

Preparation and thermomechanical properties of stir cast Al–2Mg–11TiO₂ (rutile) composite

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Abstract. Al–2Mg–11TiO₂ composite was successfully prepared by the conventional vortex method. The macrostructural observation revealed columnar structure with rutile particles being distributed throughout the matrix in the form of agglomerates. Microstructural observation showed the presence of micro voids in the particle-enriched zone. Electrical resistivity measurement showed a phase transformation at 360°C, which was consistent during DSC studies due to the precipitation of TiAl₃ phase. As-cast composite was both hot rolled and cold rolled successfully to 50 and 40% reduction, respectively. The mechanical properties of the thermomechanically-worked composite were studied. From fractographic analysis, it was clear that the crack had nucleated at the particle/matrix interface and propagated through the matrix by microvoid coalescence. Ultimate tensile strength of cold worked composite was found to be better than the hot worked material.

Keywords. Metal matrix composites; stir cast; rutile.

1. Introduction

The need for the lightweight materials having high strength and high stiffness in automotive and aerospace application have generated considerable interest to develop Al based metal matrix composites (Rohatgi 1991). Among the various methods to fabricate metal matrix composites, liquid metallurgy vortex method has drawn keen attraction among the researchers due to its industrial feasibility. The major limitation in many cases in fabricating metal matrix composites by liquid phase route resides upon the incompatibility of the reinforcement and the matrix (Alonso *et al* 1993). This problem in case of Al-based metal matrix composite is due to the formation of stable tenacious oxide film, resulting in poor wettability with the ceramic particle (Chu and Wu 1999). One of the common practice to improve wettability of an Al melt is through addition of small amount of reactive metals like magnesium, titanium etc (Rohatgi *et al* 1986) prior to the incorporation of ceramic particle. In the present work, 2 wt% magnesium was added to Al to improve wettability.

The objectives of the present investigation were to prepare Al–2Mg–11TiO₂ by liquid metallurgy vortex method and study its thermomechanical behaviour through metallography. Physical properties were also measured. Fractured surfaces of the composites were also studied under scanning electron microscope and the observations were

correlated with the tensile properties. Electrical resistivity and differential scanning calorimetric studies were carried out to understand the effect of ageing on the mechanical properties of the composite.

2. Experimental

500 g of commercial grade Al (99.8%) was taken to prepare the composite by stir casting. The average size of rutile powder used in this work was 113 µm with a standard deviation of 38 µm. This size range was chosen because with finer particles (< 20 µm) rejection rate was very high and only limited amount could be retained in the melt (Maity *et al* 1995). Al was melted in electric resistance furnace and the temperature was maintained at 800°C. A known amount of rutile powder (preheated to 200°C) was added, while the melt was stirred with a graphite stirrer at a rotation speed of 200 rpm. Prior to powder addition, about 2 wt% magnesium was plunged into the melt to increase wettability of the melt. Technical details of the process is described elsewhere (Maity *et al* 1993). Density of both as-cast and mechanically worked composite was measured using Archimedes' principles. The resulting densities are shown in table 1 with calculated volume fractions of porosity.

Electrical resistivity as a function of temperature was measured by a d.c. four-probe method in an electrical resistivity unit (TER-2000, Ulvac/Sinku-Rico, Japan), where the temperature was varied from 50–500°C. The dimension of the specimen for resistivity measurement was

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$20 \times 3 \times 0.65$ mm. The calorimetric study was carried out using a Perkin-Elmer DSC-7 calorimeter. The rate of heating was $10^\circ\text{C}/\text{min}$ in case of both resistivity and DSC measurements. Isothermal electrical resistivity was done at the temperature corresponding to the onset of physical transformation for 30 min. The X-ray diffractograms of the composite were taken using Siemens-D500 diffractometer using CoK_α radiation. The microstructural characteristics were studied using optical microscope (Lietz make). Ageing was conducted at 300°C and 400°C in a resistance furnace by holding the thermomechanically processed composite samples for various intervals of time and then quenching in oil. Hardness was measured using a standard Vicker's hardness testing machine by applying 1 kg load. Tensile testing was done in Honsfield tensometer at the crosshead speed of 3 mm/min. Fractographic analysis of the thermomechanically processed composite materials was studied under scanning electron microscope (Jeol JCXA 733 analyser).

3. Results and discussion

3.1 Processing and structure

The composite was cast in a rectangular cast iron die (preheated at 600°C) of dimension $120 \times 65 \times 9$ mm. Macrostructural observations (figure 1) revealed columnar structure with finer structure at the top and bottom of the casting and coarser equiaxed in the centre region. The optical micrograph of as cast composite showed considerable segregation of particles. Particles were agglomerated in the form of cluster within microvoids (figures 2a,b). The porosity level was greatly reduced by hot and cold rolling as shown in figures 3a and b, respectively. This is reflected in the increase of density of the mechanically worked composite as shown in table 1.

The back scattered image and line profile of Al, Ti, Mg and O across TiO_2 particle in the composites are shown in figures 4a–e. It indicates preferential segregation of Mg at the interface. This is due to the formation of Al_2MgO_4 during preparation of the composite. The formation of Al_2MgO_4 spinels is due to the *in situ* reaction between liquid Al and TiO_2 particles.



Figure 1. Macrograph of stir cast Al-2Mg-11TiO₂ composite.

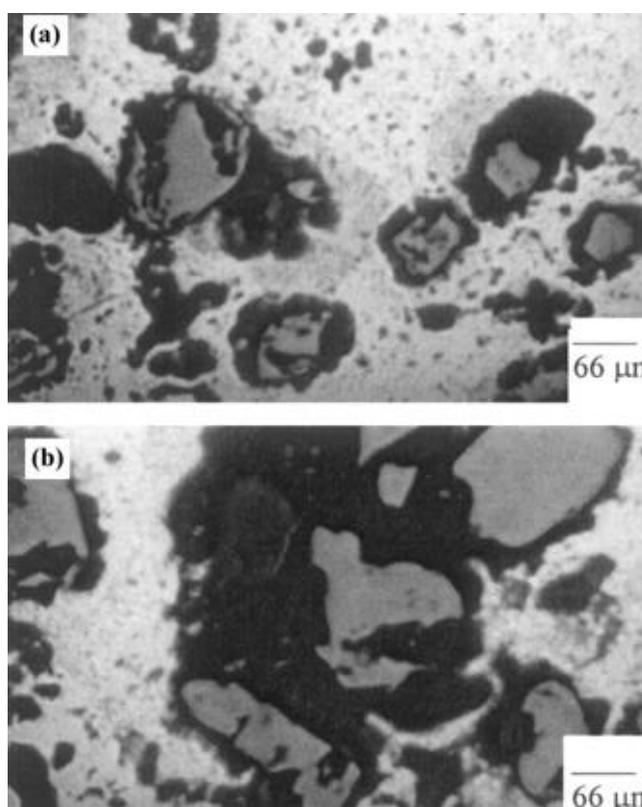


Figure 2. Optical micrograph of as-cast Al-2Mg-11TiO₂ composite: (a) top and (b) bottom of the casting.

Table 1. Results of density measurements.

Parameter	As-cast			Hot rolled	Cold rolled
	Top	Centre	Bottom		
Density (g/cc)	2.29	2.43	2.52	2.65	2.68
V_f of porosity (%)*	18.0	13.0	9.7	5.1	4.0

*The volume fraction of porosity was calculated assuming that the distribution is uniform throughout the matrix.

3.2 Physical properties

Figure 5 shows the variation of electrical resistivity with temperature for as-cast composite and the base alloy (Al-2Mg). It was observed that electrical resistivity of the alloy increased with the addition of ceramic particles. This was due to the low conductivity of rutile particle. Both the base alloy and the composite showed a linear rise of resistivity with temperature at the rate of 0.011 $\mu\text{m}/^\circ\text{C}$ up to 360°C, where a peak was observed only in case of composite (figure 5a). Further, the isothermal resistivity measurement of Al-2Mg-11TiO₂ composite done at 360°C showed that the phase transformation was over in 8 min (figure 5b). DSC measurement showed a broad exothermic peak with its onset at 375.52°C and peak at 386.83°C (figure 6). These peaks indicate phase transformation. There was a difference of about 15°C in the onset values between the electrical resistivity and DSC measurements. This could be because electrical resistivity measurements are more sensitive to the changes at atomic level, whereas the DSC peaks correspond to bulk phenomena and hence the response was less sensitive. The XRD results of the composite aged at 300°C and 400°C are shown in figures 7a and b, respectively. In the composite aged at 400°C, Al₃Ti was present whereas in the composite aged at 300°C, it was absent. This indi-

cates that Al₃Ti phase was formed due to an *in situ* reaction between Al and rutile particles. This has also been observed by the presence of exothermic peak in DSC studies and deviation in linearity during electrical resistivity measurement.

3.3 Mechanical property

The study on hardness measurements revealed that the addition of rutile particles tend to increase the hardness of as-cast composites (43.2 VPN) as compared to the base alloy (39.2 VPN). The hardness of the composite increased appreciably with mechanical working. The hardness of cold rolled and hot rolled composites was 84 VPN and 78 VPN, respectively. The effect of ageing on hardness of the cold rolled composite is shown in figure 8. The decay in hardness was more rapid in case of composite aged at 400°C *vis-à-vis* with those aged at 300°C. The hardening mechanism in the composite is due to matrix strengthening resulting from cold working. The ageing curve indicated that the decrease in hardness with time is due to softening of the matrix. To understand the effect of precipitation of Al₃Ti phase on the tensile properties of the composite, ageing of cold rolled samples was carried out at 300°C (below onset of phase transfor-

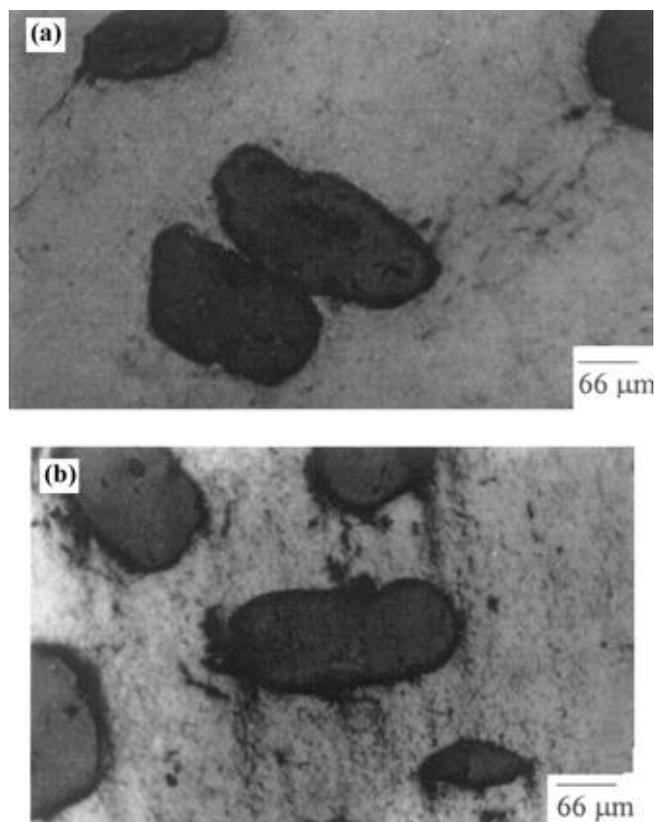


Figure 3. Optical micrograph of Al-2Mg-11TiO₂ composite: (a) hot rolled and (b) cold rolled.

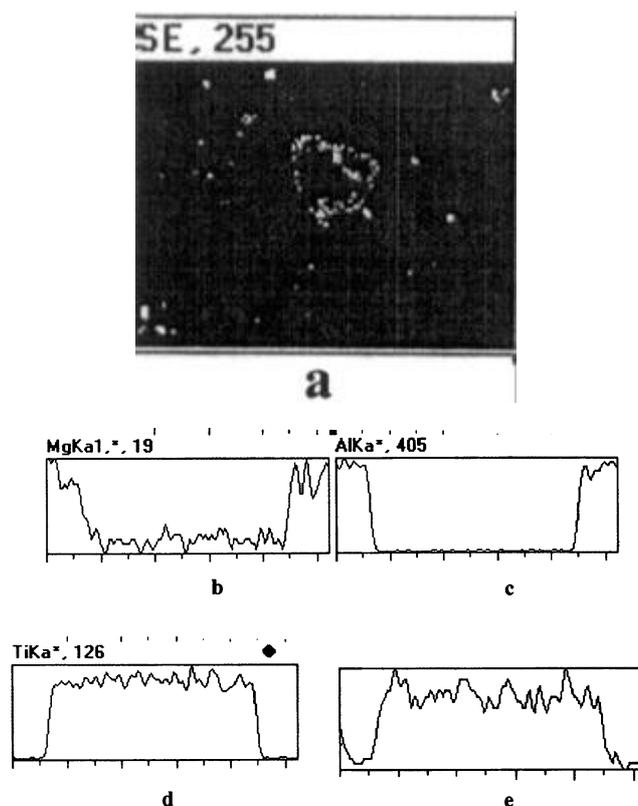


Figure 4. Line profile of stir cast Al-2Mg-11TiO₂ composite: a. back scattered electron image, b. Mg, c. Al, d. Ti and e. O.

mation temperature) and 400°C (above onset of phase transformation temperature) for 8 min (holding time) and then quenched in oil. Hot rolling of the as-cast composite was done at 400°C. The result is shown in table 2. The

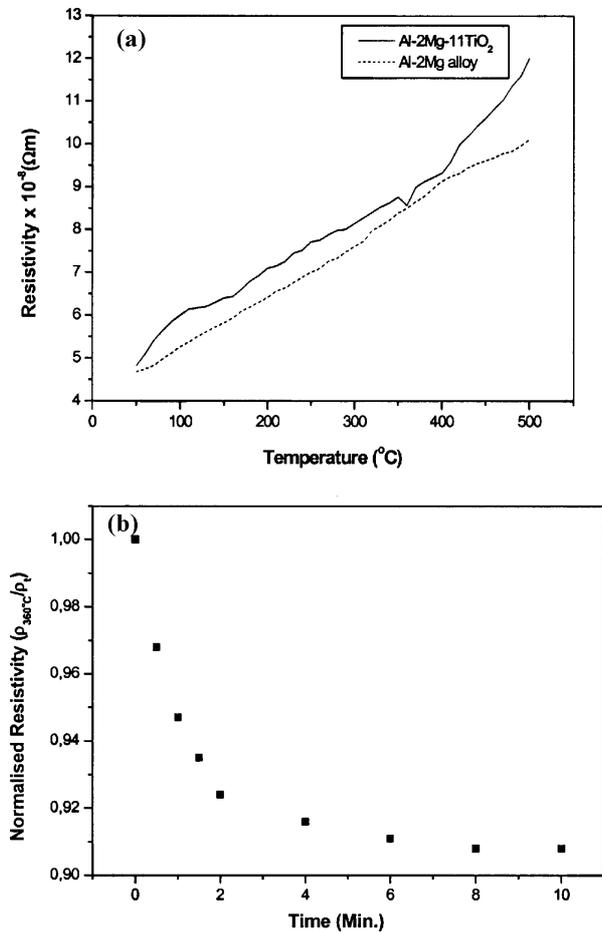


Figure 5. (a) Variation of electrical resistivity with temperature and (b) normalized resistivity plot of as-cast Al-2Mg-11TiO₂ composite.

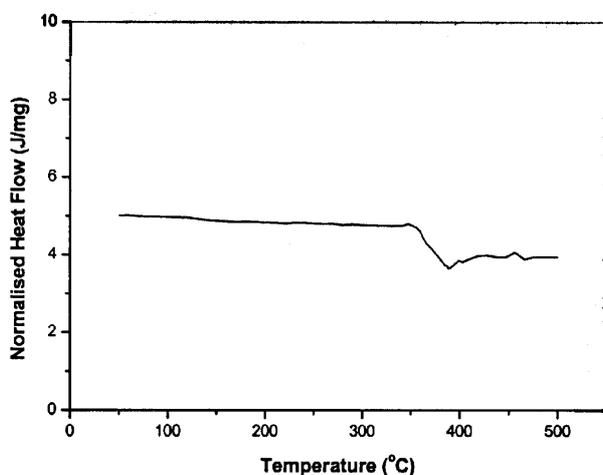


Figure 6. DSC plot of Al-2Mg-11TiO₂ composite.

ultimate tensile strength of the cold rolled composite was slightly higher than the hot rolled sample, but the elongation of former was much lower than the latter. This was because cold rolling resulted in greater degree of work hardening than hot rolling. Although ultimate tensile strength of samples aged at 400°C was little higher than that aged at 300°C, the increase in ductility of composite aged at 400°C over the composite aged at 300°C was remarkably high. This showed that precipitation of Al₃Ti particles might have played a vital role in strengthening the interface that lead to the better performance of the composite aged at 400°C. However, further investigations are needed in this direction.

The fractographic analysis of fractured sample aged at 400°C revealed that the crack had nucleated at the particle matrix interface (figure 9) and then propagated through the matrix by inter void coalescence. Similar obser-

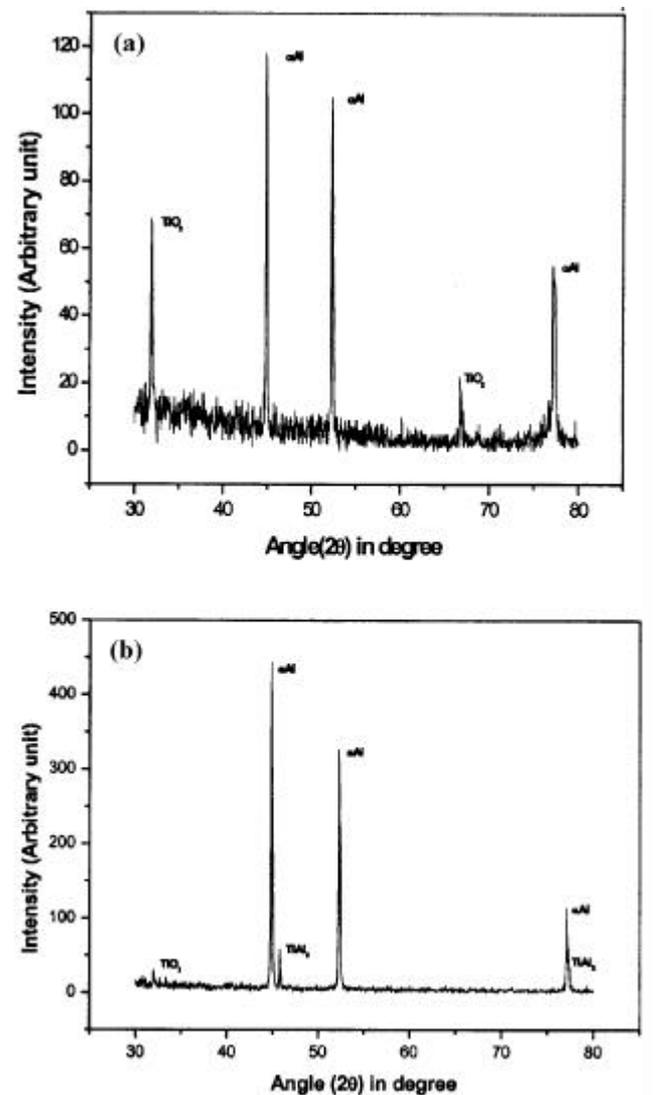
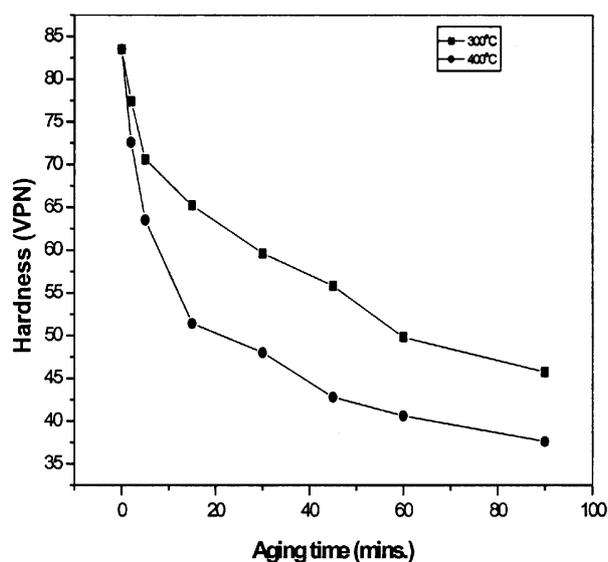
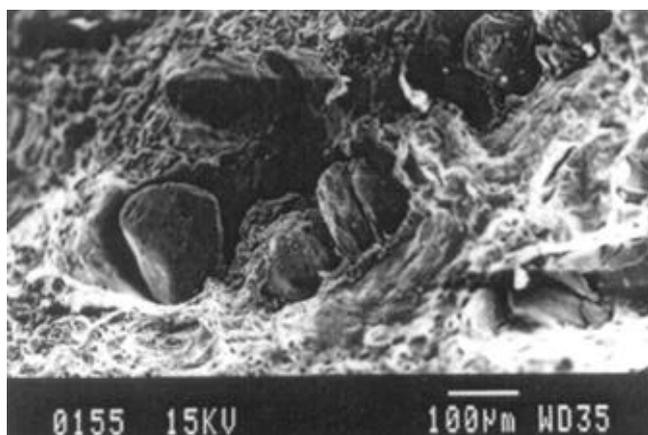


Figure 7. XRD plot of Al-2Mg-11TiO₂ composite aged at (a) 300°C and (b) 400°C.

Table 2. Tensile properties of Al-2Mg-11TiO₂ composites at different conditions.

Tensile properties	Rolling		Ageing after cold roll for 8 min	
	Hot	Cold	300°C	400°C
U.T.S. (MPa)	76	108	95	113
Elongation (%)	4.7	2.5	5.4	6.3

**Figure 8.** Ageing behaviour of Al-2Mg-11TiO₂ composite.**Figure 9.** SEM fractograph of Al-2Mg-11TiO₂ composite aged at 400°C.

variations are reported in the literature (Bhanuprasad *et al* 1995). The high percentage of elongation was supported by the presence of dimples on the fractured sample of the composite (Vanstone *et al* 1985).

4. Conclusions

11 wt% of TiO₂ could be successfully incorporated into the Al-2Mg melt by vortex method. Greater degree of segregation of TiO₂ has been observed at the top and bottom of castings. Microvoids are observed in the particles enriched zone. A phase transformation has been observed during the resistivity and DSC measurements owing to the precipitation of TiAl₃ phase. Cold and hot rolling of the composite is successfully carried out to 40% and 50% reduction, respectively. Hardness of the composite is greater than the base alloy, which can be attributed to the presence of higher dislocation density in the matrix due to the difference in thermal properties between the matrix and dispersoids. The improved tensile behaviour of cold rolled composite aged at 400°C could be due to the precipitation of the Al₃Ti phase.

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