## **Abstract**

The present work deals with the preparation and thermo-mechanical processing of Al-5Ti and Al-5Ti-1B master alloys that are used for grain refinement of Al and its alloys. The present detailed study is aimed at understanding the influence of melt temperature during master alloy preparation and their subsequent thermo-mechanical processing by rolling, extrusion and heat treatment on the microstructure and in turn on the grain refining efficiency of binary Al-5Ti and ternary Al-5Ti-1B master alloys. The major objectives of the present study are:

- > To understand the influence of thermo-mechanical treatments such as rolling, extrusion and heat treatment on the microstructure, particularly the morphology, size and size distribution of the TiAl<sub>3</sub> particles.
- > To establish the correlation between the morphology, size and size distribution of TiAl<sub>3</sub> particles on the grain refining performance of the master alloys.
- > To improve the grain refining performance of Al-Ti and Al-Ti-B master alloys prepared at high reaction temperature, which usually have poorer grain refining performance.
- > To optimise the thermo-mechanical processing conditions during the manufacture of master alloy rods.

In the present investigation, Al-5Ti master alloys have been prepared both by the reaction of K<sub>2</sub>TiF<sub>6</sub> salt with molten Al (at 800 and 1000°C for 60min), and by melting of Ti sponge together with Al at 1200°C. Ternary Al-5Ti-1B master alloys have been prepared by salt route by the reaction of molten Al with K<sub>2</sub>TiF<sub>6</sub> and KBF<sub>4</sub> salts at 800 and 1000°C for 60min. The ternary master alloy prepared at higher temperature (1000°C) has been rolled at 200, 300 and 400°C for reduction levels of 20, 40, 60 and 80%. Based on the grain refining performance of the rolled master alloys, the rolling temperature of 300°C has been used for the rolling of binary master alloys prepared at higher melt temperatures of 1000 and 1200°C. The ternary master alloy has also been extruded at 300°C at extrusion ratios of 6.86, 9.25, 16.02 and 22.55. Both the binary and ternary as cast master alloys have been heat treated at 300, 400, 500 and 600°C for 4h. In order to study the influence of thermal

exposure after deformation, the extruded ternary master alloys have also been heat treated at 300, 400, 500 and 600°C for 4h. Both the as cast and thermo-mechanically processed master alloys have been characterized by XRD, optical microscopy, SEM, EDX microanalysis and TEM. Commercial purity Al has been grain refined at 720°C for different holding times up to 120min, using both the as cast and thermo-mechanically processed binary and ternary master alloys in order to assess their grain refining performance.

The present results indicate that blocky TiAl<sub>3</sub> particles form at lower melt temperature and plate like particles at higher melt temperatures during the master alloy preparation, irrespective of the processing route. The results point out that both the binary and ternary master alloys with blocky TiAl<sub>3</sub> particles are fast acting grain refiners and have better grain refining efficiency than the ones containing plate like particles, which are slow acting.

Thermo-mechanical processing by rolling, extrusion and heat treatment has strong influence on the microstructure and grain refining performance of both binary and ternary master alloys. Rolling and extrusion resulted in the fracture of plate like TiAl<sub>3</sub> particles present in the as cast master alloys resulting in a change in the morphology of the particles to blocky type also leading to a reduction in the particle size and increase in the number of fine particles. The increased number of particles by rolling and extrusion has led to an improvement in the grain refining performance of both binary and ternary master alloys.

Heat treatment of both binary and ternary as cast master alloys has resulted in an increase in the amount of TiAl<sub>3</sub> by the precipitation of fine TiAl<sub>3</sub> particles from the supersaturated solid solution of Ti in  $\alpha$ -Al. The supersaturation of  $\alpha$ -Al is caused by the sluggishness of TiAl<sub>3</sub> precipitation from  $\alpha$ -Al while cooling the casting from the peritectic temperature to the room temperature. The fine TiAl<sub>3</sub> particles precipitated during heat treatment appear to act as additional nucleating sites for Al during grain refinement studies, thus improving the grain refining performance of the master alloy.

Heat treatment after extrusion appears to result in the dissolution of TiAl<sub>3</sub> and TiB<sub>2</sub> particles, particularly at the highest extrusion ratios studied. This could be due to reduced stability of these intermetallic particles caused by their fine particle size and the presence

of high stress concentration at their vicinity at larger deformation levels. Such dissolution of intermetallic particles appears to result in a loss of grain refining efficiency suggesting that heterogeneous nucleation by these intermetallic particles is responsible for the grain refinement achieved by these master alloys.

In summary, the present work has not only led to the understanding of the influence of thermo-mechanical processing on the microstructure and grain refining performance of Al-5Ti and Al-5Ti-1B master alloys but also has identified the optimum thermo-mechanical processing conditions for the benefit of Al industries.