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Particulate Distribution improvement for Al/SiC MMC and Al/MgO MMC Prepared by Modified Liquid State Stir Mixing-Casting

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Abstract: *When a specific* ***reinforcing material*** *is dispersed in a* ***metal matrix****, the resultant is* ***Metal Matrix Composite*** *(MMC). This dispersion can be achieved by powder metallurgy or by casting. The casting technology is cheap, easy, and widely available, but it suffers some technical challenges specially the challenge of achieving a homogeneous distribution of reinforcement particulates through the matrix, where the distribution affects the composite properties. Its common that the Aluminum is one of the best alternatives for its unique availability (8% of the earth's crust), and making composites enables let's say to tailor the properties as required. In this experimental, the composites under investigation are prepared in two formulations. The first one is* ***Al/SiC MMC*** *by addition of* ***Silicon Carbide*** *(****SiC****) particulates to the Aluminum matrix, and the second is* ***Al/MgO MMC*** *by addition of* ***Magnesium Oxide*** *(****MgO****) to the Aluminum matrix. Weight percentages of 5%, 7.5%, 10%, 15% and 20% are applied. The composites are prepared by* ***liquid state mixing*** *technique. To ensure homogeneous dispersion of ceramic particulates in the Aluminum matrix, specimens are taken from different locations in each cast and subjected to microscopic observation after proper preparation where and volumetric fractions are investigated, micro-structural examination and micro-structural analysis are carried out using optical microscope. Tensile, wear, hardness, & impact tests are conducted as well, and then fracture surfaces observation is employed using* ***Scanning Electron Microscope (SEM)*** *equipped with energy dispersive x-ray analysis (EDX). Adding SiC, MgO particulates to the matrix increased the ultimate tensile strength (UTS), and hardness, and decreased elongation (ductility) of the composite compared with those of the pure Aluminum. Increasing weight percentage of SiC, MgO increased its strengthening effect, with higher strength, higher hardness, and finer grain size.*

Keywords: *liquid state mixing, mechanical properties, SEM fracture surface observation, micro-structural examinations, SiC, MgO, Al-MMC, Al/MgO MMC, Al/SiC MMC, MMC.*

I. Introduction

We study composite materials for its importance in the advanced technology, high temperature applications for their high (strength/stiffness-to-weight ratio), more thermal stability, more corrosion and wear resistance, high fatigue life that is required in such applications. Composite technology combines the most important properties of the components together in order to obtain a material with overall properties suitable for the design of the engineering part required.

Composite materials consist of two or more physically and/or chemically distinct phases, suitably arranged or distributed. They have the characteristics that cannot be achieved by any of the components. It is prepared by combining two or more materials in such a way that the resulting material has certain design properties usually improved as desired upon the initial properties.

Aluminum Silicon carbide composite materials are widely used for a huge number of applications such as engineering structures, industry and electronic applications, sporting goods and many other applications. Generally the continuous phase is referred to as the matrix, while the distributed phase is called the reinforcement or additive.

In the last decades, a lot of work was done in this subject where the production advances was highly affected by composites where you can tailor material properties as you need by mixing two different materials without chemical reaction between constituents. Some of composites are of **Metallic Matrices** with ceramics additives where we call this type **Metal Matrix Composite** (MMC), and some are of **Polymeric Matrices** (PMC) and some are of **Ceramic Matrices** (CMC). Most of the studies on metal matrix composites (MMC) paid a lot of attention to Aluminum (Al) as a matrix metal. This trend was for Aluminum's lightweight, environmental resistance and adequate mechanical properties and that what makes Aluminum and its alloys, and

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composites very popular materials and have a lot of applications. The melting point of aluminum is high enough to satisfy many application requirements, where in the same time low enough to render composite processing conveniently. Also, Aluminum can accommodate a variety of reinforcing agents.

Particulate Al-MMCs are reinforced usually with SiC and Al_2O_3. Conventional processing methods include powder metallurgy and molten metal methods [1]. Discontinuous Al/SiC-MMC and Al/ Al_2O_3-MMC have found widespread applications in aerospace, transport, military energy and electric industries, for example, they have been used in electronic packaging aerospace structures, aircraft and internal combustion engine components and a variety of recreational products [2],[3],[4],[5]. A number of other reinforcing materials such as graphite, illite clay, Zirconia etc have been incorporated in Al using molten metal method. The basic limitation of this method is the poor wetability of ceramic particles with liquid Al alloys,[6],[7],[8]. Wetability can be defined as the ability of a liquid to spread on a solid surface, and it represents the extent of intimate contact between a liquid and a solid [9], and this enhances the tendency of reinforcement agglomeration. This represents a great challenge of producing cast metal matrix composites. This would normally result in poor distribution of the particles, high porosity content, and low mechanical properties. For that we need improve the wetability of matrix with additives. Studies proved a high improvement efficiency of SiC wetability in Aluminum by addition of silicon or magnesium.

The retention and distribution of the particulates are very important in production of composites materials. Si addition improves the retention and distribution of SiC within the matrix [8],[10]. Stirring was useful to obtain a range of particulate percentages [11].

The properties of Al-MMC mostly depend on the processing method which is capable of producing good properties to comply the industrial demand. Al-SiC composites can be more easily produced by the liquid sate mixing technique due to its good cast-ability and relatively inexpensive. Jeevan et al. [12], Hashim et al. [13], Neelima Devi et al. [14], Yano et al.[15] reported that the melt stirring method is economical, easy to apply and convenient for mass production. However, the problem encounter for this technique was low wetability and particle settling. To improve wetability and particle homogeneity during casting, various method have been used including coating or oxidizing the reinforcement particles, adding some surface active elements (magnesium and lithium) into the matrix, increasing the liquid temperature and stirring of molten matrix alloy for an adequate time period during incorporation. Study of wear properties of Al-SiC composite was carried out by Singla et al. [16] He found that wear rate decreases linearly with increasing SiC.

J. Hashim et al. studied the improvement of wetability by using clean SiC particles and magnesium as a wetting agent, and stirring continuously while the MMC slurry is solidifying were found to promote wetability of SiC with A359 matrix alloy. Decreasing this solidification time was also found to improve the wetability whereas increasing the volume fraction of SiC particles present will give the opposite effect [17].

Sajjad et al. employed a new method for uniform distribution of very fine SiC particles with average size of less than 3 μm was employed. The key idea was to allow for gradual in situ release of properly wetted SiC particles in the liquid metal. For this purpose, SiC particles were injected into the melt in three different forms, i.e., untreated SiC_p, milled particulate Al– SiC_p composite powder, and milled particulate Al–SiC_p–Mg composite powder. The resultant composite slurries were then cast from either fully liquid (stir casting) or semisolid (compo-casting) state. Consequently, the effects of the casting method and the type of the injected powder on the microstructural characteristics as well as the mechanical properties of the cast composites were investigated. The results showed that the distribution of SiC particles in the matrix and the porosity content of the composites were greatly improved by injecting milled composite powders instead of untreated-SiC particles into the melt. Casting from semisolid state instead of fully liquid state had similar effects. The average size of SiC particles incorporated into the matrix was also significantly reduced from about 8 to 3 μm by injecting milled composite powders. The ultimate tensile strength, yield strength and elongation of Al356/5 vol.% SiC_p composite manufactured by compo-casting of the (Al–SiC_p–Mg)c_p injected melt were increased by 90%, 103% and 135%, respectively, compared to those of the composite manufactured by stir casting of the untreated-SiC_p injected melt [18].

W. Zhou et al. studied a composites based on two aluminum alloys (A536 and 6061) reinforced with 10% or 20% volume fraction of SiC particles were produced by gravity casting and a novel two-step mixing method was applied successfully to improve the wetability and distribution of the particles. The SiC particles were observed to be located predominantly in the inter-dendrite regions, and a thermal lag model is proposed to explain the concentration of particles [19].

K. R. Suresh et al. studied tensile and wear properties of aluminum composites fabricated by squeeze casting method and checked uniform particulate distribution. The squeeze cast composites show peak strength of 216 MPa showing an increase of 11.6% in tensile strength. The new composites also have improved wear resistance when compared to gravity cast composites. [20]. Elimination of casting defects such as pores and non-uniform distribution of the particulates is essential in improving the properties of the composite materials [11],[21].

The matrix should bond strongly with the reinforcement but should not be chemically affected by adverse reactions. Proper matrix and reinforcement selection will promote part formability by various processes [22]. Reinforcement, as either continuous or discontinuous may constitute 10 to 60 vol % of the composite [23].

The first aim in this study is to make the Al-MMC using a modified liquid state mixing called stir casting method by employing the ceramic particulates of MgO, Si, SiC to aluminum in the liquid state. Many trials are made to determine heating temperature and casting temperature so that we can gather two opposing targets:

1. High enough temperature to have proper time to add the additives and mix them properly and then cast them before aluminum solidification starts.
2. As low as possible to avoid burning the most of the additives before starting their mixing with the matrix. Not to take a lot of time to solidify after casting so that the particulates may settle in the bottom of the mould due to gravity.

The second aim in this study is to investigate the effect of the additives on aluminum alloy highlighting their merits and demerits. Major issues like agglomerating phenomenon, fiber-matrix bonding and the problems related to distribution of particles are also investigated. The effect on the mechanical properties like tensile strength, strain, hardness, wear, and stiffness will be studied. To do so, properly mixed and casted specimens are subjected to various mechanical tests and micro-structural observation.

II. Experimental Work

The reinforcement particulates are prepared by using **ball mill** then sieving to separate the desired particle size which is as found in previous work in literature to be 50 μm. Aluminum and all the prepared additives are weighted and prepared according to the suitable percentages. Then, Aluminum for each cast is melted by using an electric resistance furnace in a ceramic crucible. It is heated initially to 900 °C (220 °C higher than Aluminum melting point so that time before solidification is enough for mixing and casting and it is found convenient to compensate for the temperature drop during transferring the crucible from the furnace to the mixing apparatus and temperature drop while mixing the particulates). Probably, this temperature is determined after many trails for various temperatures.

Ceramic particles previously weighted for each cast are heated to 300 °C before addition into the molten Aluminum to avoid high drop in temperature just after particulates addition. Also, all the additives; Si, and the degasser. Heating all the additives found to be helpful in giving extra time for impeller to stir the mixture well and cast the aluminum before solidification and hence more homogeneous dispersion for the particulates in the produced composite.

Then the crucible is taken from the furnace to the composite mixing apparatus as shown below in the schematic in figure 1 which consists of: a speed adjustable **drill press tool machine**, a steel rod with a welded steel impeller at its end is fixed to the drill press tool post to stir the molten Aluminum in the crucible.

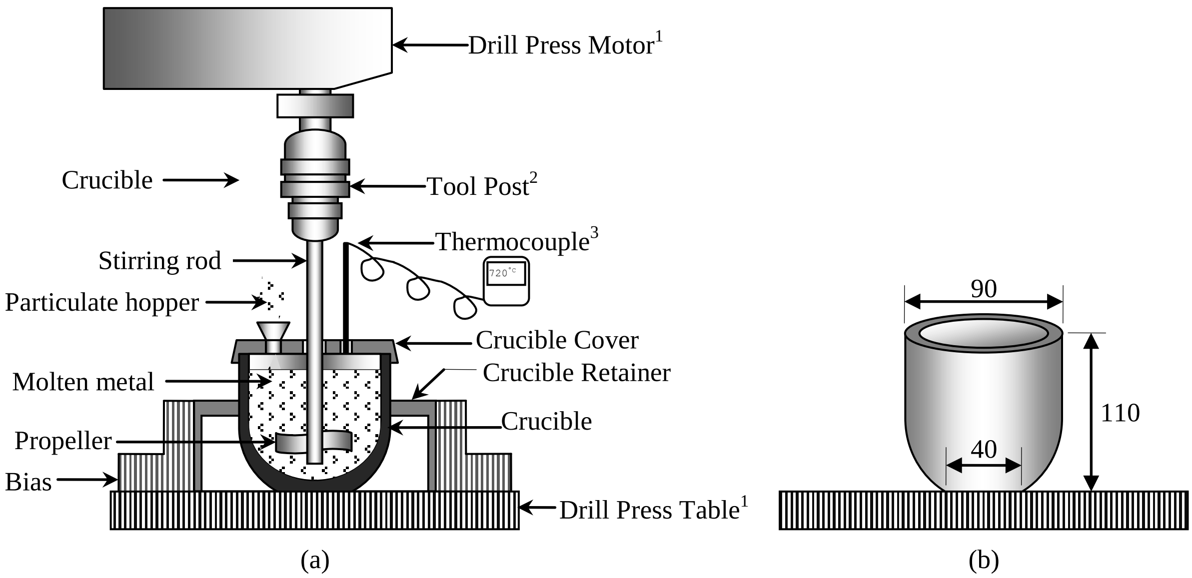

Figure 1: Liquid state mixing schematic

The steel cover for the crucible is provided to avoid molten Aluminum splashing during the impeller rotation while mixing and to avoid particulate blowing as it is powder like. Three holes are made in the steel cover to insert the stirring rod, the thermocouple, and to drop the particulates inside the crucible while mixing. Also, a stand of steel plate is designed to retain the crucible firmly on the drilling machine table during mixing and it is fixed by the tool machine bias. The drill press is operated at 450 RPM to start mixing.

Firstly, for the Al/SiC MMC casts previously weighted and heated 10 wt. %Si is added to the melt while stirring to improve wetability of the particulates with aluminum[2]. Secondly, pre-heated ceramic particulates are added in the suitable amount to achieve the desired percentage for each cast. Stirring process is continued to make the ceramic powders uniformly dispersed through the matrix (the liquid Al) before casting into the metallic mould.

The temperature of the melt is monitored during mixing by the thermocouple until temperature reaches 700 °C, then the degassing powder is added to remove gasses from the melt[3] and stirring is continued for seconds then the molten mixture is poured carefully in a metallic mould of dimensions (150 x 80 x 10 mm). After waiting 10 minutes after casting to ensure complete solidification, the metallic mould is opened and the cast is removed.

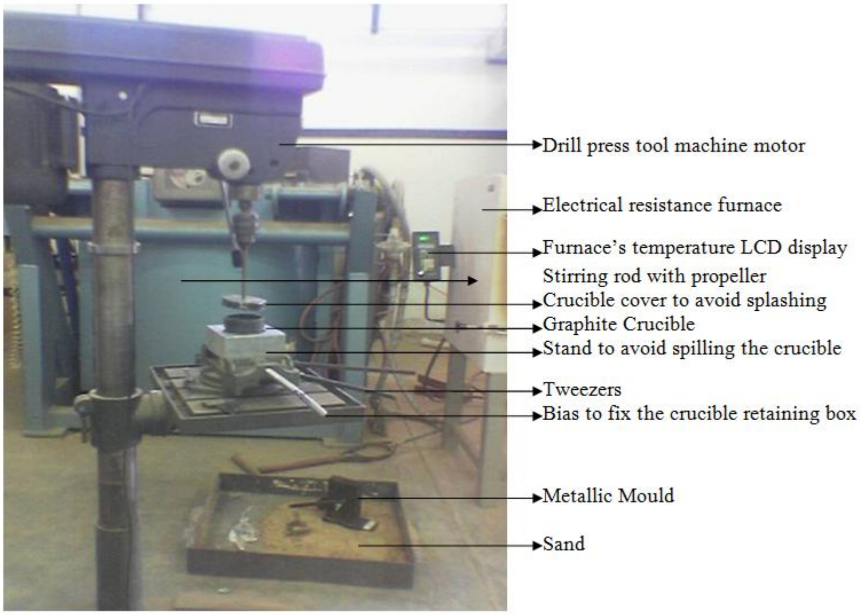

Figure 2: Liquid state stir mixing-casting

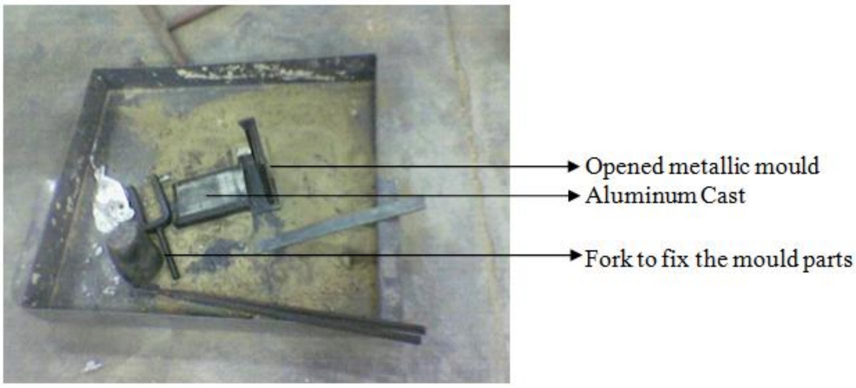

Figure 3: Metallic mold opened

Specimens for various tests are prepared from each melt as shown in figure 4.

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[2] No need for Si addition for Al/MgO MMC were MgO has a good wettability with Aluminum.

[3] The degassing powder is previously weighted and put in a closed small aluminum foil to facilitate its entry to the mixture because it is too fine and has difficulty in the addition to the mixture specially the mixtures temperature is relatively low at the end of mixing, while the mixture is thicker.

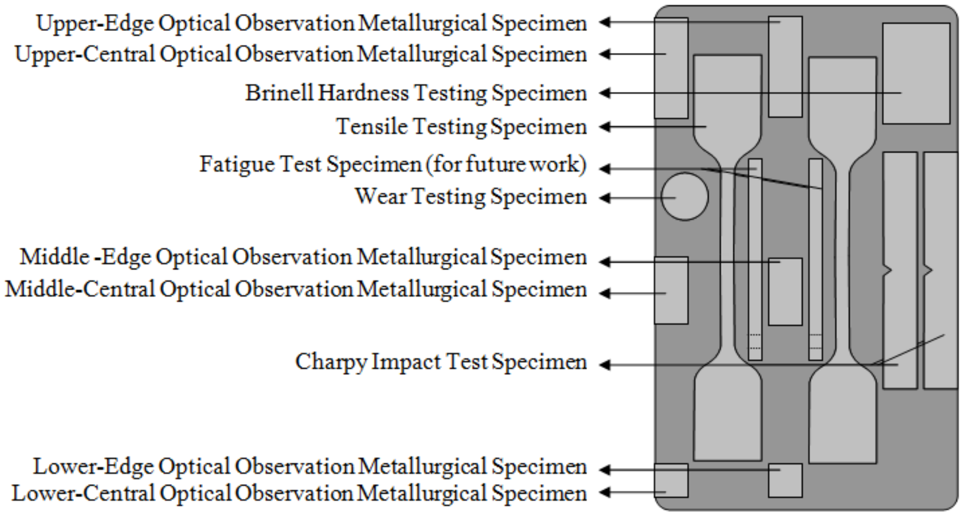


Figure 4: Specimens prepared for each cast

Details for all of the melts are shown in table 1.

Table 1: Detailed percentages for all of the cast groups prepared

Group	Alloy	Group	Alloy
1	Pure Aluminum		
2	Al–5wt%MgO	7	Al–10wt% Si, 5wt%SiC
3	Al–7.5wt%MgO	8	Al–10wt% Si, 7.5wt%SiC
4	Al–10wt%MgO	9	Al–10wt% Si, 10wt%SiC
5	Al–15wt%MgO	10	Al–10wt% Si, 15wt%SiC
6	Al–20wt%MgO	11	Al–10wt% Si, 20wt%SiC

For microstructural observation, the constitutional metallographic technique is utilized. The volume fractions of the particulates and their distribution, and the grain size of the Al matrix are determined. Also, tensile test samples are prepared according to ASTM-E8-95a and impact test samples are prepared according to the ASTM E23. Chemical analysis of the samples are done using EDX system attached to SEM, Model XL-30W/TMP Philips.

III. Results And Discussion

1- Microstructure

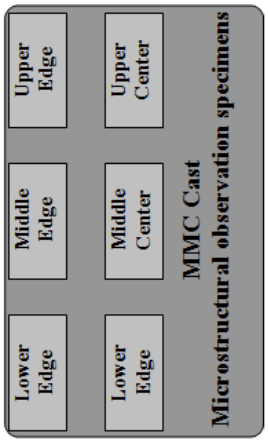
Volume Fraction

Volume fractions (V_f) of SiO and MgO particulates are determined using the network of 1 mm square grid fixed on the photograph of each microstructure. The points that lay on the particulates are counted & this value is divided by the total grid points from which volume fraction of particulates is obtained. Horizontally and longitudinally distributed specimens are taken as shown in figure in table 2 to ensure uniform distribution of particulates in matrix.

For validation, an optical microscope is utilized to determine the particulate volume fractions (V_f) again, and their distribution in the casting. Also, the grain size of the Al matrix. 1 mm square grid attached to the microscope eye piece at x50 magnification is used for V_f determination [26]. V_f was calculated from various parts of the cast and the results are very close in the two techniques and they are shown in table 2 and table 3. The results indicate that the V_f across the casting, horizontally and vertically are quite uniform at various parts of the cast, and the variation of V_f in horizontal direction is less than that in vertical direction as shown in figures 5 and 8. These results indicate that the method used in the composite preparation is good enough for relatively uniform distribution and this is reflected on the morphology within the matrix as shown in table 3.

Table 2: Detailed actual additive percentages as volume fractions for all the constituents of the cast groups prepared. Volume fractions are measured through the specimens horizontally and longitudinally.

Additive %	Actual Percentage / Constituents	Actual Additive volume fractions, %					
		Upper Edge	Up Center	Mid Edge	Mid Center	Low Edge	Low Center
5	Al-10wt% Si, 5wt% SiC	4.4	4.3	4.2	4.2	3.7	3.6
7.5	Al-10wt% Si,7.5wt% SiC	6.6	6.5	6.3	6.3	5.8	5.9
10	Al-10wt% Si,10wt% SiC	8.8	8.7	8.3	8.3	7.5	7.4
15	Al-10wt% Si,15wt% SiC	13.4	13.1	12.6	12.6	11.2	11.5
20	Al-10wt% Si,20wt% SiC	17.5	17.1	16.8	16.8	15.8	15.1
5	Al-5wt% MgO	4.7	4.5	4.4	4.5	4.8	4.7
7.5	Al-7.5wt% MgO	7.3	6.9	7.1	6.8	7.8	7.6
10	Al-10wt% MgO	9.6	9.5	9.3	8.9	9.6	9.4
15	Al-15wt% MgO	14.2	14.1	13.5	13.5	12.7	12.8
20	Al-20wt% MgO	18.9	18.7	17.8	17.9	18.2	17.9



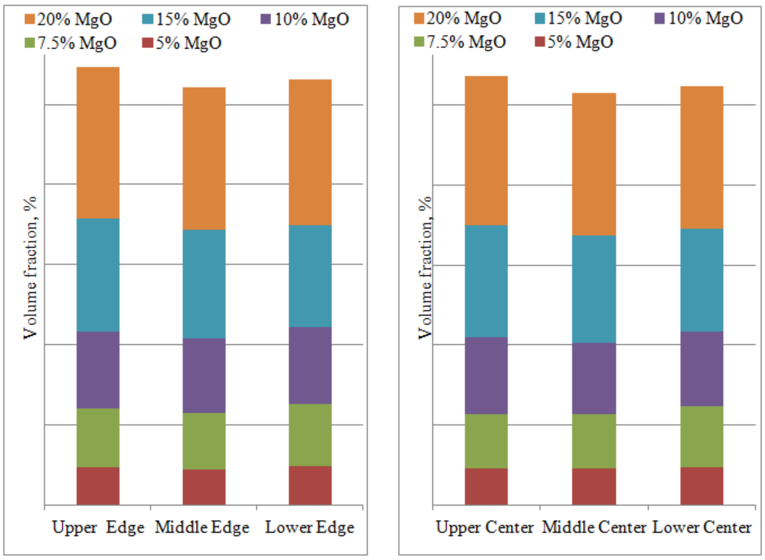


Figure 5: Measured Volume Fraction versus Particulates Weight Percentage for Al/MgO MMC

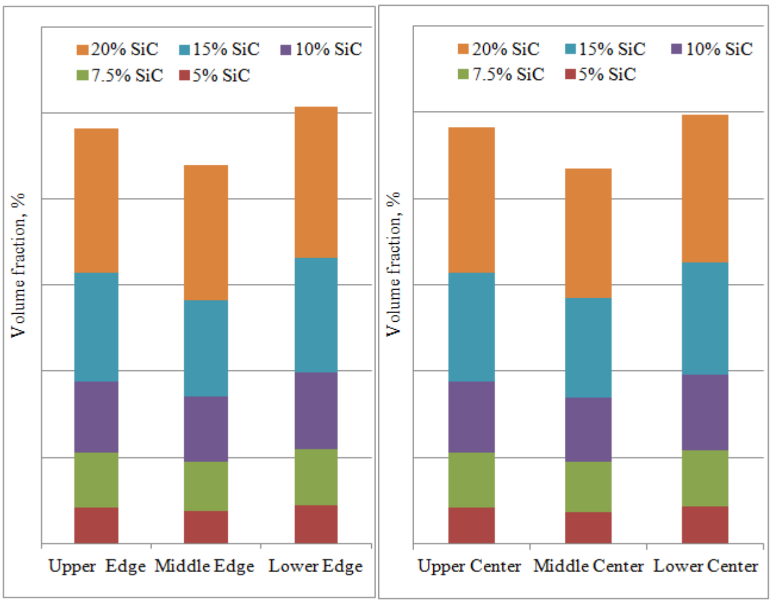


Figure 6: Measured Volume Fraction versus Particulates Weight Percentage for Al/SiC MMC

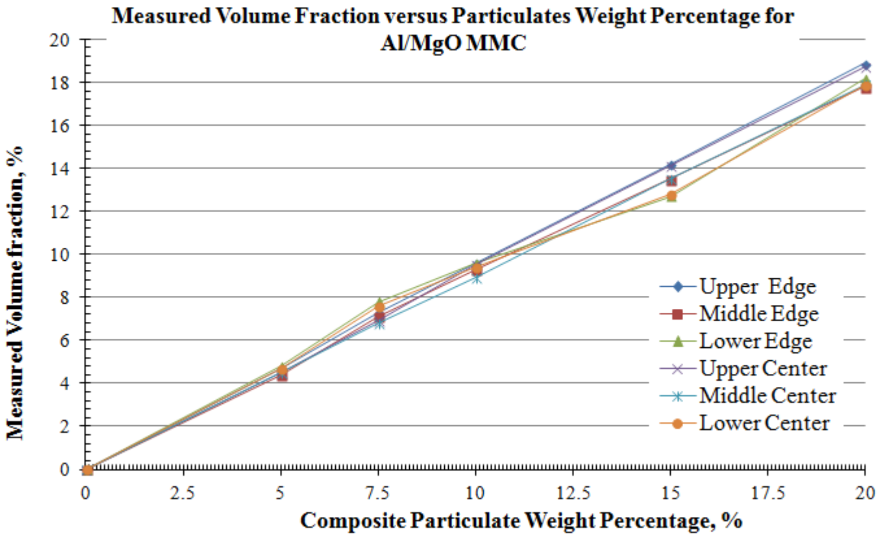


Figure 7: Measured volume fraction versus particulates weight percentage for Al/MgO MMC

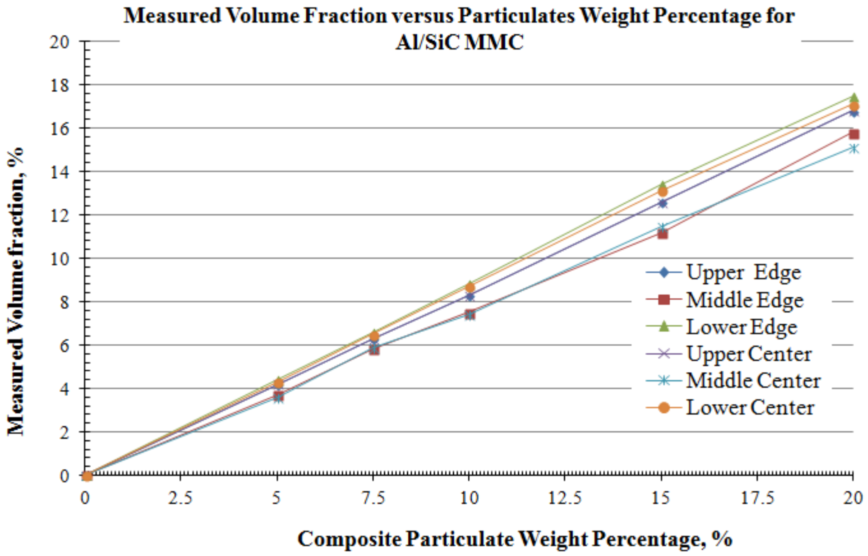


Figure 8: Measured volume fraction versus particulates weight Percentage for Al/SiC MMC

The grain size of the Al matrix was measured by mean lineal intercept method from the same specimens used for V_f determination. The average grain sizes are given in table 3. The average grain size for pure Al in as-cast condition was 786 µm. Addition of the particulates decreased the grain size at rates shown in table 3. The largest decrease was noticed at initial additions of the particulates, namely at 5 wt% in all cases, then the decrease was tapered and at 20 wt% particulate additions the grain sizes were almost constant. The composite grain sizes are finer than that of the monolithic matrix. This refinement is caused by the ceramic particulates acting as nuclei for the grain formation during solidification and at the same time these particulates would inhibit the processes of the grain growth [27]. Similar observation was seen by [28] where 20% Al_2O_3 addition produced 90% reduction in the grain size of the composite.

Table 3: The average grain size measured for each constituent and average grain size reduction

Constituents	**Average Grain Size (□m)**	**Average Grain Size reduction,**	**Constituents**	**Average Grain Size**	**Average Grain Size**
Pure Al	786				
Al-10wt%Si, 5.0wt%SiC	106	86.6	Al-5wt%MgO	167	78.8
Al-10wt%Si, 7.5wt%SiC	107	86.4	Al-7.5wt%MgO	123	84.4
Al-10wt%Si, 10wt%SiC	79	90	Al-10wt%MgO	115	85.4
Al-10wt%Si, 15wt%SiC	63	92	Al-15wt%MgO	87	89
Al-10wt%Si, 20wt%SiC	58	92.6	Al-20wt%MgO	74	90.6

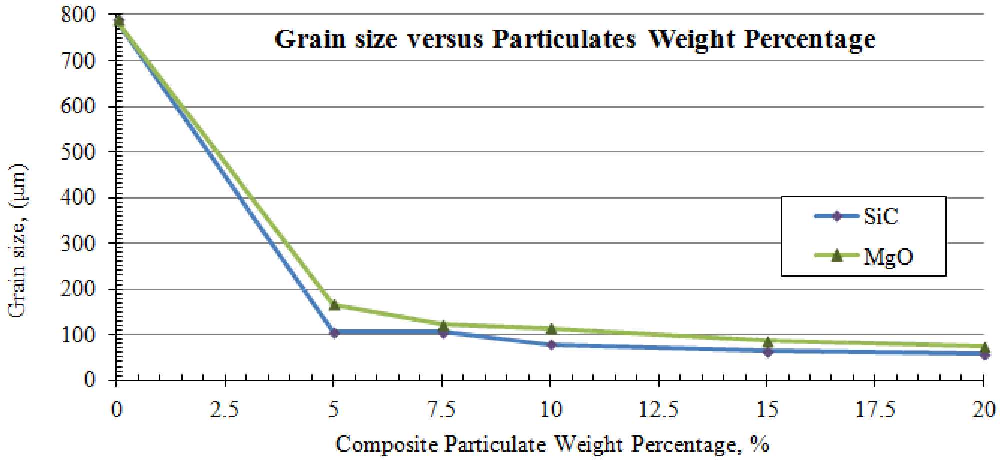


Figure 9: Effect of particulates percentage on g rain size

2- Mechanical properties

a- Tensile test (strength and strain)

All tensile tests are carried out on Universal Tensile Testing Machine (Dartic). Figures 10 and 11 show the stress-strain curves for all of the composite constituents prepared in this investigation. These curves are constructed using load-elongation diagrams printed by the tensile testing machine.

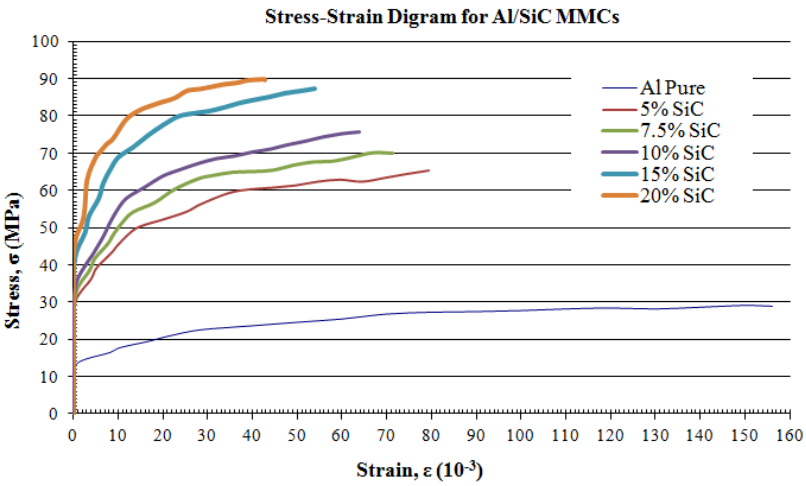


Figure 10: Stress-Strain Diagram for Al/SiC MMCs

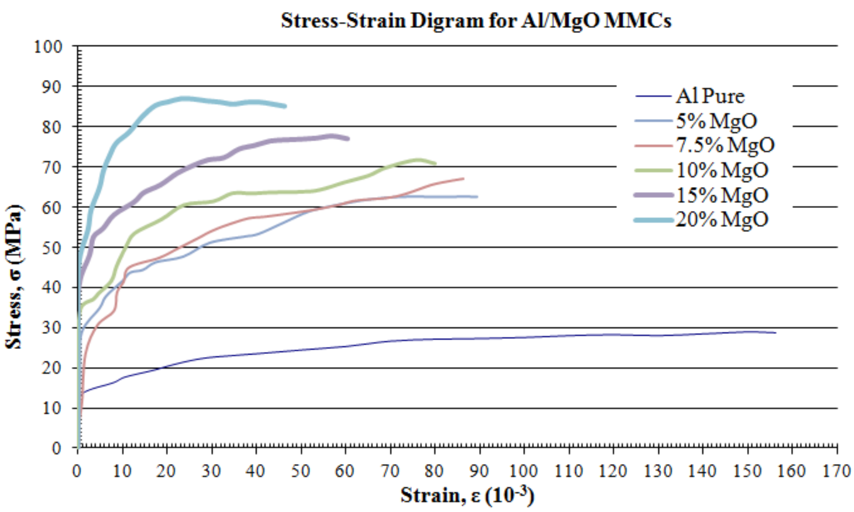


Figure 11: Stress-Strain Diagram for Al/MgO MMCs

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Ultimate Tensile Strength

The effect of ceramic particulates at different percentages on the ultimate tensile strengths (UTS) is shown in figure 11. The results show that the UTS increase in all the constituents with increasing wt% of the ceramic particulates, Al-SiC MMC has larger increase when compared with Al-MgO MMC. This indicates a high effectiveness of the particulates in strengthening Aluminum.

The UTS of the composites is increased by 2.26-3.10 for Al-SiC, and 2.16 -3.10 for Al-MgO over the corresponding values of the as cast pure Al at ambient temperature as shown in figure 12. Also, an increase in yield strength can be noticed as shown in figure 13.

The increase in UTS of the composites is accompanied by a decrease in strains (ductility) with increasing wt% of the ceramic particulates as shown in figure 14. The lowest strain was observed in case of Al-SiC MMC, so the retention of the particulates confers an overall embrittling effect on the composites. The increase of UTS of the composites over the pure Al matrix can be related to the interaction between the particulates and dislocations within the matrix, and to the grain refinement of Al with increasing addition of the particulates.

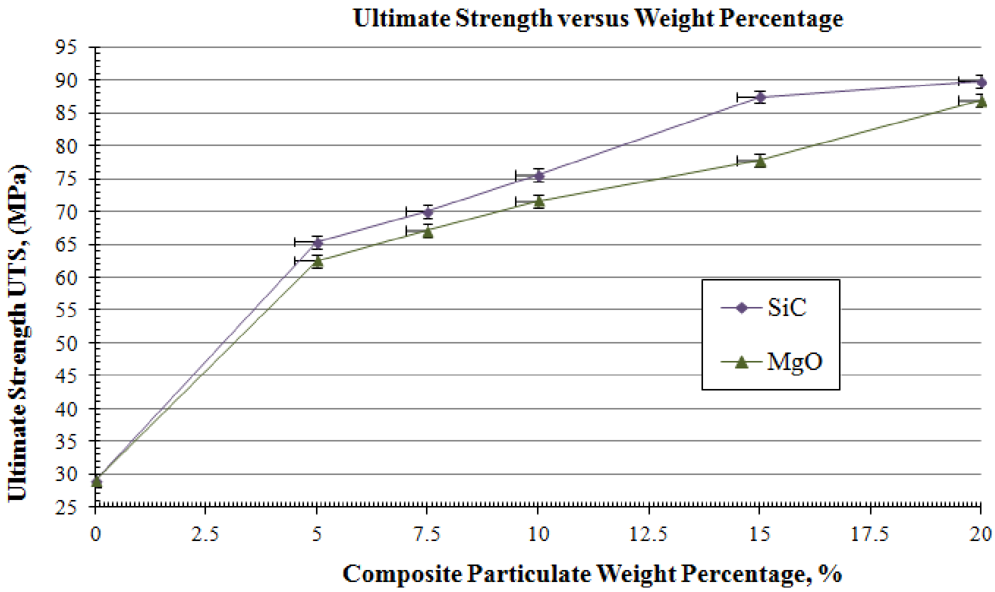


Figure 12: Effect of particulates (additives) percentage on ultimate tensile strength

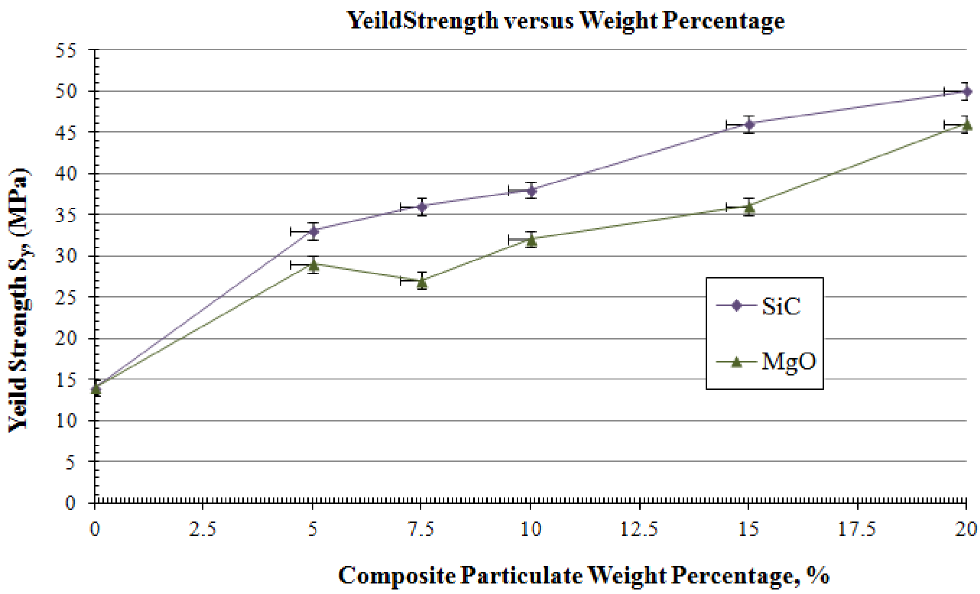


Figure 13: Effect of particulates (additives) percentage on yield strength.

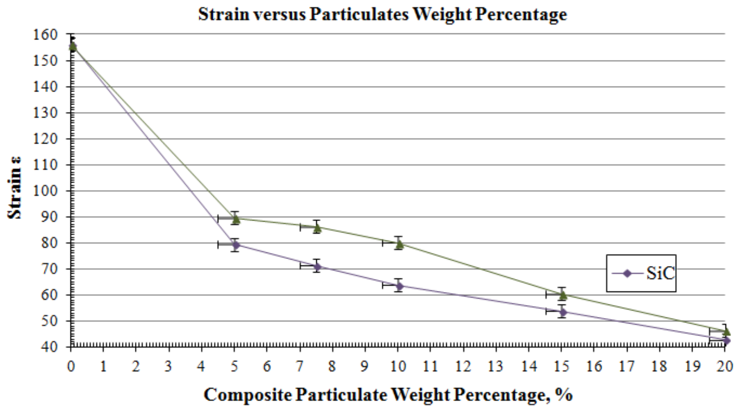


Figure 14: Effect of particulates (additives) percentage on strain.

b. Stiffness

A slight increase in stiffness is noticed as shown in figure 15 where SiC has higher stiffening effect than MgO.

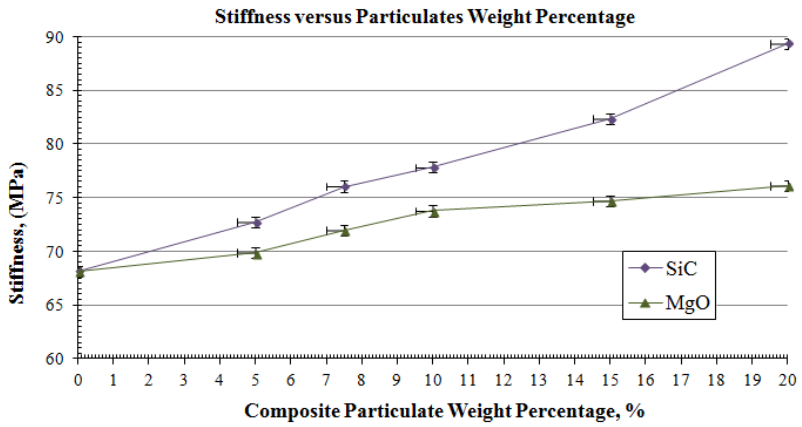


Figure 15: Effect of particulates (additives) percentage on stiffness.

c- Impact Test

Specimens of each group are prepared for Charpy Impact Strength test using Roell Amsler impact strength machine, ASTM standard test method of impact testing of metallic materials at room temperature is employed, and the impact values are determined.

Figure 16 shows the variation of the impact strengths (a_k) in Joules (J) for various Al-MMCs with different wt% of the particulates. The results show that the a_k value for Al-MgO MMCs increased slightly in general by increasing wt% of the particulates while in Al-SiC MMC the a_k decreased slightly by increasing wt% of SiC particulates.

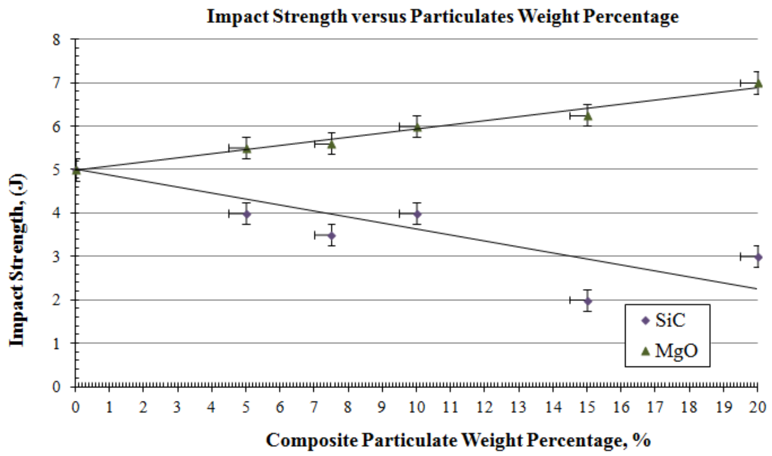


Figure 16: Effect of particulates (additives) percentage on impact strength.

d- Hardness Test

The results of the Brinell hardness measurements are shown in figure 17. It increases with increasing wt% of the particulates used in this work. These increases can be related -as mentioned before- to the interaction of the dislocations with the particulates and grain refinement with increasing wt% of the particulates.

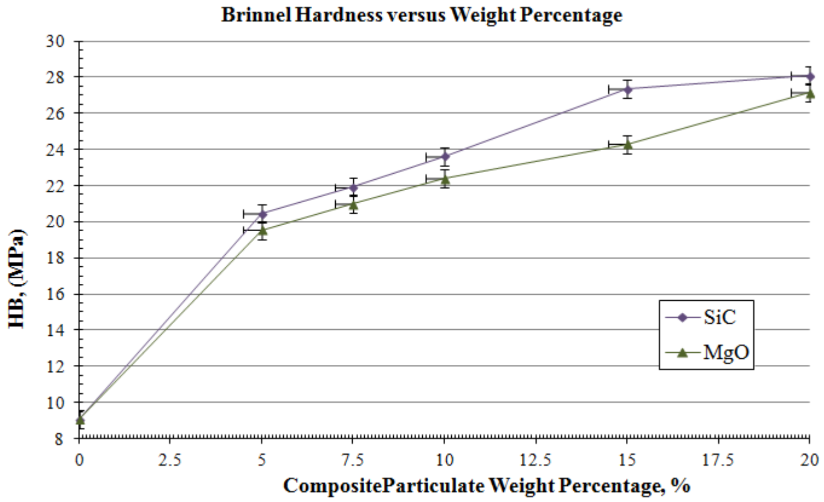


Figure 17: Effect of particulates on hardness.

3- Scanning Electron Microscopy (SEM)

Chemical Analysis (EDX)

As shown in figure 18, the chemical quantification of the pure aluminum we used in our work to ensure that its chemical composition is within the applicable range.

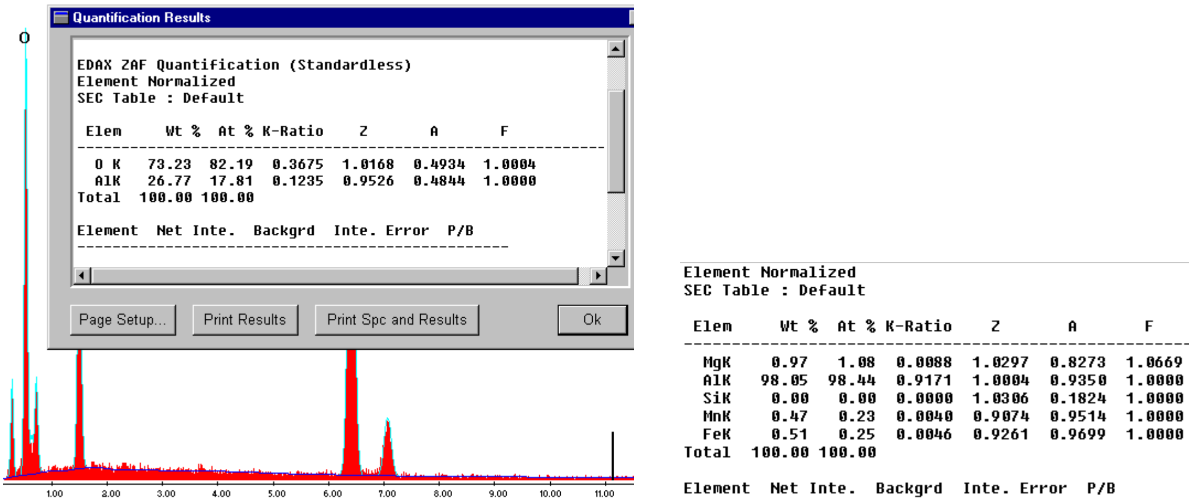
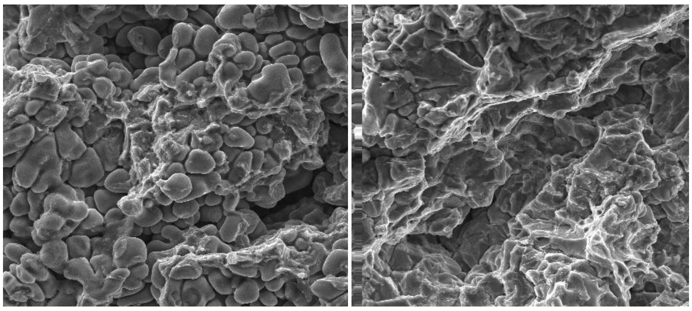


Figure 18: Chemical composition of matrix.

Fractography:

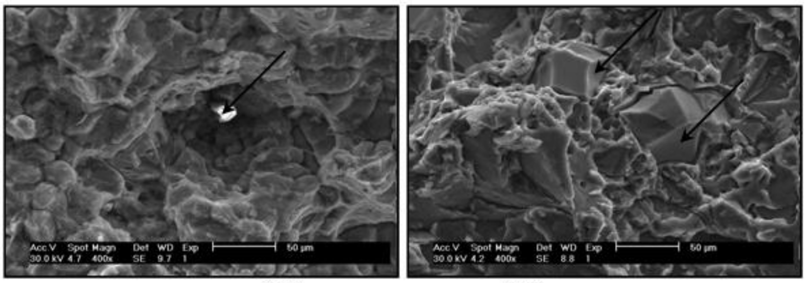
SEM observations of fractured matrix of pure aluminum is shown in figure 19. The fractured surface consists of dimpled morphology, revealing ductile fracture of the matrix. The fracture surface of the composites reinforced with SiC particulates essentially consist of a bimodal distribution of dimples. SEM observations of fractured matrix of 20 wt% SiC MMC is shown in figure 20. The dimples of large sizes are associated with the particulates and the smaller ones are associated with ductile fracture of the matrix. In many cases the fracture surface of the particulates show smooth surfaces indicating that the particulate has fractured rather than de-cohered, where in many cases a whole particulate surface where observed which means that high and low interfacial strengths exists in these composites and the composites failed through particulate fracture and particulate de-cohesion then matrix ligament rupture.

The fracture surface of the composites reinforced with MgO particulates essentially consist of a bimodal distribution of dimples. SEM observations of fractured matrix of 20 wt% MgO MMC is shown in figure 22. The needle like fractured particulates are observed (Higher magnification is provided in figure 23 to show the needle like particulates. In most cases the fracture surfaces of the particulates show smooth surfaces indicating that the particulate has fractured rather than de-cohered, which means that high interfacial strengths dominate in these composites and the composites failed through particulate fracture and matrix ligament rupture. Also, a partial fusion in the MgO particulates is also observed and assigned to the melting and casting process.



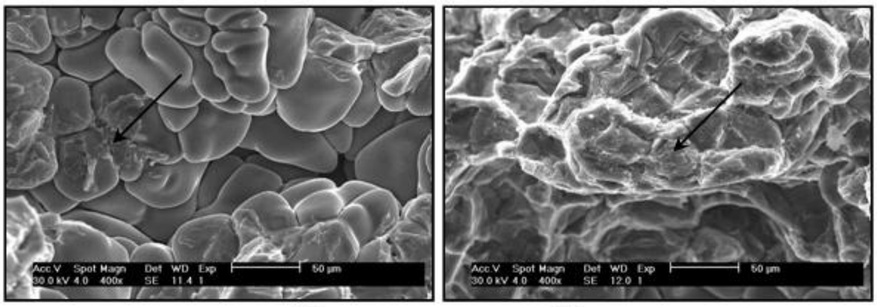
(a) **(b)**

Figure 19: SEM fracture surfaces of pure Al at X200. (a) Tensile. (b) Impact.



(a) **(b)**

Figure 20: SEM fracture surface photographs of Al-20%SiC MMC at 400X.
(a) Tensile (b) Impact.



(a) **(b)**

Figure 21: SEM fracture surface photographs of Al-20%MgO MMC at 400X.
(a) Tensile. (b) Impact.

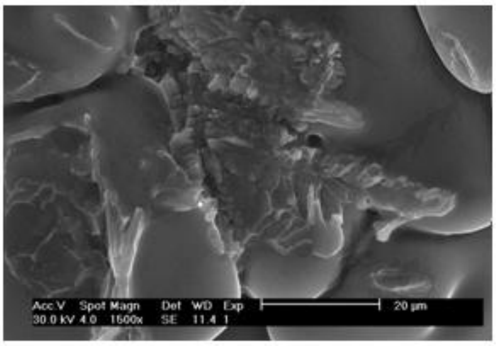


Figure 22: Higher magnification SEM photograph of MgO needle-like structure in Al-20%MgO MMC at 1500X.

IV. Conclusion

Adding SiC, and MgO individually at different percentages to aluminum matrix composite using modified stir mixing casting to achieve homogeneous distribution along the matrix resulted in:

- MMCs grain size is highly reduced as its compared with grain size of the initial matrix grain size, where the particulates within the matrix act as grain nucleation cites.
- A significant increase in the MMC yield strength, ultimate tensile strength (**UTS)** and hardness.
- A significant decreases in the MMC ductility.
- The improvement of mechanical properties by particulate addition needed homogeneous distribution and that needs a good particles wetability with matrix material.
- Si addition to matrix before SiC addition improved wetability and hence, facilitated homogeneous distribution.
- Increasing wt% of SiC, and MgO increases their strengthening effect.
- SiC have higher strengthening effect than MgO, for higher strength, hardness, and grain size reduction.
- SiC addition has higher decreasing effect on ductility.
- MgO addition increased toughness and impact strength where SiC addition decreased toughness and impact strength.
- Particulates failed mainly through particulate decohesion followed by ductile failure of the matrix in the composite reinforced with SiC. Also, a little particulate fracture was observed.
- The composite reinforced with MgO, particulates failed mainly through particulate fracture.

V. Recommendations

Some of the worthwhile investigating parameters:

- Study the effect of stirring speed, stirring time & mixing impeller angle on MMC's homogeneity & mechanical properties. Stirring speed allows vortex formation, & hence penetration of air to the melt increasing oxidation, where low speed decreases stirring efficiency in mixing.
- The effect of addition of more than one additive on MMC's mechanical properties.
- Study the effect of application of secondary plastic deformation process on the mechanical properties.

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