

Thermal Characterization of Aluminium Alloy-Redmud Composite Material Processed by Equal Channel Angular Pressing (ECAP)

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Abstract - The Aluminium metal matrix composite material with Al-5%Mg matrix and Red mud particles reinforcement of different proportions (5, 10 and 15wt.%) were prepared by stir casting technique. The specimens prepared with this composition were subjected to severe plastic deformation by Equal channel angular pressing (ECAP) technique. The ECAP die with the channel angle of 105° and curvature angle of 30° are prepared and the ECAP process was carried out at room temperature for a single pass. The effect of ECAP on the thermal properties of Al-5%Mg -Red mud composite was evaluated for various weight fractions of red mud. The thermal analysis of composite before and after ECAP is investigated by using Thermo Gravimetric – Differential thermal analyzer (TG-DTA) and Differential scanning calorimeter (DSC). The thermal properties such as thermal decomposition, thermal stability, thermal conductivity and phase transformation of the as-cast Al-5%Mg -Red mud and ECAPED composites are compared with the Al-5%Mg base alloy material.

Keywords: Aluminium metal matrix composite, Red mud, ECAP, TG-DTA, Differential scanning calorimeter

1. INTRODUCTION

Metal Matrix Composites materials have a combination of different, superior properties to an unreinforced matrix which are increased strength, higher elastic modulus, higher service temperature, improved wear resistance, high electrical and thermal conductivity, low coefficient of thermal expansion and high vacuum environmental resistance. These properties can be attained with the proper choice of matrix and reinforcement. The Bauxite residues, also known as red clay, red mud or alumina refinery residues (ARR), are a very alkaline waste product consisting mainly of iron oxide which is created in the industrial production of alumina. This is the primary material used in manufacturing of aluminium metal, as well as widely used in the manufacture of ceramics, abrasives and refractories). In terms of metal production, the ratio of aluminium to red mud is 1:2. The rising demand for inflation has made it interested in the usage of Red mud as reinforcement element in the composites, as it contains major elements such as Al_2O_3 , Fe_2O_3 , TiO_2 and Na_2O elements with properties that are suitable for thermal applications. It has higher strength than untreated heat-resistant alloys. In this present study, the stir casting method was employed to fabricate the Al-5%Mg and Red mud composite material. Thermal characterization of metal matrix materials using differential scanning calorimetry measurement is a resourceful technique to determine the distribution of heat flow and specific heat ability and enthalpy. Measurement of the thermal properties of materials is fundamental to a better understanding of thermal design. Differential Scanning Calorimeter (DSC) is the technique that measures the difference in heat flow to a sample and to a reference sample as a direct function of time or temperature under heating, cooling or isothermal conditions. There is extensive research conducted, in order to assess the specific thermal capacity and enthalpy of Al6061, silicon carbide and graphite metal composite matrix from room temperature to $300^{\circ}C$ based on heat flow response. (S.A Mohan Krishna@2016). Equal Channel Angular Pressure (ECAP) has the ability to achieve a secondary element Grain size. Aluminum alloy was deformed by ECAP until eight passes at 473 K. The grain size was changed to 300nm by deformation. Specific heat capacity was monotonically increased. (Jong Cheon Lee @2009).

2. EQUAL CHANNEL ANGULAR PRESSING

Severe plastic deformation (SPD) characteristics are seen to be beneficial by strengthening pure metals and alloys and refining the grains by introducing a larger deformation of plastic as a true strain value which is greater than 1. SPD is performed under high pressures and relatively at low temperatures; it is often less than ideal $0.4T_m$, where T_m is the highest temperature in Kelvin which refers to the homologous temperature threshold at which the materials start to exhibit significant time dependent

deformation under stress, known as creep. Among the different SPD techniques, equal-channel angular extrusion/pressing (ECAE/P) process is important because it is unique in permitting the determination of a large number samples with no change in the appropriate coatings that the industries use. The angle of the channel is the most important experimental factor that affects the refinement of the grain because it forces the total tension imposed on each pass. Most experimental works reported to date used channel angle values of 90° to 120° , and there was little or no attempt to compare the results obtained when using die with different channel angles. Despite the efficiency of the ECAP process, the primary challenges of using a 90° die angle in Equal Channel Angular Pressing (ECAP) are the extremely high pressing forces required and the increased risk of material cracking or failure, particularly in materials with limited ductility. It is empirically easier to press billets when using dies with angles that are larger than 90° for very hard materials or for materials with low ductility. It is claimed that it is difficult to compress commercially pure tungsten at a channel angle of 90° degrees even at a high temperature of 1273 K due to crack generation in the billets. However, excellent results are achieved at the same temperature with ECAP die shown in the figure 1, when the angle of the channel increased from 110° to 120° .

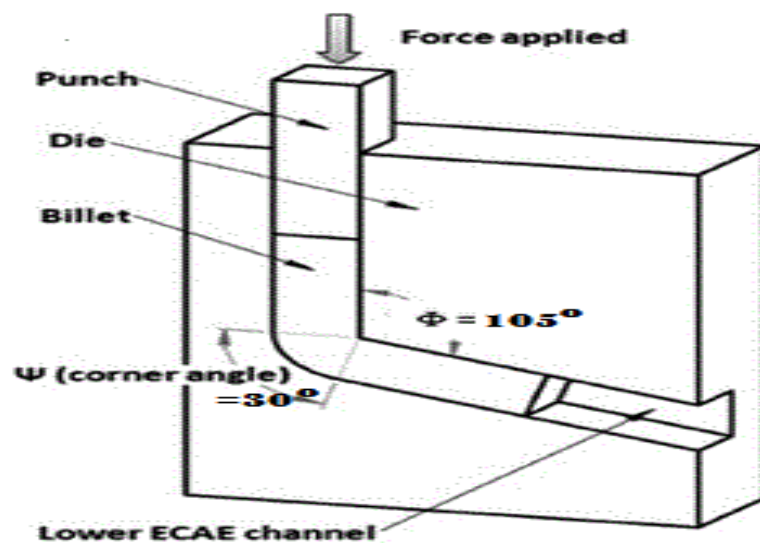


Figure 1: ECAP Die with channel angle of 105° and outer channel angle 30° ,

3. EXPERIMENTAL INVESTIGATION

3.1 Sample Preparation: Al-5%Mg alloy ingots were kept in a graphite crucible and charged into electric resistance furnace and the temperature was raised up to 750°C . To remove the moisture content from Red mud particles of three proportion specimens, they were kept at 350°C for 30 minutes. Preheated Red mud particle was uniformly added to Al-5%Mg molten metal at 750°C and stirring operation was carried out at 200rpm for 7 minutes. After the casting process, specimens were prepared by removing the excessive material from casted billets. The casted composite billets were machined to dimensions as $16 \times 16 \times 150$ mm by using CNC milling machine to align with the cross section of ECAP die which is in the square shape.

Before doing the ECAP process the samples should undergo the heat treatment process to homogenize the microstructure of samples. The temperature of the annealing of samples depends on the materials that are performing ECAP process and generally for aluminium alloy the annealing temperature is performed at 350°C to 400°C and for aluminium composites, it is lower than the aluminium alloy i.e, 200°C to 300°C . In this present study, before doing ECAP the samples were annealed at a temperature of 230°C for 30 minutes to homogenize the microstructure of the samples.

3.2 Extrusion process of ECAP

In the present investigation, the extrusion process of ECAP has been carried out by Universal testing machine (UTM) as shown in figure 2 with required loads for all the combinations of composite material that is 5, 10 and 15wt%'s. High carbon and high chromium punch is used while using the lubricant Molybdenum disulphide (MoS_2), which is applied to the die channels and cross-sections of the samples in order to reduce the friction between the samples and walls of the die.



Figure 2: Universal testing machine (UTM)

4. THERMAL ANALYSIS OF COMPOSITE SPECIMENS

4.1 Thermo Gravimetric-Differential Thermal Analysis (TG-DTA):

The materials undergo physical and chemical transactions when they are heated and cooled.

TG-DTA is one in which the heat effect analysis associated with chemical and physical changes of a material which is heated at a linear rate is experimented. In other words, differential thermal analysis is a technique in which the temperature difference between the sample and the thermally inert reference substance is continuously recorded as a function of furnace temperature or time. Its effects may be either endothermic or exothermic.

Thermogravimetric analysis is quantitative where any weight change associated with transition can directly record the loss in weight with time or temperature due to dehydration or decomposition. Thermogravimetric curves are characteristic curves for a given compound or system because of a unique sequence of physiochemical changes which occurs over definite ranges and at rates that are a function of molecular structure. Changes of weights are as a result of rupture and/ or formation of various physical or chemical bonds at elevated temperature that lead to the evaluation of volatile products or the formation of heavier products. The usual ambient temperature is maintained up to 1200°C with reactive or inert atmosphere. Figure 3 show the apparatus used to conduct Thermo Gravimetric-Differential Thermal Analysis.



Figure 3: Hitachi 7000 series TG-DTA

4.2 Differential scanning calorimeter (DSC):

Differential Scanning Scanner (DSC) is a thermal analysis technique used for more than two decades to measure temperatures and heat flows associated with shifts in materials as a function time and temperature. These measurements provide quantitative and qualitative information about the physical-chemical changes involving heat or exothermic processes, or changes in heat capacity. DSC shown in figure 4 is the most widely used thermal analysis technique with the applicability to metal alloys, polymers and

organic materials as well as different inorganic substances. A DSC measures the difference in heat flow rate ($mW=mJ$) between a sample and inert reference as a function of time and temperature.

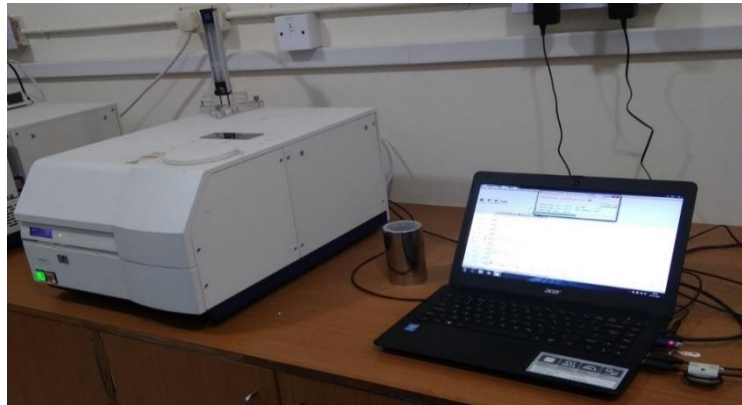


Figure 4: Differential scanning calorimeter

.5 .RESULTS AND DISCUSSIONS

Thermal analysis was performed in the temperature range of 25 to 850 °C at a heating range of 10 °C/min on Hitachi Simultaneous Thermogravimetric Analyzer (STA) 7000 Series apparatus (A facility at Advanced analytical laboratory (AAL), Andhra University Visakhapatnam) shown in figure 3. All the prepared samples (Al-5%Mg matrix composite with red mud compositions) were subjected to TG and DTA to investigate thermal behaviour and analyzed the variations with respect to pure Al-5%Mg alloy. The samples (as casted and after ECAP) are subjected to a heat range of 10 °C/min under N₂ environment to employ degradation rate and heat difference. The heat difference occurs due to phase transformation that takes place in the material and these phase transformations can be seen as Exothermic, Endothermic and Enthalpy reactions. Thermal decomposition of material can be calculated by the amount of weight or mass change which occurs at that various temperature. Thermal conductivity values are listed for before and after ECAP samples in the table 1.

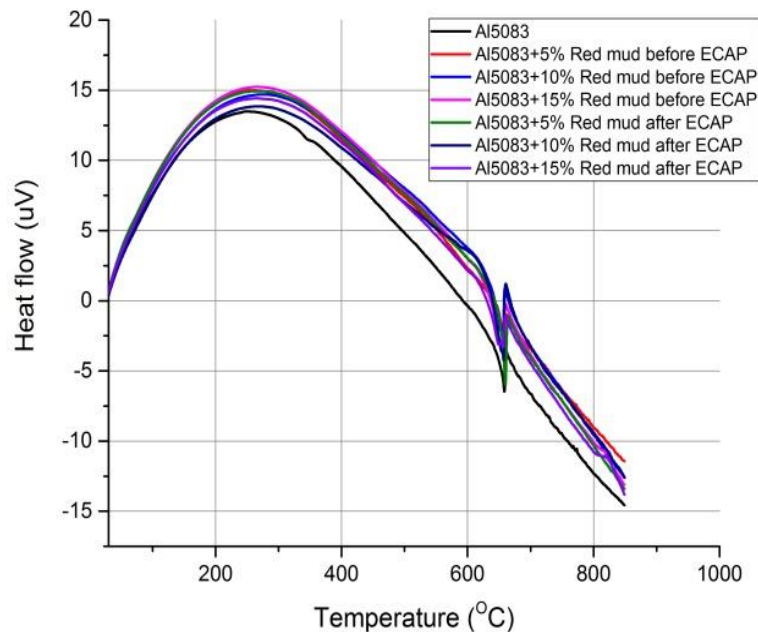
$$\text{weight loss or mass loss (\%)} = \frac{\text{mass initial}(Mi) - \text{mass final}(Mf)}{\text{mass initial}(Mi)} \times 100$$

Table 1: Thermal conductivity values of before and after ECAP samples

Material	Thermal conductivity (K)
Al-5Mg alloy	121
Before Equal channel angular pressing	
Al-5Mg -5%Redmud composite	112.8
Al-5Mg -10%Redmud composite	124.8
Al-5Mg -15%Redmud composite	124.8
After Equal channel angular pressing	
Al-5Mg -5%Redmud composite	118.32
Al-5Mg -10%Redmud composite	134.64
Al-5Mg -15%Redmud composite	126.6

5.1 Thermal stability:

Thermal analysis was performed in the temperature range of 25 to 850 °C at heating range of 10 °C/min on Hitachi Simultaneous Thermogravimetric Analyzer apparatus. Thermo Gravimetric analysis (TG) can be used to evaluate the thermal stability of the material. In this present investigation, the thermal stability of composite material is observed from the TG curves in graph 1. From the above TG – DTA analysis, thermal stability of 5, 10, and 15wt% of composite before ECAP was found up to 250°C and thermal stability up to the temperature range of 300 to 320°C was observed for ECAPed samples. Therefore it is concluded that the thermal stability of ECAPed composite material is higher than non ECAPed material because of the grain refinement in severe plastic deformation.



Graph 1: TG-DTA analysis curves

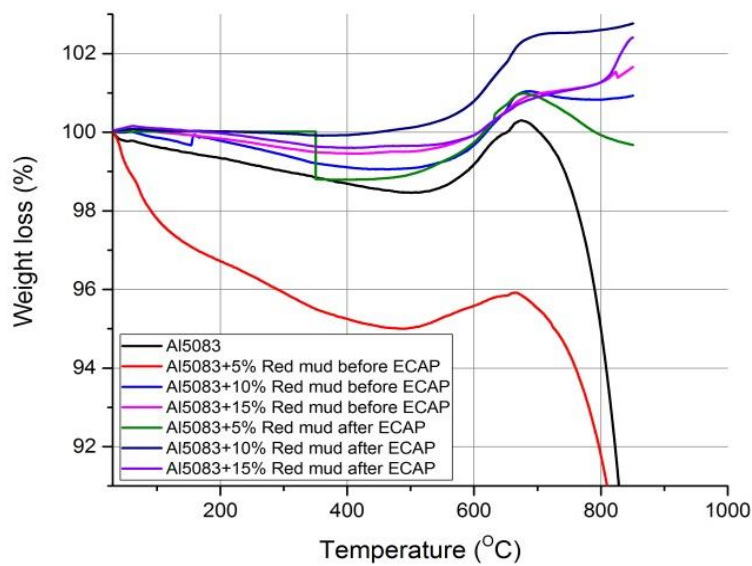
5.2 Mass loss or weight loss:

Thermal analysis was performed on Thermogravimetric Analyzer (STA) 7000 Series apparatus. All the prepared samples (Al-5%Mg and red mud composite) were subjected to TG and DTA to investigate thermal behavior and to analyze the variations with respect to pure Al-5%Mg alloy. The samples (as casted and after ECAP) are subjected to a heat range of 10°C/min under N₂ environment to employ degradation rate and heat difference. The heat difference occurs due to phase transformation that takes place in the material and these phase transformations can be treated as Exothermic, Endothermic and Enthalpy reactions.

The graph 2 shown in TG curves, which indicates weight loss percentage with the observations made from the graphs. In between the temperature in the range of 25 °C to 400 °C the weight loss was observed to be 1.32% for Al-5%Mg alloy and 4.75%, 0.9%, and 0.55% mass loss was observed in 5, 10, and 15wt% of red mud composite before ECAP process. After the ECAP process, the weight loss was observed for three wt % as 1.21%, 0.38, and 0.30% respectively. The gain in mass was observed for the before ECAP specimens is 1.45% at 400°C to 700 °C in Al-5%Mg alloy material and 0.17%, 2.01% and 1.40% in three selected weight percentages of composite before ECAP. Subsequently after ECAP it was observed that the gain in mass is about 2%, 2.85% and 1.29% for the samples. By comparing TG and DTA analysis it can be concluded there is a negligible mass loss in composite material and the loss of mass up to 100 °C may result from vapour, moisture present in the material. In the nitrogen environment of the samples at high temperatures, the gain of mass was due to Mg and Zn present in the composite.

The loss of mass in composite was observed as, a significant loss of mass occurred was in 5% red mud percentage and very negligible mass in 10 and 15% percentages. This is due to the lower thermal conductivity and lower heat of diffusivity of reinforcement (Red mud). It stores the energy at higher temperatures in the melting range, because of Al-Red mud composite having high mass gain is observed at elevated temperature. By doing equal channel angular pressing (ECAP) the grain growth

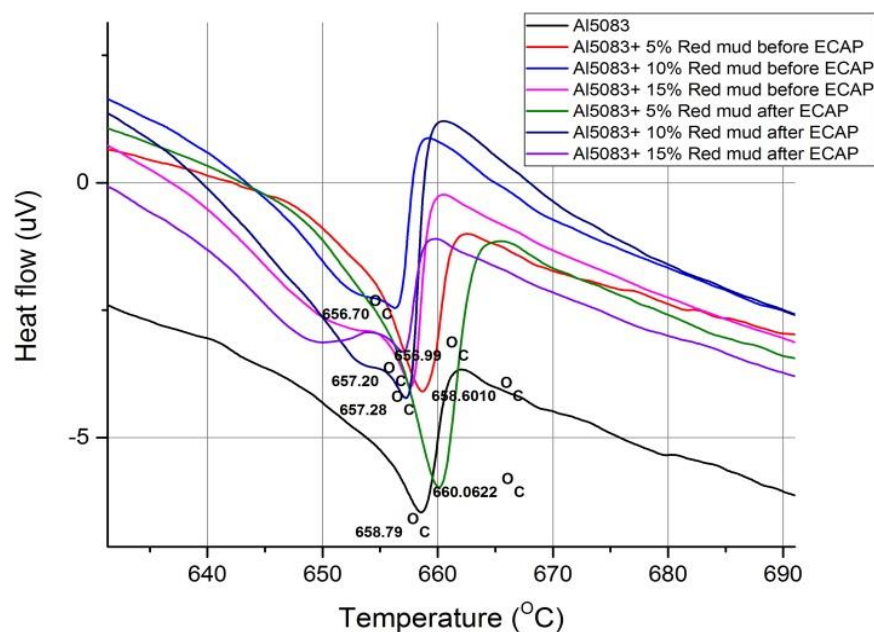
along the material is closely packed so that the mass loss is observed as less and high bonding strength is observed than before ECAPed samples.



Graph 2: Weight loss vs temperature

5.3 Melting point:

The DTA curves are investigated for the composite material as shown in graph 3. The endothermic peaks are obtained in the temperature range of 650 to 670 °C due to phase transformation from solid to liquid (melting). From the TG-DTA graph, it is concluded that there is only 1 °C of variation between all the compositions and Aluminum alloy and it can be observed that by the addition of red mud reinforcement with varying proportions, all the samples of the present composite material are having melting points with a negligible change in the values as furnished in table 2.



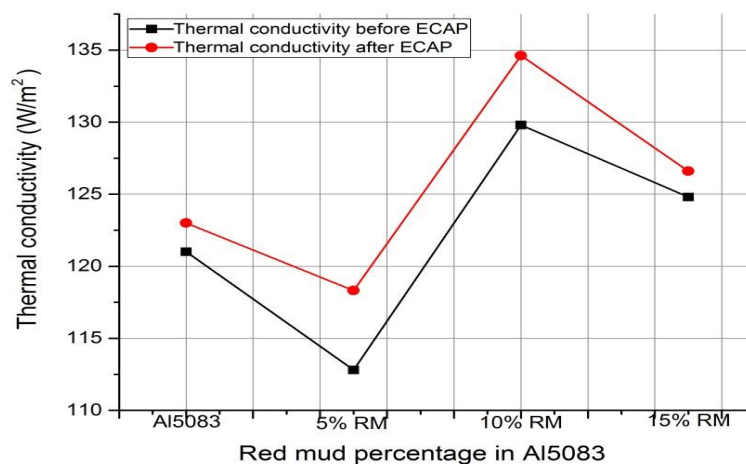
Graph 3: Melting points

Table 2: Melting points of Al-5Mg-Redmud composite

Material	Melting point(°C)
Before Equal channel angular pressing	
Al-5Mg-5%Redmud composite	659.51
Al-5Mg -10%Redmud composite	658.27
Al-5Mg -15%Redmud composite	656.95
After Equal channel angular pressing	
Al-5Mg -5%Redmud composite	660.22
Al-5Mg -10%Redmud composite	657.738
Al-5Mg -15%Redmud composite	665.96

5.4 Thermal conductivity:

The thermal conductivity of the Al508 alloy and three wt% of the red mud composite material are characterized in the Modulated DSC as shown in the graph 4. The thermal conductivity of the base material is 121 W/m² before ECAP and 123 W/m². The variation of the thermal conductivity is observed that initially, thermal conductivity is increased by doing the ECAP process for alloy and composite material. It is also observed that at the 10% of red mud proportion in the matrix material the thermal conductivity is increased and then decreased at 15% of red mud proportion.



Graph 4: Thermal conductivity vs % of red mud

CONCLUSION:

The conclusions drawn from the present investigation are as follows:

1. The stir casting method was employed successfully to prepare composite specimens with Al-5%Mg alloy reinforced with red mud particles.
2. The specific heat of the samples and enthalpy is successfully measured by using differential scanning calorimeter (DSC). Specific heat capacity of Al-5%Mg alloy is more compared to casted and after ECAPED samples. It is concluded that by the addition of red mud particles into aluminum alloy there is a gradual decrement of specific heat capacity (Cp) and Enthalpy (H).
3. The Differential thermal analysis (DTA) is used to find the melting points of the composite. It is concluded that there is no significant effect of red mud content on the melting points of the composite.

4. Less thermal decomposition was observed in the ECAPED samples because of its grain growth due to severe plastic deformation and more mass loss was occurred in as casted composite material.
5. Thermal stability of the ECAPED samples is increased than non-ECAPED samples also it is concluded that where the mass loss is more there will be less stability and vice versa.
6. Thermal conductivity of 10%Redmud reinforced composites shows an increment of 3.1% thermal conductivity value and doing equal channel angular pressing 11.2% increment compared with pure casted Al-5%Mg. From all comparisons reinforcement of red mud into Al-5%Mg at 10% values shows good thermal properties.
7. Al-5%Mg -Red mud composites subjected to ECAP procedure are promising engineering materials and exhibit good thermal properties.

REFERENCES:

- [1] A.K.Dhingra, "metal replacement by composite", *JOM* 1986, Vol 38 (03), p. 17.
- [2] K.Upadhya, "composite materials for aerospace applications, developments in ceramic and metal matrix composites", KamaleshwarUpadhya, ed., warrendale, PA: TMS publications, 1992, pp. 3-24.
- [3] Greg Fisher, "Composite: Engineering the ultimate material", *Am. Ceram. Soc. Bull.* Vol. 63 (2), pp. 360-364.
- [4] T. W. Clyne, An Introductory Overview of MMC System, Types and Developments, in *Comprehensive Composite Materials*, Vol-3; Metal Matrix Composites, T. W. Clyne (ed), Elsevier, 2000, pp.1-26.
- [5] L.M.Manocha& A.R. Bunsell "Advances in composite materials", Pergamon Press, Oxford, 1980, Vol.2, p 1233-1240.
- [6] Berghazan, A. *Nucleus*, 8(5), 1966, (Nucleus A. Editeur, 1, rhe, Chalgrin, Paris, 16(e).
- [7] J.O.Outwater, *The Mechanics of Plastics Reinforcement Tension*, Mod. Plast: March 1956.
- [8] R. Mehrabian, R.G. Riek and M. C. Flemings, "preparation and casting of Metal- Particulate Non-Metal Composites," *Metall. Trans*, Vol. 5A, 1974, pp 1899-1905.
- [9] J. Eliasson and R. Sandstorm, "Applications of Aluminium Matrix Composites," Part 1, G. M. Newaz, H. Neber-Aeschbacherand F. H. Wohlbiel eds., *Trans. Tech. publications*, Switzerland, 1995, pp 3-36.
- [10] John E. Allison, Gerald S. Cole, "Metal Matrix Composites on the automotive Industry: Opportunities and Challenges", *JOM*, Vol. 45(1), 1993, pp 19-24.
- [11] D. J. Lloyd, "Particulate Reinforced Composites Produced by Molten mixing," *High-Performance Composites for the 1990's*, eds. S.K.Das, C.P. Ballard and F.Marikar, TMS-New Jersey, 1990, pp 33-46.
- [12] M.G. McKimpson and T.E.Scott, "Processing and Properties of MMCs Containing Discontinuous Reinforcement", *Mat. Sci. and Engg.*, Vol. 107A, 1989, pp 93-106.
- [13] H.J. Rack, "Metal Matrix Composites", *Adv. Mater. Processes*, Vol. 137 (1), 1990, pp 37-39.
- [14] G. Ramu, "Effect of equal channel angular pressing (ECAP) on microstructure and properties of Al-SiCp composites, *Materials & Design*, Volume 30, Issue 9, October 2009, Pages 3554-3559
- [15] T. Lokesh & US. Mallik, Effect of ECAP process on the Microstructure and Mechanical Properties of Al6061-Gr Composites, *Materials today proceedings*, , Volume 5, Issue 1, Part 3, 2018, Pages 2453-2461
- [16] M.S.Arab, Nahed El Mahallawy, Farouk. Ahmed Shehata & Mohamed Ali Agwa, Refining SiCp in reinforced Al-SiC composites using equal-channel angular pressing, December 2014 *Materials & Design* (1980-2015) 64:280-286