ANALYSIS OF PROPERTIES FOR SiC, Al_{2}O_{3} And MgO As REINFORCEMENT KEEPING PARTICLE SIZE At 0.220 In (A384.1)_{1-x} [(REINFORCEMENT)]_{p}x

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ABSTRACT
The work reports on keeping the reinforcement of Silicon carbide (SiC), Alumina and Magnesium Oxide (MgO) particle size constant at 0.220 µm with A384.1 Al alloy as matrix and particulate composites composition for x=0.0 to x=0.10 by stir casting technique. The results have been explained in terms interfacial characteristics and micro-structural aspects of reinforcement and matrix alloy.

Relatively greater degree of agglomeration of SiC, Al_{2}O_{3}, MgO particles based composites is noted as compared to un-reinforced Al alloys. As the density increases, the MMC becomes stiffer since the molecules do not have as much space to move around one another. It was observed that the value of porosity is found to increase with reinforcement. By increasing the percentage of reinforcement in as-cast and peak aged conditions, value of micro hardness keeps on increasing. In the tensile measurements for SiC, Al_{2}O_{3} and MgO based metal matrix composites with varying percentage of ‘x’, it is clear that composite x=0.10 exhibits highest values of 0.2% proof stress (403 MPa) for MgO and UTS (449 MPa) for SiC among all the peak aged conditions composites fabricated. The peak aged conditions composites reinforced samples were found to have 0.2% proof stress and UTS values larger than the unreinforced alloy.

KEY WORDS: MMCs, Composites, Stir casting, microhardness etc.

1. INTRODUCTION
The Al/Al Alloys are made to control the physical properties, formability and strengthening of the metal. Ageing treatment has been used as an effective method for obtaining stabilized microstructure and improvement in micro and macrohardness. Efficiency of the composites depends on the shape, size and volume fraction of the reinforcement in Aluminum alloys, distribution of particles and ageing temperature. Aluminum metal matrix composites (MMCs) are in use because of their improved physical and mechanical properties. MMCs are used with improved engineering materials properties in aerospace, defense, automotive and consumer market [6]. Aluminum composite having low density has also gained considerable attention because of the porous nature and excellent combinations of physical properties of the aluminum are influenced by chemical composition and microstructure of matrix with which it is made of the process and microstructure. However, detailed studies on the ageing behavior of Al/Al Alloys and their composites are in question. Dislocation and excessive vacancies, introduced in the MMCs due to thermal mismatch strain between matrix and reinforcement during quenching increases the precipitation kinetics vis-a-vis ageing kinetics. Whether, it is also applicable to the A384.1 Al Alloy has not been investigated so far. The materials like B_{4}C, SiC, MgO, fly ash and Al_{2}O_{3} etc. lead to grain refinement of aluminum alloy and these could be used for manufacturing MMCs with improved properties. However, the ageing behavior of A384.1 Al Alloy with different composites with SiC, MgO and Al2O3 addition has also not been explored [4-6]. A number of systemic studies have been done on these MMC’s but there are certain gaps that require immediate attention. These include optimized fabrication methodology, interfacial effects between reinforcement and metallic matrix, micro-structural stability aspects versus improvement in composite material characteristics and age hardening impacts on the material. In this work, ageing behavior of (A384.1)_{1-x} [(reinforcement)]_{p}x composites with and without reinforcement addition has been compared. These have been further compared with that of A384.1 Al Alloy and ageing behavior of the investigated material was determined by measuring the micro and macro-hardness as a function of ageing time.

2. DESIGN OF EXPERIMENT
Keeping in view the above mentioned study, we have evaluated a number of methods reported in literature for fabricating Al alloys composites but due to high cost of manufacturing many of them have limited use. Melt stir casting technique is found to be the simplest and most economical fabrication method for these materials. In earlier studies, stirring of the melt has been done in open air or using a furnace having provision to create an
inert environment. A simple modification of the conventional technique as proposed by Surappa & Rohatgi [3][10] leads to remarkable improvement in this method. Besides the other components required in the technique, we have an additional steel cover fitted with glass wool lining to make an inert atmosphere in order to prevent reaction of aluminum with environmental gases.

The A384.1 Al alloy is used as the matrix and the reinforcement of $\text{Al}_2\text{O}_3$/MgO/SiC with grain sizes of 0.220 $\mu$m had been used with varying contents ($x=0$ to $x=10$). The grain size was determined using Sigma Scanpro Image Analyser™. The densities of the samples have been determined by standard Archimede’s principle. The porosity has been determined by knowing the difference between the theoretical and measured densities. Mechanical Properties like hardness, tensile strength, compressive strength etc. had also been determined. In order to investigate the microstructure-processing-property correlation the extruded samples of the fabricated MMC’s were prepared using hot-extrusion at $420^\circ C$ with an extraction ratio of 27:1 at the speed of 2 m/min. For ageing studies, heat treatments of unreinforced and reinforced samples is carried out in air at $530^\circ C$ using a convection furnace. After 1.5 hours the samples were taken out and quenched in water at room temperature then the samples were transferred within one minute to a container maintained at a temperature of $162^\circ C \pm 2^\circ C$ inside a furnace, to investigate the ageing effect by varying retention time from half an hour to twenty two hours. Mechanical Properties of the composites were then studied using Vickers Micro-hardness tester, MATSUAWA-MMT-X7, Japan™ at a load of 25 gm and Brinell Macro-hardness (BH) tester, Indentec Hardness Tester™ on the as-cast and peak-aged conditions at a load of 62.5 Kg [15][17]. Depending on the need, the fabricated MMC samples were also tested under compressive and tensile stresses using the Universal Testing Machine from M/S. ENKAY Enterprises, New Delhi. The microstructure and interfaces were studied using Optical microscopy (Radical-RMM-I) Scanning Electron Microscope (METEK FE-I-200F) and Energy Dispersive X-ray Spectroscopy (EDAX).

2.1. Fabrication of Samples

In order to fabricate the MMC samples, base Al alloy A384.1 was melted in an alumina crucible in furnace up to $810^\circ C$. The steel cover of the setup was then removed to add the preheated ($810^\circ C$) reinforced particles of $\text{Al}_2\text{O}_3$/MgO/SiC in the melt. A protective atmosphere was maintained during stirring by holding a pipe carrying inert gas over the melt. After the addition and through mixing, the metal matrix composites were fabricated by pouring the melt-mix into a die. The un-reinforced and reinforced casted materials were then subjected to hot extrusion described above. Hot extruded composites of $10 \text{ mm}$ diameter bars were then heat treated at $400^\circ C$ for 3 hours in the furnace. To further investigate the effect of aging, the extruded MMC’s were then pyroprocessed as per the pre-decided schedule of aging treatment. For structural, micro-structural and mechanical characterization the sample preparation was done first by polishing the sliced samples with emery paper up to 1200 grit size, followed by polishing with $\text{Al}_2\text{O}_3$/MgO/SiC suspension on a grinding machine using velvet cloth. Finally, the samples were polished with 0.5 $\mu$m diamond paste.

3. RESULTS AND DISCUSSIONS

3.1. Material and Microstructure

The microstructure of (A384.1)$_{0.9}$[(reinforcement)$_{0.1}$] composites with and without reinforcement are shown in figure 1a and figure 1b. It is noted that in the case of un-reinforced Al alloys and reinforcements particles SiC, $\text{Al}_2\text{O}_3$, MgO are fairly and uniformly distributed. But, in the case of unreinforced alloys, the grains are relatively finer than that of reinforced alloys (MMCs). Relatively greater degree of agglomeration of SiC, $\text{Al}_2\text{O}_3$, MgO particles based composites is noted as compare to un-reinforced Al alloys. The interface bonding between the matrix and reinforced particles are quite sharp indicating reasonably good bonding figure 1. As the density increases, the MMC becomes stiffer since the molecules do not have as much space to move around one another [12-13]. Also as the molecular weight increases, the entanglement of the molecules resists movement and therefore increases stiffness. The microstructure of the reinforced samples is found to have lower amount of overall porosity as compared to any other similar composite system. Along with reduction in porosity, a better distribution of particles was achieved in all alumina composites. No interaction layer or any other reaction product was found at the interface that appeared clean in the as-cast Al-$\text{Al}_2\text{O}_3$ materials. The EDX analysis, however, revealed the presence of the elements Al and O at the interface layer, as per figure 1a & b. It is
believed that this is probably coming due to an oxide layer formed during sample preparation. From figure 2a&b, microstructure and EDAX analysis shows that the uniform distribution of SiC particles is there. But the composites having reinforcement MgO, observed some amount of porosity, probably due to dissolved gases i.e. hydrogen, nitrogen etc. from Figure 3a & b.

3.2. **Porosity and Density**

A characteristic change is observed in porosity and density of the Al alloy and MMCs from as-cast to extrusion as a function of reinforcement from x=0 to x=0.10. It was observed that the value of porosity is found to increase with reinforcement. SiC with an average size of grain sizes of 0.220µm had considerably lower porosity contents, good strength and increased ductility in comparison with the as-cast samples.
Table 1: Reinforcement with SiC, Al$_2$O$_3$, MgO with particle size 0.220 µm for porosity and density after extrusion

<table>
<thead>
<tr>
<th>x</th>
<th>SiC</th>
<th>Al$_2$O$_3$</th>
<th>MgO</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Density (gm/cm$^3$)</td>
<td>Porosity (%)</td>
<td>Density (gm/cm$^3$)</td>
</tr>
<tr>
<td>0</td>
<td>2380</td>
<td>2.01</td>
<td>2380</td>
</tr>
<tr>
<td>0.05</td>
<td>2421</td>
<td>3.0</td>
<td>2521</td>
</tr>
<tr>
<td>0.06</td>
<td>2424</td>
<td>2.99</td>
<td>2499</td>
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<tr>
<td>0.07</td>
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<td>3.2</td>
<td>2525</td>
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<tr>
<td>0.09</td>
<td>2466</td>
<td>3.15</td>
<td>2530</td>
</tr>
<tr>
<td>0.10</td>
<td>2460</td>
<td>3.4</td>
<td>2533</td>
</tr>
</tbody>
</table>

In the case of composites, the interface between reinforced and matrix is associated with porosity indicating weak bonding. Table 1, shows the density and porosity of the investigated materials due to variation percentage of SiC, Al$_2$O$_3$, MgO with particle size 0.220 µm. It is noted that the huge variation in porosity is noted, but, in MgO (x=10), porosity reported is more as compare to other composites. But the density in the case of alumina with x=10 is reported more.

3.3. Micro-hardness

Hardness is the resistance of a material to penetration of its surface. It is related to the crystallinity and hence the density of the material. The hardness value depends on the shape, size, and time of the indenter used to penetrate the specimen [16]. Depending upon the hardness of the material to be tested, each hardness test has several scales to cover the entire range of hardness. For MMCs, the depth of indentation by the ball will be very dependent upon the amount of time that the specimen is under stress (due to the visco-elastic characteristics). Values of micro-hardness of un reinforced Al alloy-A384.1 and its composites in the as-cast and peak aged conditions (after extrusion) for the particles size as 0.220µm is given in Figure 4a & b respectively. It can be seen in the Figure 4a, by increasing the percentage of reinforcement in as-cast conditions, value of micro hardness keeps on increasing. At 0.05% reinforcement, the best microhardness is obtained for Al$_2$O$_3$ followed by MgO and SiC. However, at 0.10% both Al$_2$O$_3$ and MgO gives better results. Figure 4b, shows the increase in micro hardness at peak aged conditions by increasing the percentage of reinforcement in matrix. It is observed that there is increase in value of microhardness in the case of MgO as compare to Al$_2$O$_3$ and SiC when x vary from 0.05 to 0.06. By further increasing the percentage of reinforcement in matrix the microhardens decreases. However, the microhardness for Al$_2$O$_3$ decreases sharply at x=0.08 due to weak bond of interfacial structure as seen in Figure 1c. When the value of x=0.10, it is seen that there is increase in microhardness of SiC as compare to Al$_2$O$_3$ and MgO.

![Figure 4a & b, Variation of Microhardness in as-cast and peak aged conditions of MMCs respectively.](image-url)
3.4. Ageing Behaviour

In order to investigate the effect of aging on mechanical properties, the hardness versus ageing time characteristics of matrix and its aged composites were investigated for SiC in figure 5a. The observed changes in micro-hardness for the composites for the different values of ‘x’ from as-cast to peak aged condition are observed. After the peak ageing time, the values of micro-hardness were probably due to the larger precipitates that grow at the expense of smaller ones, as revealed by SEM studies. The peak micro-hardness values observed in the alloys with the reinforcements of $x=0.05$, $0.07$ and $0.10$ are, respectively, $182$ at $6.5$ hours, $181$ at $7.1$ hours and $185$ at $7.2$ hours as compared with $166$ at $6.0$ hours in the unreinforced alloy ($x=0.0$) as shown in figure 5a. The higher values of micro-hardness observed in case of peak-aged composites can be explained in terms of higher dislocation density near the particle–matrix interface due to the large difference in thermal expansion coefficient between the matrix and reinforcement as compared to unreinforced alloys [11-12]. The peak micro-hardness in $x=0.05$, $0.07$, $0.09$ and $0.10$ is $182$, $183$, $181$ and $184$ respectively, compared with $166$ in the unreinforced alloy at $x=0.0$ in the case of $\text{Al}_2\text{O}_3$. The peak ageing times in these composites are $5.5$, $5.5$, $5.3$ and $4.8$ hours respectively, compared with $6$ h for the unreinforced alloy as shown in figure 5b. Composites contain higher dislocation density near the particle–matrix interface due to the large difference in thermal expansion coefficient between the matrix and reinforcement. Therefore, a large number of precipitates are present in composites, giving rise to higher peak hardness and shorter peak ageing time.

![Figure 5a](image1.png)  
**Figure 5a, Graphs between the time in hours and the microhardness for reinforcement of SiC, in matrix.**

![Figure 5b](image2.png)  
**Figure 5b, Graphs between the time in hours and the microhardness for reinforcement of $\text{Al}_2\text{O}_3$, in matrix.**

![Figure 5c](image3.png)  
**Figure 5c, Graphs between the time in hours and the microhardness for reinforcement of $\text{MgO}$, in matrix.**
From the figure 5c., the peak values of hardness in the alloys with the reinforcements of MgO for x=0.05, 0.07, 0.09 and 0.10 are respectively 181 at 6.750 hours, 179 at 7.0 hours, 180 at 6.0 hours and 182 at 6.5 hours as compared with 166 at 6.0 hours in the unreinforced alloy (x=0.0). The main strengthening is due to the precipitates in the matrix alloy that nucleate heterogeneously on discontinuities like dislocations. Composites contain higher dislocation density near the particle–matrix interface due to the large difference in thermal expansion coefficient between the matrix and reinforcement. Therefore, a large number of precipitates are present in composites, giving rise to higher peak hardness and shorter peak ageing time, both in the case of macro-hardness and micro-hardness. Due to the formation of hardening precipitates a considerable improvement in strength of the alloy and composites after peak ageing is observed. It is noted that composites in dense condition aged much faster than those of unreinforced Al alloys. The primary ageing kinetics of the \(A384.1\), \(384.1\) is same in all the cases when \(x=0\), but variation is reported for \(x=5\) to \(x=10\). But some differences are observed in the ageing behavior of these materials. Reinforced added composites exhibits more hardness under peak aged condition as compared to that observed in matrix. All the composites show almost same rate of decrease in micro-hardness signifying that the growth rate of precipitate in over aged regime is variant to addition of SiC, Al\(_2\)O\(_3\), MgO. Lower micro-hardness values in the primary ageing in unreinforced alloy may be due to greater degree of clustering and some amount of copper in the matrix did not get precipitated due to the presence of reinforced. This is because of the fact that SiC, Al\(_2\)O\(_3\), MgO are different from Al/Al alloys in terms of valency, crystal structure, atomic diameter etc. which controls the diffusivity. Additionally, finer grain size results in greater extent of grain boundary area. Similarly, relatively weak interface bonding causes wider interfacial area and more porosity. All these factors act as sites for dislocation sinks and there is a possibility of less dislocation strengthening in the composites. This also causes delay in secondary ageing in ageing of composites and lower micro-hardness in unreinforced alloys in peak ageing. The porous structure, on the other hand, aged at slower rate as compared to the dense composites. The unreinforced Al alloys exhibit almost similar nature of ageing behavior, where in the composites exhibits higher micro-hardness. This may be attributed to relatively greater extent of difference in thermal expansion coefficient between the matrix and the reinforced elements. This leads to higher degree of residual stress in the composites as compared to that in unreinforced alloys. Slower rate of ageing in these materials as compared to that in dense materials is attributed to the presence of huge porosity which acts as dislocation sink and thus, the dislocation density is expected to be reduced significantly as compared to that in dense one. This along with relatively weak bonding between reinforced and the matrix causes slower rate of ageing. The under ageing and over ageing characteristics of all these composites are noted to be almost same.

### 3.5. Tensile properties

Using a tensile testing machine, a sample is bent while being held in a three- or four point- contact holder. The amount of stress needed to deflect the outer surface of the sample a certain vertical distance (strain) is determined. Since most MMCs do not break in this test, the true flexural strength cannot be determined. The point at which a stress causes a material to deform beyond its elastic region is called the tensile strength at yield. The force required to break the test sample is called the ultimate strength or the tensile strength at break. The strength is calculated by dividing the force (at yield or break) by the original cross-sectional area. ASTM D3552 - 96(2007) standard is used to determine the tensile properties of reinforced composites [11]. The test sample develops a “neck down” region where the molecules begin to align themselves in the direction of the applied load. This strain-induced orientation causes the material to become stiffer in the axial direction while the transverse direction (90° to the axial direction) strength is lower. Failure occurs when the molecules reach their breaking strain or test sample defects, such as edge nicks, begin to grow and cause premature failure. Fibrillation, which is the stretching and tearing of the MMCs structure, usually occurs just prior to rupture of a well-drawn sample. From the tensile measurements for SiC, Al\(_2\)O\(_3\) and MgO based metal matrix composites with varying percentage of ‘x’, it is clear that composite x=0.10 exhibits highest values of 0.2% proof stress (403 MPa) for MgO and UTS (449 MPa) for SiC among all the peak aged conditions composites fabricated. The peak aged conditions composites reinforced samples were found to have 0.2% proof stress and UTS values larger than the unreinforced alloy. It is
obvious that the SiC reinforced materials have better mechanical properties as compared to their other counterparts. From Figure 6a, it is clearly showed the variation in different MMCs for 0.2% Proof stress (MPa) with respect to the value of ‘x’. In this case, we found that values of proof stress are approximately same for SiC and MgO based systems, but a down trend is shown in the case of Al₂O₃ system. In the case of Ultimate Tensile strength and the value of ‘x’ graph in Figure 6b, it is clear that an upcoming trend is shown in SiC and MgO systems but a down trend is shown in the case of Al₂O₃. For εᵣ (%) from Figure 6c, a upward trend is shown in SiC system but in the case of Al₂O₃ and MgO based systems drastic variation is registered. As evidenced from microstructural investigations for SiC, Al₂O₃ and MgO, attribute to the generation of micro-cracks in the interfacial region during mechanical working on the samples because of the mismatching thermal expansion coefficients of reinforcement alumina and Al alloy matrix. The micro-structural studies on these composite materials have revealed that the samples having lower values of UTS and 0.2% proof stress, especially x=0.10 were found to have some cracks in the local regions at the interface between the matrix and particle due to the high thermal stresses generated at the interface during cooling as a result of the large difference in coefficient of thermal expansion between the matrix and the particles. This may lead to decohesion of particles at the particle–matrix interface i.e early failure and hence poor mechanical properties. Therefore, in order to realize the strengthening effect of the reinforcement, the interfacial bonding between the particle and matrix must be strong.

4. CONCLUSIONS
Relatively greater degree of agglomeration of SiC, Al₂O₃, MgO particles based composites is noted as compare to un-reinforced Al alloys. The interface bonding between the matrix and reinforced particles are quite sharp indicating reasonably good bonding. As the density increases, the MMC becomes stiffer since the molecules do not have as much space to move around one another. It was observed that the value of porosity is found to increase with reinforcement. SiC with an average size of grain sizes of 0.220µm had considerably lower porosity contents, good strength and increased ductility in comparison with the other counterparts.
By increasing the percentage of reinforcement in as-cast and peak aged conditions, value of microhardness keeps on increasing. At 0.05% reinforcement, the best microhardness is obtained for $\text{Al}_2\text{O}_3$ followed by MgO and SiC. However, at 0.10% both $\text{Al}_2\text{O}_3$ and MgO gives better results for as cast MMCs. In the case of peak aged conditions, the value of microhardness at 0.05 is more that of MgO as compare to $\text{Al}_2\text{O}_3$ and SiC, when the value of $x=0.10$, it is seen that there is increase in microhardness of SiC as compare to $\text{Al}_2\text{O}_3$ and MgO. In the tensile measurements for SiC, $\text{Al}_2\text{O}_3$ and MgO based metal matrix composites with varying percentage of ‘x’, it is clear that composite $x=0.10$ exhibits highest values of 0.2% proof stress (403 MPa) for MgO and UTS (449 MPa) for SiC among all the peak aged conditions composites fabricated. The peak aged conditions composites reinforced samples were found to have 0.2% proof stress and UTS values larger than the unreinforced alloy. It is obvious that the SiC reinforced materials have better mechanical properties as compared to their other counterparts.

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