Microstructure and fracture behaviour of two stage stir cast Al6061-SiC composites

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Abstract
Present work deals with a metal matrix composite (MMC) of Al6061 as matrix and SiC as reinforcing materials. These composites with different amount of SiC are produced by two stage stir casting process. They are then solutionized and aged at different temperature with varying duration. The influence of artificial aging on mechanical properties has been assessed. Microstructural details, macro-hardness and mechanism of fracture behavior after tensile test have been discussed. Fracture surface of the broken tensile sample is studied in details to know failure mode and parameters that influence the crack growth characteristics. It is found that mechanical properties of the MMCs have improved marginally with the addition of SiC reinforcing particulates while after aging this has been improved markedly due to the precipitation of secondary intermetallic phases. It is found that the fracture mode of the pure Al6061 is purely dimple rupture, characteristic of overload failure and this is due to coalescence of micro-voids. After addition of SiC reinforced particles the fracture mode becomes predominantly de-cohesive rupture mode in addition of dimple and quasi-cleavage rupture mode.

1. Introduction
Metal matrix composites (MMCs) are engineering materials in which a hard ceramic component is dispersed in a ductile metal matrix in order to obtain characteristics that are superior to those of the conventional monolithic metallic alloys [1]. Aluminium matrix composites reinforced with ceramic particles such as SiC, B₄C, Al₂O₃ and TiC are the most commonly used materials in automobile and marine industries, and therefore, have been paid more consideration because of high strength, high modulus and low density. Consequently, it has been established that the high dislocation density in SiC-reinforced aluminium alloy matrix composite, caused by the difference between the thermal expansion coefficients, is responsible for improving mechanical properties. This leads to more nucleation sites for the fine precipitates during artificial ageing [3-4]. In the MMC, the presence of reinforcement particles in aluminium alloy accelerating the aging process also and thus attain additional strength.

Al6061 alloys are considered as medium strength heat treatable alloys and have excellent formability and good corrosion resistance characteristics [5]. The presence of major solutes such as Mg and Si in the 6XXX series alloy contributes exceptional increase in strength during precipitation hardening [6-9].

In literature there are plenty of works related to the aging sequence of various Al alloys. In many of the cases variation of mechanical properties with aging temperature and time has been correlated to the phase transformations during aging treatment. The improvement in the mechanical properties during aging is due to the vacancy assisted diffusion mechanism under aged and peak aged conditions [10-13]. The formation of GP zones depends upon the aging temperature, which distorts the matrix lattice planes [14]. This distortion of the lattice planes hinders the dislocation movement as long as the coherency exists in the lattice [15]. When the precipitates are finer and more in number (lower aging temperature and peak age condition), bypassing of dislocations through precipitates becomes complex and difficult through Orowan’s mechanism, because fine sized precipitates with significantly higher structural incoherency results in dislocation pile up around the precipitation during aging treatment and becomes difficult for plastic deformation. Over aging results the coagulations of existing precipitates and grow in size at the expense of fine precipitates causing easy bypassing of the dislocations [16]. Several works have been reported on the use of two-step (double) stir casting as a
means of improving cast metallic matrix [5]. All these studies brought out differences in microstructures of aluminium metal matrix composites (AMCs) produced through different routes such as, (a.) direct casting (no stirring), (b.) manual stirring, and (c.) two step mixing etc. It was evident from these reported literatures that the two step mixing gives the best uniform distribution of the SiC particulates [3]. It was also suggested that production of AMCs without the use of two step stirring results in less dispersion of the particulates and higher porosity levels which might be in excess of the acceptable limits. Thus the present work emphasises on investigation of the effect of reinforcement and artificial aging on microstructure and fracture behavior of Al6061-SiC composites.

2. Materials and methods
2.1. Base Material
The base matrix chosen in the present study is aluminium 6061 (0.52% Si, 0.95% Mg, 0.55% Fe, 0.24% Cu, 0.14% Mn and 0.25% Cr) because it is one of the most extensively used 6000 series aluminium alloys. The silicon carbide reinforcement particles used for preparation of composite in the current work is brought from Indian Fine Chemicals, Mumbai. The size of the SiC reinforcement is in the range of 35-40μm (400 Mesh). The reinforcement materials are having irregular shape. SEM (Scanning Electron Microscope) micrograph and XRD (X-Ray Diffraction) plot of the used SiC particulates are shown in Fig.1 confirms the size and purity of the SiC particles. Al6061-SiC composites were fabricated by two stage stir casting technique by altering the amount of silicon carbide particles in the range 2-6 % wt.

![Figure 1: SEM and XRD micrographs of silicon carbide (SiC) particles](image)

2.2 Hardness and tensile testing
Hardness tests were carried out in a Brinell hardness testing machine with steel ball indenter of diameter 5mm and a load of 250 kgf (SAROJ Brinell Hardness Testing Machine, Model: B/3000/00, S# 13/06/08- India). Specimens of diameter 12 mm and length 15 mm are prepared. In order to eliminate possible segregation effect, the average of a minimum of three indentations readings is taken for each specimen at different locations of the test samples.

Tensile properties dictate how the material will react to forces being applied in tension. Tensile specimen is prepared as per ASTM-E8M standards. Circular cross section specimen with diameter 6 mm and gauge length of 24 mm is prepared. Tensile test is carried out on Tensometer. Diameter of specimen is measured using Vernier Caliper and cross sectional area is calculated. The load cell value is kept to 20.5 kN and test mode is selected as break. The cross head speed is kept constant at value of 10 mm/ min, with length increment value of 0.01 mm.

2.3 Precipitation hardening
The specimen prepared for above test is subjected to age hardening heat treatment. Specimens are soaked at 558°C for duration of 2h, then immediately quenched in water at room temperature. The quenched specimens were artificially aged in the furnace at 100, 150 and 200°C for various durations of time. According to the Al-Mg-Si phase diagram melting of ternary eutectic Mg2Si-(Al)-(Mg) phase takes place at 558°C. Therefore, the primary phases will be dissolved completely at 558°C during solutionizing and during age secondary intermetallic phases will precipitate that contributes for maximize the strengthening effect. These secondary
precipitated phases result in strengthening of the alloy system and coherency of the crystal structure of the particle and the matrix. It is also reported that the samples of Al6061 composite, with the solution heat-treated at 558°C, exhibit better strength compared to solutionized at 530°C [9].

3. Results and discussion

3.1 Hardness Measurement

Both as cast and aged samples are tested to determine the Brinell Hardness Number (BHN). Table 1, shows the peak hardness values obtained in as cast and different aging temperatures at 100, 150 and 200°C. In as cast condition it is clear that the hardness values increase with the addition of reinforcements when compared to the unreinforced alloy. The hardness value increases with increase in weight percentage of SiC. The increase in hardness is to be expected, since SiC particles being a hard dispersoid positively contribute to the hardness of the composite [17]. Increased content of reinforcement in the matrix alloy leads to higher dislocation densities during solidification due to the thermal mismatch of the matrix alloy and the reinforcement. This results in large internal stresses and mismatch strain that affects the microstructure and subsequently improves mechanical properties of the composites. The matrix deforms plastically to accommodate the smaller volume expansion of the reinforcement particles leading to increased dislocation density. Enhancement in dislocation densities results in higher resistance to plastic deformation and responsible for additional increase in hardness of composites [18].

Table 1: Hardness of Al6061- SiC (0, 2, 4 & 6% wt.) as cast and peak aged condition.

<table>
<thead>
<tr>
<th></th>
<th>As cast condition (without Heat treatment) BHN</th>
<th>Peak aged condition at 100°C; BHN</th>
<th>Peak aged condition at 150°C; BHN</th>
<th>Peak aged condition at 200°C; BHN</th>
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</thead>
<tbody>
<tr>
<td>Al6061 alloy</td>
<td>50</td>
<td>85</td>
<td>74</td>
<td>65</td>
</tr>
<tr>
<td>Al6061- 2% SiC</td>
<td>58</td>
<td>110</td>
<td>102</td>
<td>90</td>
</tr>
<tr>
<td>Al6061- 4% SiC</td>
<td>62</td>
<td>115</td>
<td>104</td>
<td>96</td>
</tr>
<tr>
<td>Al6061- 6% SiC</td>
<td>69</td>
<td>122</td>
<td>115</td>
<td>101</td>
</tr>
</tbody>
</table>

Similar to base alloy, the Al6061 matrix composite is very sensitive to age hardening irrespective of lower or higher aging temperature. It is evident that composites exhibit accelerated rate of aging kinetics as compared to unreinforced matrix alloy. Aging kinetics gets accelerated in the composites with increase in wt. % of reinforcements. Aging is accelerated due to the presence of areas with a high concentration of dislocation close to Al6061 matrix & SiC reinforcements interface. These high density locations provide heterogeneous nucleation sites for the precipitation & high diffusivity path for the diffusion of alloying elements [18]. Compared to base alloy, composites show drastic increase in the hardness in as cast & treated conditions. At the same time increase in weight percentage of SiC in the composites gives positive effect on hardness value. Lower aging temperature shows higher peak hardness of base alloy as well as composites as compared to higher temperature aging. Lower temperature aging contributes to the increased hardness by increasing the number of intermediate zones during precipitation, increase in the number of finer inter-metallic’s & therefore decreased interparticle distances. Higher the aging temperature, lower is the time required to attain peak hardness [9]. From the above results it is understandable that heat treatment has a profound influence on the hardness of matrix alloy as well as composites.

3.2 Tensile strength

Tensile test is carried out on as cast and peak aged specimens. The average value of the three readings in as cast and peak aged conditions (for both 0, 2, 4 & 6%wt. SiC composites) is tabulated in Table 2. There is a marginal increase in the ultimate tensile strength (UTS) with the addition of reinforcements when compared to unreinforced alloy in as cast condition. The reinforcement particles have little influence on mechanical properties of the composites and it is due to generation stress in the interface, which transfers and distributes the load from the matrix to the reinforcement exhibiting increased elastic modulus and strength. The UTS in composite or base alloy is very sensitive towards age hardening. There is a minimum of 40% additional increase in the UTS by age hardening over untreated specimen. From Table 2, it is clear that higher the weight percentage of reinforcement in the composite and lower the aging temperature better is the ultimate tensile strength. The increase in strength is due to the combined effect of difference in co-efficient of thermal expansion between matrix and SiC particulates and precipitation behavior of solute rich secondary phases.

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Table 2: Ultimate Tensile Strength (UTS) of the investigated composites and alloy in as cast and peak aged conditions

<table>
<thead>
<tr>
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<th>As cast condition (without Heat treatment) MPa</th>
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<td>211</td>
<td>199</td>
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<td>Al6061- 2% SiC</td>
<td>149</td>
<td>236</td>
<td>220</td>
<td>208</td>
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<tr>
<td>Al6061- 4% SiC</td>
<td>156</td>
<td>241</td>
<td>227</td>
<td>212</td>
</tr>
<tr>
<td>Al6061- 6% SiC</td>
<td>160</td>
<td>244</td>
<td>232</td>
<td>214</td>
</tr>
</tbody>
</table>

3.3 Fractured surface analysis of Al6061 alloy and Al6061-SiC composites

Figure 2: SEM micrographs of fracture surface of as cast a) Al 6061 alloy and b) same at higher magnification.

Figure 3: SEM micrographs of fracture surface of a) heat treated Al6061 alloy after peak aged at 100°C and b) the same at higher magnification.

Fracture surface of as-cast Al6061 after tensile test is shown in Fig.2. It is observed from Figs.2 that the fracture mode is predominantly dimple rupture. This is the characteristic of overload failure and fail by micro voids coalescence process. Numerous cuplike depressions are observed in Fig.2a. These are referred as dimples as shown in Fig.2b at higher magnification. Dimples are the results of formation and coalescence of micro-voids that nucleate at localized strain region (2nd phase particles, inclusions, grain boundaries, dislocations etc.). In Fig.2b, it is found that some micro-voids formed at grain boundary and other locations. In Fig.3 fracture surface of aged specimen is shown after tensile test. Here numbers of dimples are more and smaller in size indicating the formation of micro-voids (dimples after coalescence) at numerous precipitated particles at peak aging. Therefore, the dimples are evenly distributed with smaller in size.

The size of the dimples in the fracture surface section of the artificially aged specimen is smaller than that in the fracture surface section of the untreated alloy. The dimple size exhibits a direct proportional relationship with strength and ductility (i.e., if the dimple size is finer, then the strength and ductility of the respective joint is higher and vice versa). Therefore, the artificially aged specimen shows higher UTS values compared with untreated Al 6061 alloy.
Whereas in case of sample with the addition of SiC particles, in general the fracture mode is mixed type with dimple rupture and de-cohesive rupture. In few places it is also found quasi-cleavage fracture. Quasi-cleavage is localized and exhibits characteristics of both cleavage and plastic deformation. It actually resembles like cleavage. On the other hand, de-cohesive rupture can be caused by several different mechanisms. In the present case the presence of de-cohesive rupture may be due to the rupture of protective films surrounding SiC, as shown in Fig. 4(b & c). In Fig.4d the sign of both cleavage and plastic deformations (dimple rupture) are observed. Fig.5 show fracture features associated with SiC particles in the fracture surface of Al6061 with 4 wt.% SiC after the fracture with a tensile load. In Fig.5a, a low magnification fracture surface is shown.

Figure 4: SEM fractography of the composite with 2 wt.% SiC aged at 100°C (a) low magnification and (b, c & d) higher magnification from different region of (a).

Figure 5: SEM fractography of the composite with 4 wt.% SiC aged at 100°C (a) low magnification and (b, c & d) higher magnification from different region of (a).
It seems the fracture surface contain mixed mode of fracture of quasi cleavage along with de-cohesive rupture. Fig.5b indicates that the fracture surface is a mixture of dimple rupture and cleavage mode. Whereas, Fig.4c illustrates as elongated dimple along with de-cohesive rupture mode. Fig.5d indicates the region of fracture is purely de-cohesive rupture and elongated dimples along cleavage direction.

At higher amount of SiC, the de-cohesive rupture mode covers more area in the fracture surface. From Fig.6a it is observed that amount of dimple mode is decreased. Fig. 6(b & c) reveal fine dimple with cleavage mode. Whereas Fig. 6d shows fracture is dominated by de-cohesive mode.

Figure 6: SEM fractography of the composite with 6 wt.% SiC aged at 100°C (a) low magnification and (b, c & d) higher magnification from different region of (a).

Conclusion

- In as cast condition, hardness increases by 20-40% with the addition of reinforcement compared to the base alloy;
- Al6061 alloy and Al6061-SiC composites positively responds to age hardening treatment with considerable improvement in mechanical properties;
- Slower precipitation kinetics and higher peak hardness is noticed at lower aging temperature for both base alloy and the composites;
- An increase in peak hardness of 80-100% (2-6 wt.% SiC) aged at 200°C and increase of 120-145% (2-6 wt.% SiC) aged at 100°C are observed in significant duration for the composites in comparison with untreated Al6061 alloy;
- Marginal increase (10-15%) in ultimate tensile strength (UTS) is noticed in as cast Al6061-SiCp composite compared to as cast Al6061 alloy;
- During aging there is 40-70% increase in UTS of composites over untreated base alloy and highest UTS is observed after peak aging at low temperature with 6% of SiC and
- Peak aged tensile fractured surface of base alloy exhibits mixed mode of failure, whereas peak aged composite material fails by void nucleation growth (VNG) and particle-matrix interface cracking with de-cohesive rupture mode.
References


(2017); http://www.jmaterenvironsci.com/