Strengthening mechanisms in ultrasonically processed aluminium matrix composite with in-situ Al₃Ti by salt addition

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1. Introduction

Liquid Metallurgy approach to processing of aluminium matrix composites (AMCs) is economical and favours large scale production based on established technology. Reinforcements like carbides, nitrides, oxides and borides are common to AMCs [1–5]. In in-situ processing, the reinforcement particle is formed within the matrix phase as a result of favourable chemical reaction. Consequently, the second phase is thermodynamically stable and uniformly dispersed throughout the matrix with a clean interface [6–8]. As a result, in-situ formed composites demonstrate improved mechanical properties [9].

Several investigations have reported on ceramic reinforced AMCs [10–14]. The making of these composites have some limitations due to the significant difference in the coefficient of thermal expansions (CTE) between the matrix and reinforcement phase. On the contrary, intermetallic compounds are an appropriate choice for reinforcement due to their low density and high modulus. Aluminides, such as Al₃Fe, Al₃Ni, Al₃Zr and Al₃Ti have attracted attention due to their low density and high modulus. Aluminides with fcc α-Al due to thermodynamic equilibrium of Al₃Ti in Al matrix [19]. Moreover, Al₃Ti particles act as heterogeneous nuclei which lead to the grain refinement of α-Al phase [20]. Above the melting point of aluminium, Al₃Ti exhibits low coarsening rate due to the low solubility and faster diffusivity of Ti in Al [21]. Orowan strengthening, load transfer and grain size strengthening are the major strengthening mechanisms in Al₃Ti reinforced composites [22–24].

The presence of porosity is detrimental to the casting. The major cause for porosity in aluminium castings is the presence of dissolved hydrogen in molten metal (0.3–0.5 cm³ per 100 g), which is more than the industry standard which is close to 0.1 cm³ per 100 g [25]. For degassing, purifying and refinement of metallic melts, ultrasonication is a viable option, as it is both environment-friendly and economical [26–29]. In ultrasonic processing, high intensity acoustic wave interacts with molten metal producing nonlinear effects which include acoustic streaming, acoustic radiation pressure, cavitation and emulsification [30]. These effects contribute to the better distribution of the second phase, elimination of columnar dendritic structure and refinement of equiaxed grains [27]. The mechanism behind grain refinement is related to the formation of micro “hot spots” which sustain only for a few nanoseconds in the molten metal during ultrasonic vibration. These hot spots attain a temperature of the order of 5000 °C with heating and cooling rates close to 10¹⁰ K s⁻¹, and localized pressure approaching 1000 atm [31].

In the present work, K₂TiF₆ inorganic salt was added into the Al melt to form in-situ Al₃Ti particles. Ultrasonication was applied for better distribution of salt particles throughout the molten metal. It is
well known that better distribution of in-situ particles along with limited porosity will improve the mechanical properties of the composite. Therefore, an attempt is made to fabricate Al3Ti reinforced aluminium matrix composite using low cost salt-metal reaction route.

2. Experimental setup

K2TiF6 salt powder (Madras Fluorine Private Ltd, Chennai, India) and Al6061 alloy (Hindalco, India) are the materials used in the present work. The chemical analysis of Al6061 alloy was done by XRF (Rigaku supermini 200) and presented in Table 1. The schematic diagram of ultrasonically assisted casting is illustrated in Fig. 1. A 1.5 kW high power ultrasonic system (Model VCX 1500, Sonics and Materials, USA), which could produce 20 kHz frequency with air cooled converter, made from piezoelectric lead zirconate titanate crystals, was used for generating ultrasonic vibration in the molten melt. The intensity of this unit could be adjusted from 0 to 5.4 kW cm⁻².

A 250 g Al6061 ingot was taken inside a graphite crucible and heated up to 750 °C in an resistance heating furnace. After holding the melt at 750 °C for 30 min, varying amount of salt (5, 10 and 15 wt %) was added into the melt to develop composites with different weight percent (2.7, 5.4 and 8.1 wt %) of Al3Ti particles as shown in Table 2. After addition, the melt was stirred manually for 2 min by using a graphite rod for proper mixing of the powders into the molten metal. After proper mixing of the salt, ultrasonication was carried out at 750 °C for 5 min. This is achieved by inserting the niobium probe of 19 mm diameter, coated with zirconia baked at 200 °C and preheated to the processing temperature. The amplitude and frequency of ultrasonic stirring was 24 μm and 20 kHz, respectively [32].

After ultrasonication, the melt was poured and allowed to solidify in a steel mould of dimension 40 × 40 × 120 mm³ which was coated with Zirconia to avoid contamination from the mould. The mould was preheated to 400 °C to ease the flow and reduce thermal damage to the casting. Metallographic samples were cut from the cast ingot and polished with 320, 800, 1200, 1500 and 2000 grit emery papers followed by cloth polishing with MgO abrasive. These polished samples were etched with Keller's reagent to obtain the microstructure. Optical microscope (Leica, DMI 5000 M) and a scanning electron microscope (Carl Zeiss, EVO 18) in secondary electron imaging mode were used for imaging. The mean linear intercept method was used to calculate average grain size. The XRD analysis was conducted using Rigaku smart lab, X-ray diffractometer employing Cu Kα radiation. Tensile test was performed at room temperature on a H25 K-S Tinius Olsen tensile testing machine with constant crosshead speed of 0.1 mm/min. The dimensions of tensile specimen were 4 mm diameter and 20 mm gauge length according to the ASTM E8M standard and tensile testing was carried out according to ASTM B557 with a strain rate of 10⁻⁶/s. The average value of three tensile tests is reported along with standard deviation. Hardness test was performed on HPO-250 Heckert Brinell hardness tester with 15 kg load. An average of at least five hardness readings was taken and reported along with standard deviation.

3. Results and discussion

The melting point of K2TiF6 is 682 °C. The K2TiF6 salt powder was added into the aluminium melt maintained at 750 °C. After addition the temperature of the melt rose to 800 °C due to the exothermic heat generated in the reaction between the salt and aluminium.

\[3K_2TiF_6 + 13Al = 3Al_3Ti + K_3AlF_6 + 3KAlF_4\] (1)

Gibbs free energy for the formation of Al3Ti at 750 °C (1023 K) is −122.8 kJ/mol [33], which confirms that reaction between inorganic salt K2TiF6 and Al6061 takes place spontaneously.

3.1. XRD analysis and in-situ formation of Al3Ti particles

To verify the completion of the reaction, separate XRD analysis was done for aluminium reinforced composites which formed with varying amounts of K2TiF6 added to the melt (Fig. 2). The XRD peaks corresponding to the in-situ formed Al3Ti particles are clearly identified in all the compositions. Moreover, the relative intensity of the Al3Ti diffraction peaks increased as the amount of K2TiF6 increased. In the present work only Al3Ti particles are present. Other intermetallics, such as Al2Ti, AlTi3 and elemental Ti reported in other studies were not detected suggesting that the reaction between dissolved Ti and Al could have gone to completion.

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Table 1
Chemical composition of Al6061 alloy.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>Cr</th>
<th>Zn</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wt%</td>
<td>0.70</td>
<td>0.18</td>
<td>0.29</td>
<td>0.33</td>
<td>0.88</td>
<td>0.006</td>
<td>0.003</td>
<td>0.02</td>
<td>97.591</td>
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</tbody>
</table>

Table 2
Intermetallic content in each sample.

<table>
<thead>
<tr>
<th>Sample Label</th>
<th>K2TiF6 addition (wt %)</th>
<th>Al3Ti content (wt %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base Al alloy</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>C1U</td>
<td>5</td>
<td>2.7</td>
</tr>
<tr>
<td>C2U</td>
<td>10</td>
<td>5.4</td>
</tr>
<tr>
<td>C3U</td>
<td>15</td>
<td>8.1</td>
</tr>
</tbody>
</table>

Fig. 1. Schematic diagram of ultrasonic assisted casting.

Fig. 2. XRD patterns of the fabricated composites and base Al alloy.
3.2. Microstructural study

Fig. 3 shows the optical micrograph of the base Al alloy and the developed composites. It is noted that Al3Ti particles which appear as dark spots, confirmed by EDX analysis (Fig. 4), are dispersed throughout the microstructure and increased in numbers from C1U to C3U as the salt addition was increased. With higher amounts of K2TiF6 addition, more intermetallic particles form which tend to agglomerate during casting to minimize the overall interfacial energy [34]. The high intensity ultrasonic vibrations are applied into the melt to break the agglomerated Al3Ti particles and achieve better distribution throughout the matrix as observed in C1U, C2U and C3U composites (Fig. 3).

During ultrasonic treatment, ultrasonic field injected into the molten metal produce nonlinear effects such as acoustic streaming and cavitation. In cavitation, small cavities are formed throughout the molten metal when the acoustic wave produces tensile stress during the rarefaction phase. The size of these cavities increases due to inertia, until a stage comes when they collapse because of compressive stress during the compressive phase of the cycle. The sound pressure (P) in the travelling wave can be calculated according to

\[ P_k = \frac{\sqrt{2P \rho_L c}}{S} \]

where \( \rho_L \) is the melt density in (g cm\(^{-3}\)), \( P \) (kW) is the output power of the ultrasonic generator, \( c \) is the speed of sound in the melt (m s\(^{-1}\)) and \( S \) (cm\(^2\)) is the probe-face area [35–37]. In our experiment, \( \rho_L = 2.37 \text{ g cm}^{-3}, c = 1.3 \times 10^3 \text{ m s}^{-1} \) [27], \( P = 1.5 \text{ kW} \) and \( S = 2.8 \text{ cm}^2 \), thereby, \( P_k = 5.7 \text{ MPa} \), which is much higher than the threshold value of 1 MPa [36] for generating acoustic cavitation. Under the effect of high pressure pulse, the particle clusters are broken and dispersed throughout the matrix. The dispersion is facilitated by acoustic streaming, which is a form of turbulence developed near obstacles when there is a loss in wave energy. To produce these effects, fully developed cavitation must occur into the molten metal which depends upon the intensity (I) of the ultrasonic vibration. The intensity of ultrasonic vibration is defined by

\[ I = \frac{1}{2} \rho_L c (2\pi f A)^2 \]

where \( \rho_L \) is the melt density (g cm\(^{-3}\)), \( f \) is the frequency (Hz), \( A \) is the amplitude (μm) and \( c \) is the speed of sound in the melt (m s\(^{-1}\)) [27,38]. High-intensity ultrasonic vibration requires \( I \geq 100 \text{ W cm}^{-2} \). Moreover, the fully developed cavitation occurs in the molten aluminium alloys when \( I \geq 80 \text{ W cm}^{-2} \) [27,38]. In our experiment, \( A = 24 \mu m, \rho = 2.37 \text{ g cm}^{-3}, c = 1.3 \times 10^3 \text{ m s}^{-1}, \) and \( f = 20 \times 10^3 \text{ Hz} \), thereby \( I = 1400 \text{ W cm}^{-2} \), which is far greater than the threshold value. The effect of cavitation produced by the ultrasonic vibration in the melt is confirmed by the well dispersed Al3Ti particles shown in the microstructure of the ultrasonically treated melts.

The average grain size of the composites is shown in Fig. 5. It is observed from the figure that the grain size of composites decreases with increase of Al3Ti particles, suggesting that in-situ formed Al3Ti can act as an effective grain refining agent during solidification. The interfacial energy between the solid matrix and the nucleating substrate
plays an important role in microstructure refinement. If the solid and the substrate are coherent or partially coherent, the interfacial energy will be low. The following equation can be used to evaluate the lattice misfit, \( \delta \), between the substrate and the matrix:

\[
\delta = \left| \frac{\alpha_{\text{matrix}} - \alpha_{\text{substrate}}}{\alpha_{\text{matrix}}} \right|
\]

where \( \alpha_{\text{substrate}} \) and \( \alpha_{\text{matrix}} \) are the lattice constants of the substrate and the matrix, respectively. As a rule, the interface between the matrix and the substrate is coherent when \( \delta \leq 0.05 \) and partially coherent when \( 0.05 < \delta < 0.25 \) [39].

The crystal structure of \( \text{Al}_3\text{Ti} \) is tetragonal with \( a = b = 0.385 \text{ nm} \) and \( c = 0.861 \text{ nm} \) [40]. Each unit cell contains 6 Al and 2 Ti atoms. On the other hand, \( \alpha\text{-Al} \) crystal has a fcc structure with \( a = b = c = 0.404 \text{ nm} \) [41]. So, the lattice misfit values between in-situ formed \( \text{Al}_3\text{Ti} \) and \( \alpha\text{-Al} \) in both \( a \) and \( c \) directions are 0.049 and 0.065, respectively. On the basis of this calculation, it can be concluded that in-situ formed \( \text{Al}_3\text{Ti} \) particle has a good lattice match with \( \alpha\text{-Al} \) and can act as an effective heterogeneous nucleating site for primary Al during solidification. The size distribution of \( \text{Al}_3\text{Ti} \) particles in the composites was characterized by Image J software as shown in Fig. 6. It was observed that about 75% of \( \text{Al}_3\text{Ti} \) particles are between 2 \( \mu \text{m} \) and 4 \( \mu \text{m} \) in size. The average size of \( \text{Al}_3\text{Ti} \) particles is about 3.4 \( \mu \text{m} \).

Grain refinement due to \( \text{Al}_3\text{Ti} \) particles can also be understood with the help of Al-Ti phase diagram obtained by using thermo-cal software (Fig. 7). According to the phase diagram, the peritectic reaction takes place according to:

\[
\text{L} + \text{Al}_3\text{Ti} \rightarrow \alpha\text{-Al} (\text{at } 665 ^\circ \text{C})
\]

The reaction product is a new phase precipitated on \( \text{Al}_3\text{Ti} \) which acts as a heterogeneous nucleating agent. This is confirmed by the microstructures shown in Fig. 3(a–d) which show \( \text{Al}_3\text{Ti} \) particles inside Al grains.

3.3. TEM analysis

Thermodynamically unstable particles tend to react with the melt and form reaction products around the reinforcing particles, which could be detrimental from mechanical property point of view [42]. Hence, a clean interface between the matrix and the particles is an essential requirement to enhance the load bearing capacity of particulate reinforced composites. Fig. 8a and b shows TEM micrographs of the area containing \( \text{Al}_3\text{Ti} \) particle and SAED pattern of an \( \text{Al}_3\text{Ti} \) particle, respectively. It is evident from the TEM analysis that the interface between the Al matrix and \( \text{Al}_3\text{Ti} \) particles is clean (Fig. 8a) without the presence of porosity or any reaction product. The in-situ formation of \( \text{Al}_3\text{Ti} \) particles within melt reduces the possibility of oxidation of the particles, thus improving the interfacial bonding between matrix and particles [43]. Dislocations around the \( \text{Al}_3\text{Ti} \), which are generated due to difference in CTE between matrix and particles, also contribute to strength and hardness of the composites (Fig. 8a).

3.4. Porosity and density measurement

The experimental density of the base Al alloy and composites was calculated by Archimedes principle and is shown in Table 3. Theoretical density was calculated by the rule of mixture. Density was observed to increase from 2.55 to 2.67 g cm\(^{-3}\) with increase in \( \text{Al}_3\text{Ti} \) particles. Porosity in base alloy and composites was found below 6 vol %. It was noticed from the porosity measurement that the percentage of porosity was less in the composites as compared to base alloy because of ultrasonic degassing. Degassing of the melt during ultrasonication happens in three stages [38]: Stage I: Bubbles are formed in the melt and their sizes increase due to transformation of gasses from the surrounding melt. Stage II: These bubbles get consolidated and form large bubbles. Stage III: The large bubbles float to the top surface of the liquid metal and collapse releasing gas to the environment. So, when the duration of ultrasonic stirring is increased, the dissolved gases in the liquid metal are removed effectively and composites with less porosity can be formed.
4. Mechanical properties

4.1. Hardness

Fig. 9 shows the Brinell hardness number of the base aluminium alloy and the composites with 2.7, 5.4 and 8.1 wt % of in-situ Al₃Ti. It is clearly observed that the developed composites possess improved hardness over the starting alloy by 50, 72 and 103%, respectively. It is experimentally proven fact that the hardness of the matrix material is increased when the hard particles are reinforced into the soft ductile matrix [44]. The incorporation of Al₃Ti particle in the aluminium matrix improves the hardness of the composite and improves its ability to resist deformation [45]. Furthermore, the hardness of the composites improves as the amount of Al₃Ti is increased. The refinement of matrix phase also contributes to strengthening of the composites because the grain boundaries effectively resist the movement of dislocations during deformation. The generation of dislocation density around Al₃Ti particles due to difference in CTEs between Al matrix and Al₃Ti also improves the hardness of the composites [46]. Clean interface and better bonding between matrix and reinforcement increase load bearing capacity of the composite which leads to the improved hardness.

4.2. Tensile strength

The stress-strain curves of base aluminium alloy and the composites are shown in Fig. 10. Their average YS, UTS and % elongation values are listed in Table 4. It is observed that all the composites showed higher YS and UTS as compared to aluminium alloy. The improvements in YS are 12%, 25% and 42%, and in UTS are 35%, 45% and 60% for C1U, C2U and C3U composite, respectively over the starting alloy. Generally, the properties of matrix and reinforcement material govern the tensile properties of the composite material. The compatibility between reinforcement and matrix plays a significant role in improving the mechanical properties. The improvement in YS and UTS may be attributed to the interaction between dislocations and Al₃Ti particles when load is applied. These reinforcement particles resist the movement of dislocations and contribute to strengthening. Other contributing factors to increase of YS and UTS of the composites are the grain refinement and generation of dislocation density around the Al₃Ti particles due to the difference in CTEs between Al matrix and Al₃Ti

Table 3

<table>
<thead>
<tr>
<th>Material developed</th>
<th>Theoretical density (g cm⁻³)</th>
<th>Experimental density (g cm⁻³)</th>
<th>Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base Al alloy</td>
<td>2.70</td>
<td>2.55</td>
<td>5.5</td>
</tr>
<tr>
<td>C1U</td>
<td>2.72</td>
<td>2.68</td>
<td>1.5</td>
</tr>
<tr>
<td>C2U</td>
<td>2.73</td>
<td>2.67</td>
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</tr>
<tr>
<td>C3U</td>
<td>2.75</td>
<td>2.67</td>
<td>2.9</td>
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</table>

Table 4

<table>
<thead>
<tr>
<th>Casting condition</th>
<th>UTS (MPa)</th>
<th>YS (MPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base Al alloy</td>
<td>130 ± 5</td>
<td>59 ± 3</td>
<td>7.7 ± 2.1</td>
</tr>
<tr>
<td>C1U</td>
<td>175 ± 4</td>
<td>66 ± 2</td>
<td>13.8 ± 3.1</td>
</tr>
<tr>
<td>C2U</td>
<td>188 ± 3</td>
<td>74 ± 2</td>
<td>16.5 ± 1.5</td>
</tr>
<tr>
<td>C3U</td>
<td>208 ± 3</td>
<td>84 ± 2</td>
<td>23.5 ± 2.6</td>
</tr>
</tbody>
</table>
particle during solidification [47]. It should also be noted that the clean interface between matrix and reinforcement, and better distribution of reinforcement allow efficient load transfer from matrix to reinforcement which resist the crack initiation at the interface [48].

Ductility (% elongation) of C1U, C2U and C3U composite is improved significantly over the base alloy by 79%, 114% and 205%, respectively due to the retardation of crack propagation by the in-situ Al3Ti particles during deformation. Due to the presence of Al3Ti particles, the direction of crack growth is altered, which may cause crack branching, bridging and deviation of crack from its preferred path with respect to the loading direction. These processes consume more energy which ultimately enhances the work of fracture. Therefore, by increasing the resistance to crack propagation, the ductility of in-situ composite can be improved [8]. Another contribution to increased ductility is by the grain refinement wherein finer and more uniform sized grains exhibit better rheology during deformation delaying failure [49].

To study the effects of reinforcement particles on the strength of the composites, two approaches are considered which are based on continuum mechanics and micromechanics strengthening, respectively. In continuum mechanics, the load transfer to reinforcement particle is due to good bonding between the matrix and the particles. Whereas in micromechanics, the strengthening effect of the particles is the basis for improved strength. In the continuum mechanics approach, the yield strength of composites (σfc) can be expressed as

\[ σ_{fc} = σ_{ym}\left[ V_p \left( \frac{2 + S}{2} \right) + (1 - V_p) \right] \]  

where \( σ_{ym} \) is the yield stress of the matrix phase, \( V_p \) is the particle volume fraction in the composites which is 0.022 for C1U, 0.043 for C2U, 0.065 for C3U, and \( S(\mu) \) is the aspect ratio of the reinforcing particles which is 1.8, 2.8 and 2.7 for C1U, C2U and C3U, respectively.

In the micromechanics approach, the yield strength of the metallic matrix (σym) is calculated by considering the grain refinement strengthening (Δσgr), Orowan strengthening (Δσo) and strengthening due to thermal mismatch (ΔσCTE), and can be expressed as

\[ σ_{ym} = σ_o + Δσ_{gr} + \sqrt{(Δσ_o)^2 + Δσ_{CTE}^2} \]  

where \( σ_o \) is the yield strength of the as cast sample (base alloy). In the present study, the measured value of \( σ_o \) is 59 MPa.

In the strengthening due to grain refinement, grain boundaries resist the motion of dislocation due to the high lattice disorder of the grain boundary region and misorientation of adjacent grains [50]. By refining the grains of aluminium alloy, grain boundary area can be increased which further increase the resistance to the motion of dislocation improving the yield strength of the matrix. This improvement in yield strength due to grain refinement can be calculated by using Hall-Petch relationship

\[ Δσ_{gr} = k \left( d^{-\frac{1}{2}} - d_o^{-\frac{1}{2}} \right) \]  

where \( k \) is the Hall-Petch slope, which is \( \approx 74 \times 10^{-3} \text{MPa}\frac{\mu}{\text{m}} \) [51], \( d \) (μm) is the average grain size of the composites which is 104, 76 and 57 for C1U, C2U and C3U respectively and \( d_o \) (μm) is the average grain size of as-cast alloy which is 213 μm. By putting the values of \( d \), \( d_o \), and \( k \) in eqn. (8), the increment in the yield stress due to grain refinement for different composites is calculated and shown in Table 5.

The yield strength of the matrix is increased when particulate reinforced composites are quenched from the working temperature (750°C) to room temperature. According to the Taylor strengthening mechanism, volumetric strain occurs in the composite due to significant differences in CTE between the matrix and the reinforced particles. To accommodate the CTE difference, geometrically necessary dislocations (GND) are produced around the particles which increases the flow stress in the matrix [52]. Hence, the yield strength of the matrix is improved due to CTE mismatch strengthening (ΔσCTE) which can be described as

\[ Δσ_{CTE} = βG_b δ \]  

Where \( β \) is a constant, approximately equal to 1.25 [53]; \( G \) and \( b \) are the shear modulus of the matrix and the Burgers vector, respectively, whose values are 26 GPa and 0.286 nm respectively [51]; \( ρ \) is the dislocation density induced by the CTE mismatch, which can be estimated as

\[ ρ = \frac{12 Δ\alpha ΔT V_p}{b D(1 - V_p)} \]  

where \( Δ\alpha \) is the CTE mismatch between the matrix (25.2 × 10^{-6} \text{K}^{-1}) [51] and the reinforcing particles (13.0 × 10^{-6} \text{K}^{-1}) [54]; \( ΔT \) (K) is the difference between solidification start temperature (940 K) and test temperature (298 K); \( V_p \) is the volume fraction of the particles and \( D \) (μm) is the diameter of the Al3Ti particles which is 3.3, 2.9, and 3.8 μm for C1U, C2U and C3U, respectively. By using eqns. (9) and (10), the increment in the yield strength due to difference in CTE is calculated and shown in Table 5.

In Orowan strengthening, precipitate particles hinder the movement of dislocations. With the application of stress, the dislocation on interaction with the precipitates are first bowed, then reconnected, and finally the dislocation loops are formed around the particles. These dislocation loops hinder the movement of subsequently formed dislocations and thereby improve the strength of the composite [52].

However, the Orowan-strengthening is initiated when the reinforcement particles are smaller than 1 μm [52, 53]. In this work, the average size of the in-situ Al3Ti particles in all the composites are in the range of 2–4 μm. Hence, Orowan mechanism is not expected to contribute to strengthening. So, eqn. (7) can be modified as:

\[ σ_{ym} = σ_o + Δσ_{gr} + Δσ_{CTE} \]  

By using eqns. (6) and (11), the predicted value of \( σ_{ym} \) for all the composites is calculated and compared with the experimental values as shown in Table 5. It is observed that the predicted values of yield strength of C1U, C2U and C3U are computed to be 76.7 MPa, 88.4 MPa and 94.5 MPa, respectively, whereas the experimentally obtained values of yield strength of C1U, C2U and C3U are 66 MPa, 74 MPa and 84 MPa, respectively. The experimental values of yield strengths of C1U, C2U and C3U composites are 1.2, 1.2 and 1.1 times smaller than their respective theoretical values. This difference between the theoretical and experimental values may be attributed to the less uniformity in particle distribution in the matrix and measurement error of average grain size, average particle size and aspect ratio of the particle. Improvement in yield strength due to thermal mismatch is more as compared to that due to grain refinement which suggests that thermal mismatch strengthening is the dominant strengthening mechanism in in-situ Al3Ti titanium composites.

5. Fractography

The fracture surface analysis is very helpful to identify the contribution made by different mechanism to failure process and to identify the dominant failure mechanism. Therefore, fractographical examination which has been successfully used to understand the failure

<table>
<thead>
<tr>
<th>Casting condition</th>
<th>Δσ_{gr}(\text{MPa})</th>
<th>Δσ_{CTE}(\text{MPa})</th>
<th>σ_{ym} (MPa)</th>
<th>σ_{yc,e} (MPa)</th>
<th>σ_{yc,p} (MPa)</th>
<th>Absolute error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1U</td>
<td>2.2</td>
<td>14.0</td>
<td>75.2</td>
<td>76.7</td>
<td>66</td>
<td>16.2</td>
</tr>
<tr>
<td>C2U</td>
<td>3.4</td>
<td>21.0</td>
<td>83.4</td>
<td>88.4</td>
<td>74</td>
<td>19.4</td>
</tr>
<tr>
<td>C3U</td>
<td>4.7</td>
<td>23.0</td>
<td>86.7</td>
<td>94.5</td>
<td>84</td>
<td>12.5</td>
</tr>
</tbody>
</table>

Table 5 Predicted, experimental and contribution of different strengthening mechanisms to the yield strength of composites: \( σ_{ym} \), \( σ_{yc,e} \), \( σ_{yc,p} \), Predicted YS; \( σ_{yc,e} \), Experimental YS. |
mechanism of monolithic metals, was conducted on the fractured surface of the tensile specimens to identify that which failure mechanisms were operating in the composite. Fractured surface of the base alloy and the developed composites are shown in Fig. 11(a–d). The fractured surface of base alloy has cleavage facets and tear ridges which are indicative of brittle fracture. Few dimples and plastic slip bands are observed in C1U composite as shown in Fig. 11(b) which indicates the improvement in ductility of the composite over base alloy. It is observed from Fig. 11(c) and (d) that the number of dimples is increased and the size of dimples is reduced which may be attributed to the improvement in ductility and refinement of matrix as the amount of reinforcement is increased. The fractured surface of the composites indicates mixed mode fracture dominated by ductile fracture.

6. Summary

Different amounts of K2TiF6 powder was reacted with 6061 aluminium alloy at 750 °C for 5 min to form in-situ Al3Ti particles under ultrasonication to achieve better dispersion of intermetallics. Blocky morphology of in-situ formed Al3Ti particles was observed with the average size of 3.4 μm. All Al3Ti particles were observed within the grains instead of segregation at grain boundaries. The grain size of the composites decreased due to the presence of Al3Ti particles which acted as a nucleating agent and promoted heterogeneous nucleation during solidification, leading to improved yield strength, hardness and UTS. The best combination of mechanical properties with good ductility, yield strength and ultimate tensile strength are obtained in C2U composite with 5.4 wt % Al3Ti. The interface between Al3Ti particles and Al matrix was clear and well bonded which also played an important role in the improvement of mechanical properties. From the analysis, it is concluded that the thermal mismatch strengthening was the dominant strengthening mechanism followed by Hall-Petch strengthening. The contribution of Orowan strengthening mechanism is ruled out due to the coarseness of the Al3Ti particles.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.compositesb.2017.12.005.

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