used to determine the hardness of forgings and castings that have a grain structure too course for Rockwell or Vickers testing. Brinell hardness test is carried out as per IS 1500 standard. Ball shaped indenter made of hardened tungsten is used. The diameter of the indenter is 10 mm and the load applied is 500 kgf. The results are tabulated in table 6. It is evident that hardness is higher for Al (2014) - 15 % Nickel coated SiC, followed by Al (2014) - 10 % Nickel coated SiC and Al (2014) - 5 % Nickel coated SiC and Al (2014) has the least hardness in both the processes.

| **Specimen** | **Brinell Hardness [HBW]** | |
|---|---|---|
| | **Stir Cast Sample** | **ECAP (120°) Sample** |
| Aluminium alloy - 95%<br>Silicon Carbide - 5 % (Coated) | 72.1 | 109.33 |
| Aluminium alloy - 90%<br>Silicon Carbide - 10 % (Coated) | 77.4 | 113.33 |
| Aluminium alloy - 90%<br>Silicon Carbide - 15 % (Coated) | 81.6 | 120.33 |
| Aluminium alloy | 67.3 | 104.33 |

Table 6: Brinell Hardness test results

### 6.4.1 Comparison of Hardness

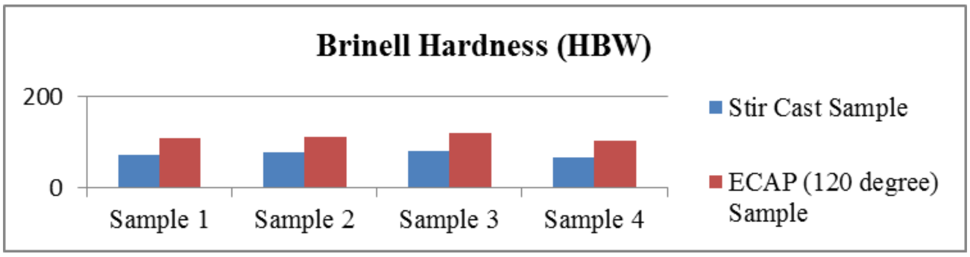
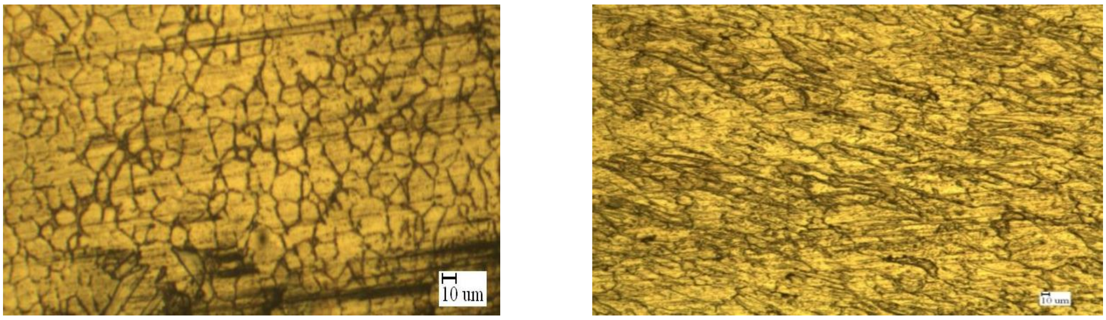



Figure 10: Brinell Hardness value of samples

Figure 10 shows the Brinell Hardness values of samples. It is clear that hardness is higher for samples subjected to ECAP ($120^o$) followed by Stir Cast samples.

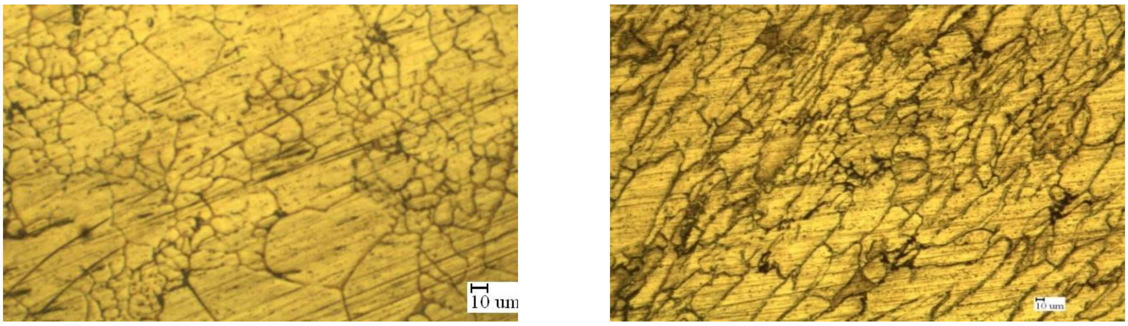
### 6.5 Microstructure and Grain Size Analysis

The microstructure of the fractured surfaces of the impact samples are analysed using Optical Microscope. First, emery polishing is done on the samples. Initially sample is polished using grit papers of various sizes (in sequence of 180, 220, 320, 600, 800 and 1200). Then polishing is done using fine emery papers of various grit sizes (in sequence of 1/0, 2/0, 3/0 and 4/0). Then polishing using diamond cloth is done. Next, the sample is washed in acetone. Then preferential etching is carried out on the sample using 5 % Hydrofluoric acid solution. Finally the sample is washed in distilled water. Thus the samples were prepared for viewing in Optical Microscope. Grain size analysis was carried out using Image analyzing software.



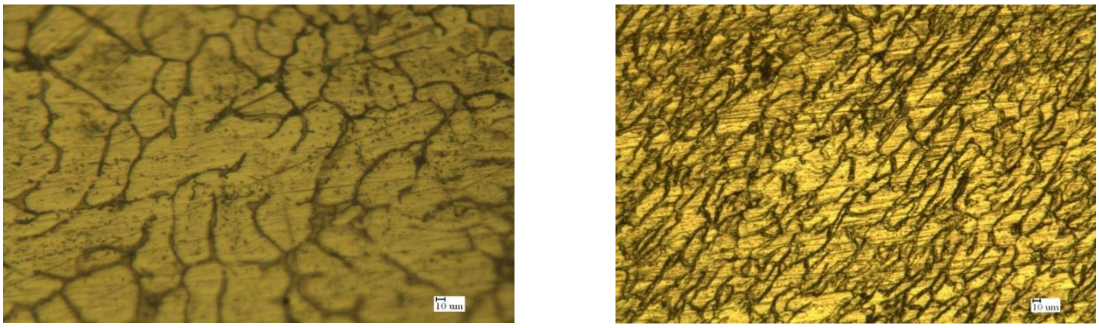

a) Stir Cast sample b) Sample subjected to ECAP ($120^o$)

Figure 11: Sample 1 at 100 X




a) Stir Cast sample b) Sample subjected to ECAP ($120^o$)

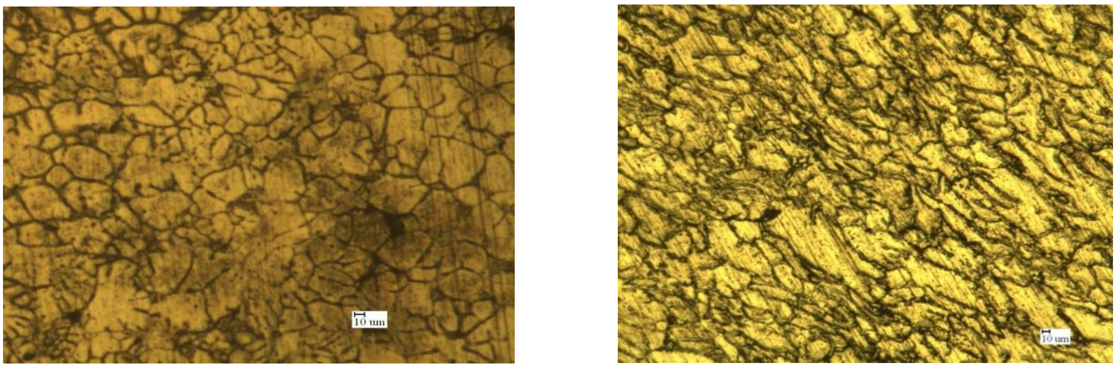
Figure 12: Sample 2 at 100 X



a) Stir Cast sample b) Sample subjected to ECAP ($120^o$)

Figure 13: Sample 3 at 100 X

Grain size is analysed as per ASTM E 112-23 standard. Microstructure of sample 1 i.e Al (2014) – 5 % SiC Metal Matrix Composites is shown after Stir Casting (figure 11 a) and ECAP (120$^o$) (figure 11 b) respectively. It is observed that the grain size is reduced for Stir Cast sample (53.4 µm) when it is subjected to ECAP 120$^o$ (37.8 µm). In the microstructure, the black patches in the sample indicate Silicon Carbide. The mechanical properties obtained are less in case of Stir Cast samples and they increase when subjected to ECAP 120$^o$ [1]. For all four set of samples, the grain size of Stir Cast samples reduces when subjected to ECAP 120$^o$. The microstructure of sample 2 i.e Al (2014) – 10 % SiC Metal Matrix Composites after Stir Casting and ECAP (120$^o$ is shown in figure 12 a and 12 b respectively. The grain size of sample 2 is lesser compared to sample 1 for Stir Cast (44.9 µm) as well as ECAP 120$^o$ (31.8 µm). Figure 13 a and 13 b show the microstructure of sample 3 i.e Al (2014) – 15 % SiC Metal Matrix Composites after Stir Casting and ECAP (120$^o$) respectively. The grain size of sample 3 is same as sample 2 for both the processes. Microstructure of sample 4 i.e Al (2014) is shown after Stir Casting (figure 14 a) and ECAP (120$^o$) (figure 14 b) respectively. The microstructure of sample 4 is slightly elongated when compared to other three samples.



a) Stir Cast sample b) Sample subjected to ECAP (120$^o$)

Figure 14: Sample 4 at 100 X

### 6.5.1 Comparison of Grain Size of Samples

From table 7 (average grain size of sample) and figure 16 (variation of average grain size), it is observed that Sample 1 (Al 2014 – 5 % SiC Metal Matrix Composites has largest grain size among the fabricated samples. All the other three fabricated samples i.e Al (2014) – SiC 10 % SiC Metal Matrix Composites, Al (2014) – SiC 15 % SiC Metal Matrix Composites and Al (2014) show the same grain size. In case of comparison within the processes, Stir Cast Samples have the maximum grain size followed by samples subjected to ECAP (120$^0$) respectively.

| **Specimen** | **Average Grain Size in µm** | |
|---|---|---|
| | **Stir Cast Sample** | **ECAP (120$^o$) Sample** |
| Aluminium alloy - 95%<br>Silicon Carbide - 5 % (Coated) | 53.4 | 37.8 |
| Aluminium alloy - 90%<br>Silicon Carbide - 10 % (Coated) | 44.9 | 31.8 |
| Aluminium alloy - 85%<br>Silicon Carbide - 15 % (Coated) | 44.9 | 31.8 |
| Aluminium alloy | 44.9 | 31.8 |

Table 7: Average Grain Size of Samples

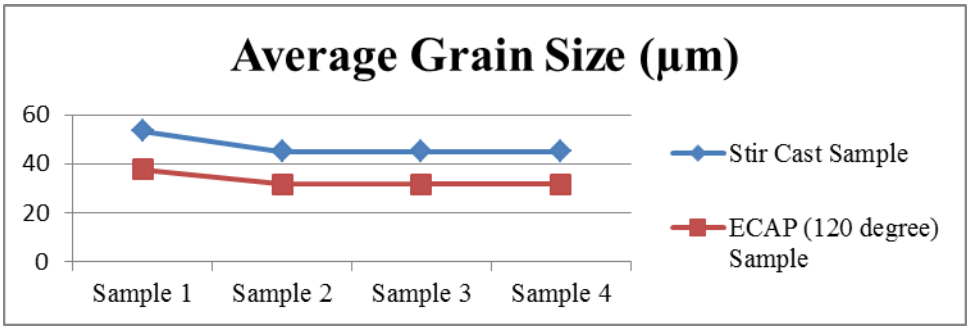



Figure 15: Variation of Average Grain Size

## 9 CONCLUSIONS

The major aim and objective of this paper is to prepare a metal matrix composite that combines the high tensile properties of materials like Aluminium with the high strength of abrasive materials like Silicon Carbide within an optimum cost limit. Considering availability and relative cost effectiveness, Aluminium alloy 2014 was chosen to be the base metal providing the composite with the good tensile strength. Silicon Carbide, with its low relative cost amongst the options, and high hardness, was chosen to be the reinforcement giving the extreme hardness properties to the composite. To increase the wettability and binding properties, Silicon Carbide was coated with Nickel. The samples were fabricated using stir casting method with Al (2014) and 5% , 10 % and 15 % SiC (coated) and 2014 Aluminium alloy alone. The Tensile test, Flexural test, Impact test and Hardness test were carried out in the Stir Cast samples and samples subjected to ECAP ($120^o$). The composite samples with Al (2014) - 15 % Nickel coated SiC have higher Tensile Strength, Flexural Break Load, Impact Strength and Hardness followed by Al (2014) - 10 % Nickel coated SiC, Al (2014) - 5 % Nickel coated SiC and Al (2014). Thus it can be concluded that, increase in content of reinforcement improves mechanical properties (Tensile, Flexural, Impact properties and Hardness). The Tensile Strength, Yield Strength, Flexural Break Load, Impact Strength and Hardness is higher for samples subjected to ECAP ($120^o$) followed by Stir Cast samples. The values of mechanical properties are better for samples subjected to ECAP (compared to Stir Cast samples) due to the refinement of grains of samples. The Tensile Strength, Yield Strength, Flexural Break Load, Impact Strength and Hardness increases by 60 % from Stir Cast to ECAP ($120^o$) process. Also it is observed that the percentage elongation of the Stir Cast samples reduces when subjected to ECAP ($120^o$), as the yield strength of Stir Cast samples increases when subjected to ECAP ($120^o$).

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