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Strengthening mechanisms in ultrasonically processed aluminium matrix composite with in-situ Al_3Ti by salt addition

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ABSTRACT

Al_3Ti reinforced Aluminium composites with different weight percent of Al_3Ti particles were developed by in-situ reaction of aluminium alloy with potassium hexafluorotitanate (K_2TiF_6). Ultrasonication of the aluminium melt during salt reaction was carried out to refine the cast microstructure and achieve better dispersion of in-situ formed Al_3Ti particles. The in-situ composites were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The Al_3Ti particles generated in the melt promoted heterogeneous nucleation, which was responsible for grain refinement of the cast microstructure. The well dispersed Al_3Ti significantly improved the mechanical properties including ductility, yield strength (YS), ultimate tensile strength (UTS) and hardness. The dominant strengthening mechanism in the composite was the thermal mismatch strengthening followed by Hall-Petch strengthening.

1. Introduction

Liquid Metallurgy approach to processing of aluminium matrix composites (AMC) 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 Al_3Fe, Al_3Ni, Al_3Zr and Al_3Ti have attracted attention due to their excellent mechanical properties, high specific strength, and high specific modulus, good oxidation and corrosion resistance [15–17]. Among these particles, Al_3Ti is very attractive as a reinforcement in AMC due to its low density (3.4 gcm^{-3}), high melting point (1350 °C) and high Young's modulus (217 GPa) [18]. Tetragonally structured Al_3Ti is strongly bonded with fcc α-Al due to thermodynamic equilibrium of Al_3Ti in Al matrix [19]. Moreover, Al_3Ti particles act as heterogeneous nuclei which lead to the grain refinement of α-Al phase [20]. Above the melting point of aluminium, Al_3Ti 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 Al_3Ti 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 cm^3 per 100 g), which is more than the industry standard which is close to 0.1 cm^3 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 10^{10} $K\,s^{-1}$, and localized pressure approaching 1000 atm [31].

In the present work, K_2TiF_6 inorganic salt was added into the Al melt to form in-situ Al_3Ti particles. Ultrasonication was applied for better distribution of salt particles throughout the molten metal. It is

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Table 1
Chemical composition of Al6061 alloy.

Elements	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
Wt%	0.70	0.18	0.29	0.33	0.88	0.006	0.003	0.02	97.591

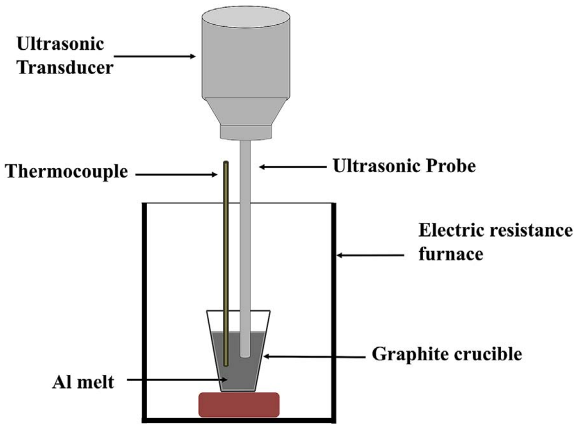

Fig. 1. Schematic diagram of ultrasonic assisted casting.

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 Al_3Ti reinforced aluminium matrix composite using low cost salt-metal reaction route.

2. Experimental setup

K_2TiF_6 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 $kWcm^{-2}$. 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 Al_3Ti 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 mild steel mould of dimension 40 × 40 × 120 mm^3 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 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^{-3}/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.

Table 2
Intermetallic content in each sample.

Sample Label	K_2TiF_6 addition (wt %)	Al_3Ti content (wt %)
Base Al alloy	0	0
C1U	5	2.7
C2U	10	5.4
C3U	15	8.1

3. Results and discussion

The melting point of K_2TiF_6 is 682 °C. The K_2TiF_6 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.

$$3K_2TiF_6 + 13Al = 3Al_3Ti + K_3AlF_6 + 3KAlF_4 \quad (1)$$

Gibbs free energy for the formation of Al_3Ti at 750 °C (1023 K) is −122.8 kJ/mol [33], which confirms that reaction between inorganic salt K_2TiF_6 and Al6061 takes place spontaneously.

3.1. XRD analysis and in-situ formation of Al_3Ti particles

To verify the completion of the reaction, separate XRD analysis was done for aluminium reinforced composites which formed with varying amounts of K_2TiF_6 added to the melt (Fig. 2). The XRD peaks corresponding to the in-situ formed Al_3Ti particles are clearly identified in all the compositions. Moreover, the relative intensity of the Al_3Ti diffraction peaks increased as the amount of K_2TiF_6 increased. In the present work only Al_3Ti particles are present. Other intermetallics, such as Al_2Ti, $AlTi_3$ and elemental Ti reported in other studies were not detected suggesting that the reaction between dissolved Ti and Al could have gone to completion.

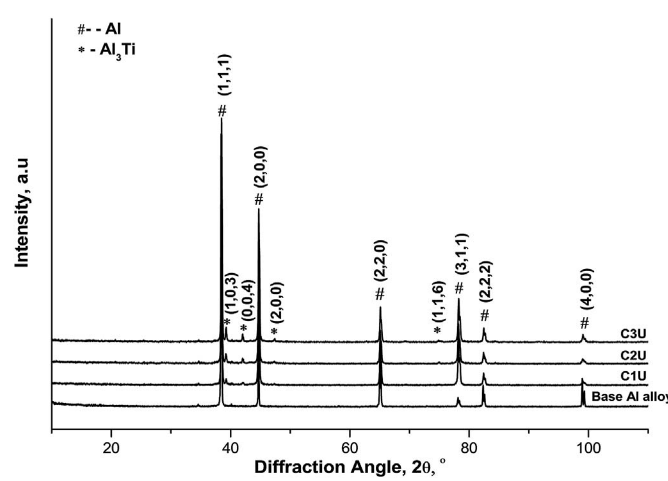

Fig. 2. XRD patterns of the fabricated composites and base Al alloy.