



# Al-(L1<sub>2</sub>)Al<sub>3</sub>Ti nanocomposites prepared by mechanical alloying: Synthesis and mechanical properties

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## ABSTRACT

The present paper reports the formation of nanocomposites containing  $\alpha$ -Al and L1<sub>2</sub>-Al<sub>3</sub>Ti in binary Al-Ti alloys containing 5, 10, 15, and 20 at.% Ti. Supersaturated solid solution of Ti in Al was observed after 20 h of mechanical alloying irrespective of the Ti content. Nanocomposites formed after mechanical alloying and subsequent annealing at 673 K for 2 h showed a high hardness (3.4 GPa), modulus (106.4 GPa) and density (95%). Hardness values of Al-(L1<sub>2</sub>)Al<sub>3</sub>Ti nanocomposites obtained by nanoindentation were similar to hardness measured with microhardness. High strength of the nanocomposites was attributed to the formation of large volume fraction of nanocrystalline L1<sub>2</sub>-Al<sub>3</sub>Ti particles and the ultrafine grain size of  $\alpha$ -Al matrix.

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## 1. Introduction

Many of the currently available Al alloys do not exhibit satisfactory results at high temperature [1] and have a useful upper limit of 598 K. The reason being the dispersoids present in Al alloys coarsen too fast at this temperature and thus the strength and creep resistance are likely to degrade. In conventionally processed metal matrix composites (MMCs), by powder metallurgy route, the reinforcing particles are formed prior to their addition to the matrix metal [2] and the scale of reinforcing phase is limited by the starting powder size, which is typically of the order of several to tens of micrometers and rarely below 1  $\mu$ m. It is widely recognized that the mechanical properties of MMCs are controlled, to a large extent, by the size and volume fraction of the reinforcements, as well as, the nature of the matrix–reinforcement interface [2]. Superior mechanical properties can be achieved when fine and stable reinforcements with good interfacial bonding are dispersed uniformly in the matrix. A possible alternative is to form the reinforcement *in situ* in the metal matrix [3]. The advantages of *in situ* MMCs are that they have more homogeneous microstructure and are thermodynamically more stable. Moreover, they also have strong interfacial bonding between the reinforcements and the matrices. *In situ* aluminum matrix composites may be fabri-

cated by various techniques, such as conventional ingot metallurgy, mechanical alloying (MA), rapid solidification processing (RSP), and combustion synthesis. However, materials produced by MA, RSP or combustion synthesis must be densified, by a hot consolidation process such as hot isostatic pressing/extrusion [4–6] or spark plasma sintering [7–9], in order to obtain the final product.

Aluminum alloys reinforced with trialuminide particles (Al<sub>3</sub>Ni, Al<sub>3</sub>Fe, Al<sub>3</sub>Ti, Al<sub>3</sub>Zr, etc.) possess high specific strength, high specific modulus, and excellent properties both at ambient and elevated temperatures [10–12]. In comparison to most other aluminum-rich intermetallics, Al<sub>3</sub>Ti is very attractive because it has higher melting point (~1623 K) and relatively low density (3.4 g/cm<sup>3</sup>). Further, Ti has low diffusivity and solubility in aluminum and hence Al<sub>3</sub>Ti is expected to exhibit a low coarsening rate at elevated temperature [13]. In addition, the Young's modulus of Al<sub>3</sub>Ti phase is reported to be 216 GPa [14]. Therefore, the presence of Al<sub>3</sub>Ti phase is very effective in increasing the stiffness of aluminum alloys. Al–Al<sub>3</sub>Ti nanocomposites have been fabricated by RSP [12,15,16] and MA [17,18] to achieve high strength. The major strengthening mechanisms, which contribute to the high strength of Al–Al<sub>3</sub>Ti alloys, have been suggested to include Orowan strengthening, grain size strengthening, and load-shearing effects of Al<sub>3</sub>Ti particles [12,19,20].

The objective of the present work was to produce fully dense Al-based nanocomposites, with different volume fractions of nanometer-sized metastable L1<sub>2</sub>-Al<sub>3</sub>Ti intermetallic as reinforcement particle, by MA. The basic idea of fabricating the Al-based nanocomposites by MA lies in examining the stability of the metastable L1<sub>2</sub>-Al<sub>3</sub>Ti phase so as to maintain the Ni-based

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**Table 1**

Compositions and conditions of MA used in the present study for the synthesis of Al-based nanocomposites.

Systems	Compositions studied	Milling conditions
Al–Ti	Al <sub>100-x</sub> Ti <sub>x</sub> for x = 5, 10, 15 and 20	Mill: Planetary Fritsch P5  Vials and balls: Tungsten carbide Milling speed: 300 rpm Milling medium: Toluene Milling time: 20 h Diameter of balls: 10 mm BPR = 10:1

superalloy like microstructure both at room temperature, as well as, at elevated temperatures. Also, MA was adopted for the synthesis of nanocomposites because, (a) being a far-from-equilibrium process provides ample scope for easy formation of metastable phase like L1<sub>2</sub> structure [21–24], (b) involves severe plastic deformation to promote uniform mixing and refining of the constituent phases in the all materials, and (c) easy consolidation and subsequent annealing for good densification, as reported recently by the present authors [23,24].

## 2. Experimental details

Table 1 illustrates the conditions of MA used and the compositions studied in the present work. Raw materials used for MA were high purity elements viz. Al (99.9 wt.%) and Ti (99.7 wt.%). Al powder was of particle size less than 45 μm (–325 mesh) and Ti was used in the form of sponge. To avoid oxidation of powders toluene was used to fill the vials up to a level to just cover the balls and powders. The cold compaction of MA powders was carried out using a load of 375 MPa into pellets of 12 mm diameter. The cold compacted pellets were sintered at 573 K and 673 K for 2 h under a flowing argon atmosphere. In addition, mechanically alloyed Al–Ti alloy powders were hot pressed at 673 K using a load of 490 MPa into a cylindrical shape of 20 mm diameter. Density of green compacts, annealed compacts and hot pressed samples was calculated using simple Archimedes' principle.

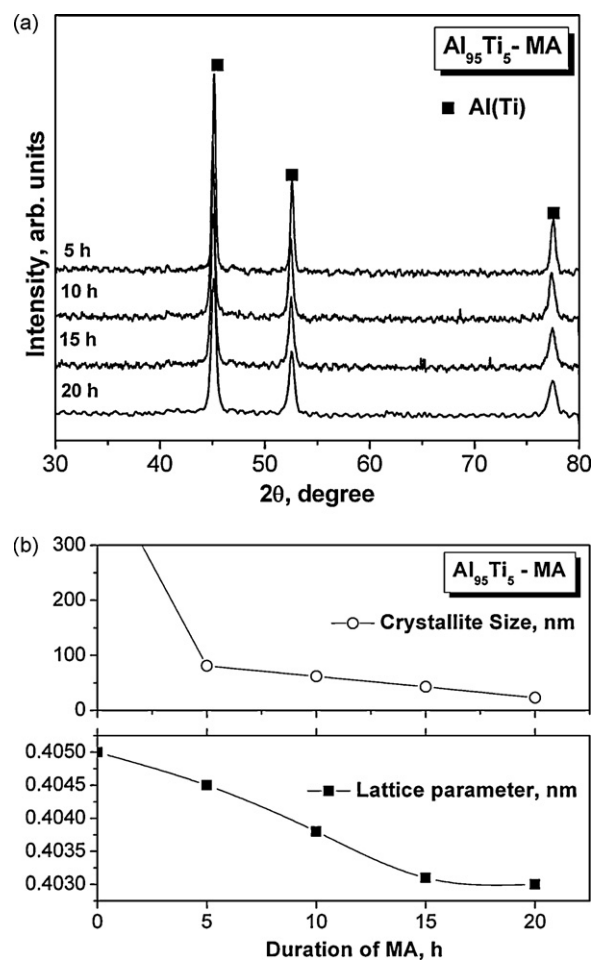
Mechanically alloyed samples and cold-consolidated pellets were characterized by Phillips PW 1710 X-Ray Diffractometer using Co-K<sub>α</sub> radiation, whereas phase analysis of annealed samples was done using PHILIPS X'Pert PRO using Cu-K<sub>α</sub> radiation. Crystallite size of mechanically alloyed powder particles was determined from the X-ray peak broadening by the peak profile analysis using Voigt's single peak method [25] after eliminating the instrumental broadening. Grain coarsening and phase transformation, if any, also were analyzed using X-ray diffraction (XRD). Microhardness was measured under a load of 300 g with a dwelling time of 15 s using a LECO DM-400 hardness tester. An average of at least 10 measurements was taken for each microhardness value. Nanoindentation on pellets of Al–Ti alloys was carried out for the measurement of hardness and elastic modulus in a MTS Nano Indenter<sup>®</sup> XP using a diamond Berkovich indenter with equilateral triangular faces.

## 3. Results and discussion

### 3.1. MA of Al–Ti alloys

Fig. 1a shows the XRD patterns of Al–5Ti<sup>1</sup> alloy after different duration of MA. Peaks of Ti were not observed within 5 h of MA suggesting the formation of Al(Ti) supersaturated solid solution (SSSS) that was stable even after prolonged MA, i.e. for 20 h. Close observation of the XRD patterns showed a continuous broadening of XRD peaks with duration of MA, indicating refinement of powder crystallites. The crystallite size measured using (1 1 1) peak profile of Al was 81 nm after 5 h of MA (Fig. 1b). The crystallites got refined further with prolonged MA and the size reached a value of 23 nm after 20 h of MA (Fig. 1b). A gradual decrease in crystallite size from 81 nm (after 5 h of MA) to a final size of 23 nm (after 20 h MA) can be seen from the plot of crystallite size and duration of MA (Fig. 1b). Apart from peak broadening, a small shift in the peak position towards the higher diffraction angle was observed (Fig. 1a)

<sup>1</sup> All compositions reported in this work are in atomic percentage, unless otherwise specified.



**Fig. 1.** (a) XRD patterns of Al<sub>95</sub>Ti<sub>5</sub> alloy after 5, 10, 15 and 20 h of MA and (b) variation of lattice parameter and crystallite size of Al<sub>95</sub>Ti<sub>5</sub> alloy with duration of MA.

indicating a decrease in the lattice parameter of the Al as illustrated in Fig. 1b. This suggests incorporation of Ti into the Al lattice. Formation of metastable or stable intermetallics such as Al<sub>3</sub>Ti was not observed in Al–5Ti blend up to 20 h of MA. So, it may be concluded that all of Ti are in the form of SSSS.

Similar to Al–5Ti blend, rest of the compositions also showed evidence of formation of SSSS of Ti in Al after 20 h of MA (Fig. 2a). In order to elucidate this aspect, variation of lattice parameter of Al with duration of MA in all the alloys is plotted in Fig. 2b. From the difference in lattice parameter [26] of the Al after 0 h and 20 h of MA in the Al–Ti alloys studied, it can be inferred that 2–3% Ti has dissolved in Al (Table 2). This value is higher than that previously reported by MA [27]. Kim et al. [27] have reported Ti solubility of 2.4 wt.% (1.37%) in Al–20 wt.% Ti (12.3%Ti) alloy. Furthermore, as there was no indication of formation of any new phase after 20 h of MA, it may be concluded that all Ti are in the SSSS formed during MA. In addition to dissolution of Ti into Al, there was evidence of refinement of the crystallites, as indicated by the peak broadening in XRD (Fig. 2a). A continuous decrease

**Table 2**

Lattice parameter, Ti solubility (as calculated from the change in lattice parameter of Al) and crystallite size of Al–Ti alloys after 20 h of MA.

Composition	Lattice parameter, nm	Solubility, %	Crystallite size, nm
Al–5Ti	0.4030	2.0	23
Al–10Ti	0.4027	2.3	21
Al–15Ti	0.4026	2.4	20
Al–20Ti	0.4020	3.0	19

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