

# Effect of equal channel angular pressing on microstructure and grain refining performance of Al–5%Ti master alloy

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## ABSTRACT

The Al–5%Ti master alloy has been subjected to the equal channel angular pressing (ECAP) by route A at room temperature, and the grain refining performance of Al–5%Ti master alloy before and after ECAP with the use of an industrial purity Al has been evaluated. The effects of the ECAP on the size and the distribution of the TiAl<sub>3</sub> particles, grain size, microhardness and electrical conductivity of pure Al ingots with and without adding the Al–5%Ti master alloy have been investigated. The large platelet-like TiAl<sub>3</sub> particles were fragmented into the fine blocky TiAl<sub>3</sub> particles from ~270 μm to ~13 μm uniformly distributed in the Al matrix after three ECAP passes. It has been revealed that microhardness of pure Al cast samples have been increased by adding the Al–5%Ti master alloy processed by ECAP; however, the electrical conductivity has deteriorated. The Al–5%Ti refiner processed by ECAP appeared to have an enhancing grain refining effect in comparison with that before ECAP. It has been proved that the grain size of α-Al could be well fitted by the length of TiAl<sub>3</sub> particles and the growth restriction factor. The conclusion has been made that it is practical to enhance the grain refining effectiveness of the Al–5%Ti master alloy through the ECAP process by no more than three passes.

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## 1. Introduction

The classical nucleation theory believes that most nucleation occurs heterogeneously through the contribution of foreign substrates in the melt that provide effective sites to reduce the nucleation energy barrier. Accordingly inoculation by adding particles (such as particles of master alloys) which can act as substrates for heterogeneous nucleation is widely practised in the aluminum industry [1]. It is well documented that TiAl<sub>3</sub>, TiB<sub>2</sub> and TiC particles in Al–Ti, Al–Ti–B and Al–Ti–C master alloys are responsible for the heterogeneous nucleation of the melts, resulting in a finer grain structure of the as-cast materials.

Using an edge-to-edge matching crystallographic model, Zhang et al. [2] predicted that TiAl<sub>3</sub> is a more powerful nucleating substrate than TiC and TiB<sub>2</sub>. In the Al–5%Ti–1%B master alloy, TiAl<sub>3</sub> particles are found at grain centers and the TiB<sub>2</sub> particles at grain boundaries, indicating that when both particles are present TiAl<sub>3</sub> particles are the preferred nucleation sites [2,3]. TiAl<sub>3</sub> particles usually appear as platelet shaped in the Al–Ti–C master alloy, which dissolve slowly and impair the assistant function of Ti on grain refinement. The addition of fine blocky TiAl<sub>3</sub> particles (~20 μm) in the Al–10%Ti master alloy can improve the nucleation

rate of TiC particles because the fine blocky TiAl<sub>3</sub> particles dissolve in three-dimensional space [4], leading to Ti atoms release in a short time from TiAl<sub>3</sub> particles.

There have been a lot of controversies on the effect of the morphologies of TiAl<sub>3</sub> particles on the refining effectiveness. Usually TiAl<sub>3</sub> phases have three different shapes, namely, the petal-like shapes, the platelets and the blocky crystals, depending on the temperature history of the master alloys [3]. The grain refining efficiency of petal and plate-like structures improved with time and survived longer, and it faded quickly for blocky crystals [5]; in other words, its grain refining effectiveness decreased after a long contact time in the melt. However, Liu et al. [6] believed that the TiAl<sub>3</sub> morphologies do not have an essential effect on the grain refining efficiency of Al–Ti–C master alloys. Excluding the role of TiC particles, Li et al. [7] examined the morphologies of TiAl<sub>3</sub> in Al–Ti master alloys and their refining efficiency. They found that the large plate-like TiAl<sub>3</sub> crystals faded away markedly; however, for the finer blocky particles the grain refining effect remained for the whole holding time (~1.5 h).

Equal channel angular pressing (ECAP) is considered as the most promising severe plastic deformation (SPD) technique for tailoring the ultrafine microstructure, distribution and size of particles in composites [8]. The microstructure and the grain refining performance of the Al–5%Ti–0.25%C alloy processed by ECAP were investigated by Zhang et al. [9]. It was found that the mean size of TiC and TiAl<sub>3</sub> particles decreased dramatically, and a

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double grain refining effect appeared in the Al–5%Ti–0.25%C refiner in comparison with that before ECAP, which was significantly determined by the finer TiC and TiAl<sub>3</sub> particles. Moreover, Al–5%Ti master alloys processed by 15 passes of ECAP appeared to have a better grain refining efficiency than that before ECAP, which was mainly contributed to the fine particles and the TiAl<sub>3</sub> precipitates from the supersaturated solid solution [10]. In fact, it is difficult to bring into effect in the industrial practice that a refiner was repetitively processed by 15 ECAP passes. It should be mentioned that there is a transition for the TiAl<sub>3</sub> phase from platelet shapes to blocky particles during ECAP [9,10]. Therefore, it is indispensable to examine the effect of the morphology, size and distribution of TiAl<sub>3</sub> particles on the grain refining performance of the Al–5%Ti master alloy before and after ECAP.

The present work is initiated to explore the possibility of enhancing the grain refining efficiency of the Al–5%Ti master alloy through the ECAP process with no more than three passes. The effect of ECAP on the distribution and the size of TiAl<sub>3</sub> particles, grain size, microhardness and electrical conductivity of pure Al ingots with and without adding the Al–5%Ti master alloy have been investigated, and the grain refining performance of the Al–5%Ti alloy before and after ECAP with the use of an industrial purity Al has been evaluated.

## 2. Experimental procedures

The Al–5wt% Ti master alloy ingots with a dimension of 500 × 250 × 40 mm<sup>3</sup> were supplied by Xuzhou Huasheng Aluminum Co. Ltd. After removing the oxidation layer and surface defects, the billets with a size of 12 × 12 × 80 mm<sup>3</sup> were cut in the longitudinal section. ECAP was conducted at room temperature with a pressing speed of 0.2 mm s<sup>−1</sup> using a die with  $\phi=110^\circ$  and  $\psi=0^\circ$  [8]. All samples were pressed using a molybdenum disulfide lubricant and without rotation between each pass, which has been designated as route A [11]. The equivalent strain is 0.8 for each pass [12].

For studying the grain refinement efficiency of the Al–5%Ti refiner before and after the different number of ECAP passes, pure aluminum (99.7% Al) was melted at 1033 K in an Al<sub>2</sub>O<sub>3</sub> crucible using a 5 kW electric resistant-heating furnace. The addition of Al–5%Ti refiner before and after ECAP into the Al melt was 0.57 wt%, and the resultant Ti concentration in the cast alloy was 0.03 wt%. The melt was poured into a ring steel mold with an outside diameter of 45 mm and inside diameter of 25 mm. Each test sample was cut horizontally at 20 mm distance from the bottom. The sectioned surface of the sample was polished and etched in a hydrofluoric acid (2.5 ml), nitric acid (12.5 ml) and water (85 ml) reagent, and then the mean size of  $\alpha$ -Al grains was calculated by using a mean linear intercept method.

The macrostructural evolution of pure Al cast samples with Al–5%Ti alloy before and after ECAP was observed by an optical microscope. All samples were polished and etched in a hydrofluoric acid (1 ml), nitric acid (5 ml) and hydrochloric acid (5 ml) reagent. Microhardness was measured by using HVS-5Z with a load of 300 g for 15 s at different locations. Electric conductivity was measured by a 7501-A eddy current electric conductivity instrument with an accuracy of  $\pm 2\%$ .

## 3. Results and discussion

### 3.1. Optical microstructure of the Al–5%Ti alloy before and after ECAP

Optical photographs of the Al–5%Ti alloy before and after ECAP are shown in Fig. 1. The needle-like TiAl<sub>3</sub> particles were observed in the as-cast Al–5%Ti alloy before ECAP and were reported to have a platelet shape [10,13,14]. The TiAl<sub>3</sub> particles were obviously fragmented and aligned along the direction of ECAP shearing deformation in Fig. 1(b) and (c). After three ECAP passes, the platelet-shaped TiAl<sub>3</sub> particles almost disappeared, and have been transformed into fine and blocky particles, which were uniformly distributed in the Al matrix as shown in Fig. 1(d).

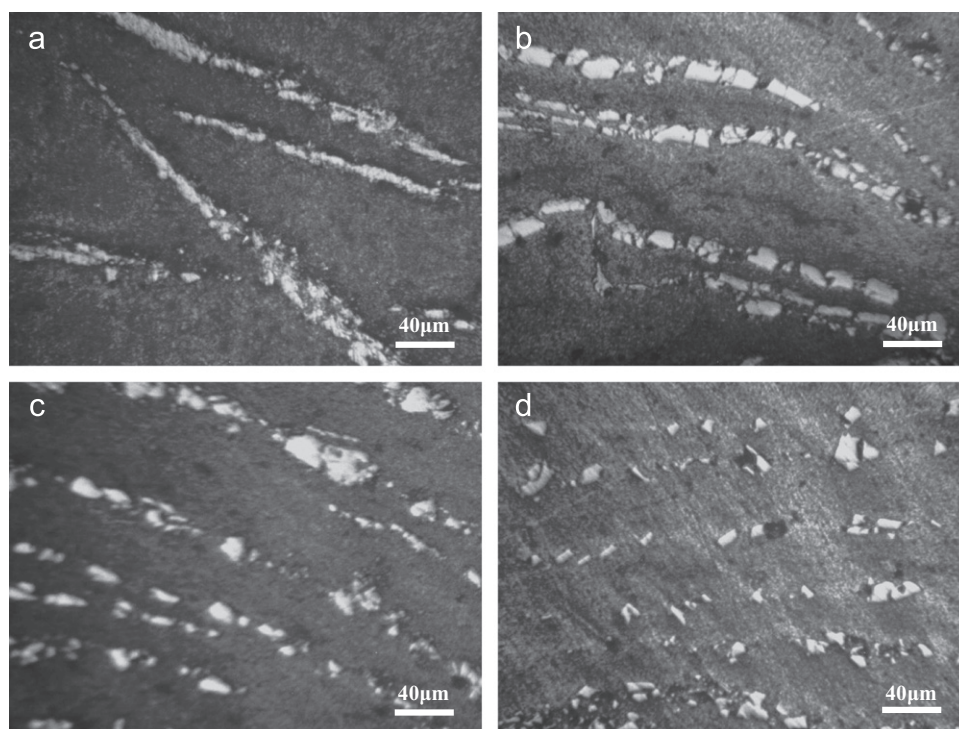


Fig. 1. Optical micrographs of Al–5%Ti alloy before and after ECAP at different passes: (a) as-cast, (b) one pass, (c) two passes, and (d) three passes.

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