Effect of in-situ formed Al₃Ti particles on the microstructure and mechanical properties of 6061 Al alloy

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Abstract. In this study, in situ Titanium-tri-aluminide (Al₃Ti) particles reinforced Al 6061 alloy matrix composites were fabricated by the reaction of potassium hexafluorotitanate (K₂TiF₆) inorganic salt with molten Al 6061 alloy via liquid metallurgy route. The development of in-situ Al₃Ti particles and their effects on the mechanical properties such as yield strength (YS), ductility, ultimate tensile strength (UTS) and hardness, and microstructure of Al 6061 alloy were studied. It was observed from the results that in-situ formed Al₃Ti particles were blocky in morphology whose average size was around 2.6 ± 1.1 µm. Microstructure studies showed that grain size of Al matrix was reduced due to the nucleating effect of Al₃Ti particles. It was observed from the mechanical properties analysis that when the volume fraction of Al₃Ti particles was increased, the hardness, UTS and YS of the composites were also increased as compared to that of Al 6061 alloy. An improvement in ductility was observed with the dispersion of Al₃Ti particles in base alloy which is contrary to many other composites.

1. Introduction

Particulate reinforced aluminium metal matrix composites (PRAMMCs), as structural engineering materials, are increasing more consideration in the aerospace and automobile industries due to their high mechanical properties and low density [1]. Various ceramic particulates such as Al₂O₃ [2], SiC [3], TiC [4], Si₃N₄ [5], AlN [6], TiB₂ [7] etc. have extensively been used in the fabrication of PRAMMCs which have some drawbacks such as significant difference in coefficient of thermal expansion (CTE) and densities between matrix and reinforcement. These drawbacks can be overcome by selecting trialuminide intermetallics as a reinforcement. Al₃Ti as a trialuminide intermetallics has very attracting properties such as high Young’s modulus (220 GPa), high temperature strength, good oxidation resistance and low density (3.4 g/cm³) [8, 9]. In addition, Al₃Ti, as a very powerful nucleating substrate, refine the Al matrix effectively [10].

The combination of the properties of the matrix, reinforcement and their interface generally govern the performance of PRAMMCs. For getting better mechanical properties, clean interface between matrix and reinforcement, small size of the reinforcement and small grain size of the matrix are always recommended. Based on this principle, Al₃Ti can be considered as a promising reinforcement for PRAMMCs. In-situ Al₃Ti particles are obtained when there is a direct reaction between the Ti element containing inorganic salts and Al melt. These Al₃Ti particles, which are nucleated and grown in the Al melt, have clean interface between the matrix and the reinforcement. In addition, these in-situ formed Al₃Ti particles has the advantage over other in-situ formed reinforcements because they act as a nucleation site for α-Al crystals and refine the Al matrix. In the present work, different wt % of K₂TiF₆ inorganic salt were added into the molten Al 6061 alloy to fabricate in-situ Al₃Ti reinforced Al 6061
alloy matrix composite. Their mechanical properties and microstructure have been investigated. The aim of this work is to fabricate in-situ composite by employing simple and low-cost method.

2. Experimental method
Al 6061 alloy and K$_2$TiF$_6$ salt powders are the materials used to fabricate composite. The chemical analysis of Al 6061 alloy is shown in table 1. In a muffle furnace, 350 g Al 6061 alloy was taken in a graphite crucible and heated up to 750 °C. When the temperature of molten metal reached to 750 °C, 10 or 15 wt % K$_2$TiF$_6$ salt powders, which was preheated at 200 °C, was added into the molten Al alloy. After powder addition, the melt was manually stirred for 5 minutes by using graphite rod to avoid the aggregation of in-situ Al$_3$Ti particles in the melt. After 5 minutes stirring, the slug on the surface of molten metal was removed and melt was poured into a metallic mould, which was preheated at 400 °C, for solidification. A control sample (without the addition of K$_2$TiF$_6$) was also casted by using the same experimental parameters. The composites prepared in the present work along with the Al$_3$Ti particulate reinforcement are given in table 2.

For microstructure analysis, small samples were sectioned from the developed composites and polished up to 2000 grit emery paper followed by cloth polishing with MgO abrasives. After cloth polishing, these samples were etched with poulton’s reagent to reveal the grain boundaries. Optical microscope (Leica, DMI 5000 M) and scanning electron microscope (Carl Ziess, EVO 18) were used for imaging. The average grain and particle size were calculated by using Image J software. For the x-ray diffraction analysis, Rigaku smartlab, X-ray diffractometer was used with Cu Kα radiation. The H25 K-S Tinius Olsen tensile testing machine was used to perform tensile test of a specimen having 20 mm gauge length and 4 mm gauge diameter with constant crosshead speed of 0.5 mm/min. The average value of three tensile test was reported along with standard deviation. At least five Vickers hardness values were taken by using Leitz Vickers Hardness testing machine under an applied load of 1kg and average value was reported along with standard deviation.

| Table 1: Chemical composition of Al 6061 alloy |
|---------------------------------------------|
| Elements | Wt% |
| Si       | 0.70 |
| Fe       | 0.18 |
| Cu       | 0.29 |
| Mn       | 0.33 |
| Mg       | 0.88 |
| Cr       | 0.006 |
| Zn       | 0.003 |
| Ti       | 0.02 |
| Al       | 97.591 |

| Table 2: Weight percentage of Al$_3$Ti. |
|---------------------------------------|
| Sample Label | K$_2$TiF$_6$ addition (wt %) | Al$_3$Ti content (wt %) |
|-------------|-------------------------------|------------------------|
| Sample A    | 10                            | 5.4                    |
| Sample B    | 15                            | 8.1                    |

3. Results and discussions
At 750 °C, when the salt powder was added into the molten Al 6061 alloy, it quickly reacted with melt as the melting point of K$_2$TiF$_6$ is 682 °C and the surface of melt was turned red. At this point, temperature of the melt recorded by thermocouple was over 850 °C which shows that huge amount of heat was produced during following exothermic reactions between the salt and aluminium.

$$3K_2TiF_6 + 13Al = 3Al_3Ti + K_3AlF_6 + 3KAlF_4$$

Gibbs free energy for the formation of Al$_3$Ti [12] at 750 °C (1023 K) is -122.8 kJ/mol, which confirms that reaction between inorganic salt K$_2$TiF$_6$ and Al6061 takes place spontaneously.

3.1 XRD analysis
The XRD analysis of the Al 6061 alloy, sample A and sample B are shown in figure 1. The XRD peaks corresponding to the Al₃Ti and Al are clearly identified in all the composites which confirm the formation of Al₃Ti particles. It is also observed from XRD analysis that on increasing the amount of Al₃Ti, the intensity of the Al₃Ti diffraction peaks is also increased.

3.2 Microstructural examinations

Figure 2 shows optical micrographs of Al 6061 alloy, Sample A and Sample B. It is observed from the micrograph that as the amount of salt is increased more amount of Al₃Ti is formed which leads to more clusters formation. These clusters are easily formed during casting because small particles have tendency to agglomerate together to decrease the free energy of the whole system [11]. In-situ formed Al₃Ti particles are observed within the grains instead of segregation at grain boundaries. It is observed from grain size measurement as shown in figure 3 that the average grain size of Al 6061, sample-A and sample-B are 135, 92 and 68 µm, respectively. The reduction in grain size is observed for all the composites as compared to Al 6061 alloy which may be attributed to the nucleation effect of Al₃Ti on α-Al [12]. Figure 4 shows SEM micrographs of in-situ formed Al₃Ti particles. It reveals that most of the Al₃Ti particles are blocky in shape and having average size of 2.6 ± 1.1 µm. There are no other casting defects such as porosity or shrinkages are observed in micrograph which showcase the quality of castings.

![Figure 1. XRD patterns of the Al6061 alloy and the fabricated composites.](image)

![Figure 2. Optical micrograph of (a) Al 6061 alloy, (b) Sample A and (c) Sample B](image)
3.3 Hardness
Figure 5 (a) shows the variation in Vickers hardness (VHN) of base alloy, sample A and sample B. It is observed from the figure that the hardness of sample A and sample B is increased as the amount of Al₃Ti is increased. The increase in hardness may be imputed to the generation of dislocations around the Al₃Ti particles due to difference in CTE of the Al matrix (25.2×10⁻⁶ K⁻¹) [13] and the Al₃Ti particles (13.0×10⁻⁶ K⁻¹) [14, 15]. Further, reduction in grain size of the matrix and the presence of hard Al₃Ti particles in the matrix also contributes to the hardness. Other factors which contribute to the hardness are clean interface and better bond between particles and matrix which increase the load carrying capacity of the composites [16]. The improvement in hardness of sample-A and sample-B over base alloy are around 20 % and 35 %, respectively.

3.4 Tensile properties
Figure 5 (b) shows variation in UTS and YS of Al 6061 alloy, sample A and sample B. It is observed from the figure that sample A and sample B showed higher UTS and YS as compared to base alloy. The maximum improvement in UTS (52 %) and YS (41 %) for sample B and the minimum improvement in UTS (42 %) and YS (25 %) for sample A have been observed as compared to base alloy. The tensile properties of composites are basically depended on the nature and properties of the
matrix material and reinforcing elements. The improvement in mechanical properties of the composites, particularly UTS, is strongly dependent on the compatibility between the reinforcement and the matrix. This enhancement in tensile strength may be imputed to the reduction in grain size of the matrix due to the nucleating effect of Al₃Ti particles. During solidification, the generation of dislocation density around the Al₃Ti particles due to mismatch of CTE of Al and Al₃Ti particles also contributes to strengthen of the composites [17]. These Al₃Ti particles provide hindrance to the movement of dislocations under load. In this condition, movement of dislocations is possible at much higher stress level than those required to move through matrix phase. Therefore, more load is required to nucleate and propagate voids which further enhance the tensile strength of the composites [18].

It is observed from the figure 5 (c) that the ductility or % elongation of the developed composites has increased significantly as compared to base alloy. Due to the presence of Al₃Ti particles in the matrix, the crack propagation is retarded when the direction of crack growth is changed and crack is deviated from its favourable orientation with respect to the direction of applied load. These processes consume more energy. Hence, by increasing the hindrance to crack propagation, the % elongation of the composites is also increased [16]. Further, % elongation of the developed composites is also increased when grain size is reduced which resist the crack propagation by convoluting the grain boundaries. Thus, the fracture toughness of the developed composites is enhanced which leads to improved ductility. However, at higher volume fraction of Al₃Ti, the ductility of the composite (Sample B) is reduced as shown in figure 5 (c) which may be attributed to the aggregation of Al₃Ti particles and result in early interface debonding of the matrix and easy crack propagation during tensile deformation [19].

**Figure 5.** Variation in (a) Hardness, (b) Tensile strength and (c) Ductility of Al6061 alloy and the developed composites
4. Conclusions
Different amounts of K$_2$TiF$_6$ powder was reacted with 6061 aluminium alloy at 750 °C for 5 min to form in-situ Al$_3$Ti particles. Blocky morphology of in-situ formed Al$_3$Ti particles was observed with the average size of 2.6 ± 1.1 µm. All Al$_3$Ti particles were observed within the grains instead of segregation at grain boundaries. The grain size of the composites decreased due to the presence of Al$_3$Ti particles which acted as a nucleating agent and promoted heterogeneous nucleation during solidification, leading to improved ductility, yield strength, hardness and UTS.

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