TWIN-ROLL CAST ALUMINIUM ALLOYS PROCESSED BY ECAP

Michaela Poková, Miroslav Cieslar and Přemysl Málek

Charles University in Prague, Faculty of Mathematics and Physics, Department of Physics of Materials, Ke Karlovu 5, 121 16, Prague 2

pokova@karlov.mff.cuni.cz

Abstract

Two aluminium alloys from the AA3003 series (Al-Mn-Fe-Si), one of them with the addition of zirconium, were prepared by the twin-roll casting method. These highly supersaturated alloys were heat treated at 450 °C in order to form small coherent Al₃Zr particles, which are known to enhance recrystallization resistance of deformed aluminium alloys. Afterwards the alloys were subjected to severe plastic deformation by equal channel angular pressing (ECAP) with 1 and 4 passes. The microstructure after ECAP was investigated by means of light and transmission electron microscopy. Thermal stability of both alloys at elevated temperatures was monitored by Vickers microhardness measurements. The addition of zirconium was found to have a positive effect on the thermal stability and to shift recrystallization to higher temperatures.

Keywords: Aluminium alloys, Al₃Zr, ECAP, TEM, Thermal stability

1. INTRODUCTION

Materials prepared by twin-roll casting (TRC) have a high potential for industrial applications as this method leads to energy and material savings as compared to conventional casting methods. Their microstructure and mechanical properties are different from the materials prepared by conventional casting due to a high solidification rate [1]. Higher solid solution supersaturation, finer grain size and finer dispersion of secondary particles represent the main benefits [2].

Thermal stability at elevated temperatures is important for technical applications. It is known that one of the most efficient methods how to stabilize the microstructure of aluminium alloys is the addition of a small amount of zirconium or scandium or their combination [3, 4]. The stabilizing effect of Sc is better in comparison with Zr, however a high price of Sc limits its use in industry [5, 6]. Coherent Al₃Zr precipitates are formed during a suitable heat treatment and they can pin moving dislocations and grain boundaries and thus enhance the recrystallization resistance of aluminium alloys [7, 8].

Methods of severe plastic deformation (SPD) introduce a large amount of deformation into the materials and result generally in a strongly reduced grain size and enhanced strength of materials. One of the most promising SPD techniques is equal channel angular pressing (ECAP). Its main advantage is that the shape of the material is preserved after ECAP and the deformation step can be repeated. Many works have been devoted to the investigation of the influence of ECAP on mechanical properties of aluminium alloys [9-12]; however, the experiments on the twin-roll cast sheets, which exhibit higher solid solution supersaturation and different second phase particles distribution, are very scarce.

In the present work we focused on the influence of the addition of Zr and of the number of ECAP passes on the microstructure evolution of a TRC aluminium alloy and on its thermal stability and recrystallization behaviour during annealing at elevated temperatures.

2. EXPERIMENTALS

We studied two modifications of an aluminium alloy from the AA 3003 series (1.0 – 1.5 wt.% Mn, ≤ 0.7 wt.% Fe, ≤ 0.6 wt.% Si, 0.05 – 0.2 wt.% Cu and ≤ 0.2 wt.% Zn), one of them with the addition of 0.16 wt. % of Zr.
The twin-roll cast materials were subjected to annealing from room temperature to 450 °C with a heating rate 0.5 °C/min and subsequently held at this temperature for 8 hours. This treatment leads to the precipitation of Al5Zr particles in the Zr-containing alloy [13]. Samples with the cross section of 10x10 mm² were afterwards processed by ECAP at room temperature, using route Bc and pressing speed 10 mm/min.

The grain size and grain structure were studied by the means of light microscopy. The samples were treated by electrolytic etching with Barker solution and observed in polarized light. The microstructure was studied using transmission electron microscopy (TEM).

The thermal stability and restoration processes at elevated temperatures were monitored by Vickers microhardness measurement performed post-mortem at room temperature.

3. RESULTS

3.1 Microstructure of the as prepared materials

Figure 1a shows that a high density of secondary particles nucleated in both alloys during annealing at 450 °C. These particles were identified mainly as the \( \alpha \)-Al\(_{15}\)(Mn,Fe)\(_3\)Si\(_2\) phase [14]. The dark field image (Figure 1b) reveals the presence of Al5Zr precipitates in the Zr-containing alloy.

The light optical micrographs document that the grains preserve their elongated shape resulting from the original TRC process after 1 ECAP pass. On the contrary, four ECAP passes completely destroy this elongated structure and a hardly recognizable equiaxed microstructure is formed (Figure 2b).

No significant differences were observed between both alloys in the deformed state by TEM. The microstructure after 1 ECAP pass consists of high number of subgrains of sub-micron size and the dislocation density is high (Figures 3a, 3b). After four ECAP passes, the grains and subgrains are well defined, maintaining the sub-micron size (Figures 3c, 3d) and the dislocation density is much lower.

![Fig. 1: (a) Particles of the \( \alpha \)-Al\(_{15}\)(Mn,Fe)\(_3\)Si\(_2\) phase and (b) Al5Zr precipitates (dark field image) after annealing at 450 °C for 8 hours in the Zr-containing material.](image-url)
Fig. 2: The light optical micrographs of the grain structure of the Zr-containing alloy after 1 ECAP pass (a) and 4 ECAP passes (b). The direction of pressing is vertical.

Fig. 3: (a), (b) Dislocation substructure and Al₃Zr precipitates after 1 ECAP pass. (c), (d) Subgrains and grains after 4 ECAP passes in the Zr-containing alloy.

3.2 Thermal stability at elevated temperatures
The samples of both alloys after 1 and 4 ECAP passes were subjected to isochronal annealing at temperatures ranging from 100 to 550 °C with a heating scheme 50 °C/25 min. In order to monitor softening processes and recrystallization, Vickers microhardness with a load of 100 g (HV0.1) was measured after quenching from the annealing temperature at room temperature. Figure 4 shows that the microhardness of the un-annealed samples increases with the number of ECAP passes. For comparison the HV values of both...
materials prior to ECAP are 49.1 MPa for the alloy without Zr and 50.3 MPa for the Zr-containing alloy, respectively [13]. The addition of Zr enhances the HV values not only in the initial state after ECAP but during the whole annealing cycle.

The decrease in HV0.1 values occurring during isochronal annealing can be divided into two stages. At lower annealing temperatures, the HV0.1 values decrease only moderately with increasing annealing temperature, nevertheless, a significant reduction of microhardness is achieved during this stage. This reduction is more pronounced in materials after 4 ECAP passes for both alloys. During the second stage, a steep drop of HV0.1 associated with full recrystallization was observed in all materials. The onset of this stage depends both on the number of ECAP passes and on the presence of Zr. While the increasing number of ECAP passes displaces the onset of the recrystallization stage to lower temperatures, the addition of Zr displaces it to considerably higher temperatures. The lowest temperature of the onset of recrystallization stage (~ 300 °C) was found in the material without Zr after 4 ECAP passes, the highest one (~ 450 °C) was found in the Zr-containing alloy after 1 ECAP pass.

The light microscopy clearly confirms (Figure 5) that after annealing to 550 °C the microstructure is fully recrystallized with newly formed grains. The impact of initial rolling during TRC is still apparent in the materials processed only by one ECAP pass; however the grains are more equiaxed as compared to the initial state after ECAP. While the grain size is slightly smaller in the Zr-free material after ECAP, the final grain size after annealing up to 550 °C is much higher in the Zr-free material.

![Figure 4](image_url)

**Fig. 4:** The evolution of Vickers microhardness (HV0.1) for both materials (Al-alloy and Al+Zr-alloy) during isochronal annealing up to 550 °C.
Fig. 5: The light optical micrographs of the alloys after annealing up to 550 °C: the Zr-free alloy after 1 (a) and 4 ECAP passes (b), the Zr-containing alloy after 1 (c) and 4 ECAP passes (d).

4. DISCUSSION

Similarly to other Al-based alloys, the addition of zirconium has a positive effect on microstructure stability and enhances the microhardness also in the Al-Mn-Fe-Si alloy. Tiny coherent Al₃Zr precipitates with the average size of 10 nm successfully inhibit the grain boundary migration at lower temperatures. This can be demonstrated by the evolution of Vickers microhardness: the recrystallization temperature of the Zr-containing alloy was displaced to higher values by approximately 50 °C in the material processed by 4 ECAP passes and even by 100 °C in the material after 1 ECAP pass.

The equal channel angular pressing introduces deformation into the material. After one pass the microstructure contains sub-micrometric subgrains with high dislocation density. Further ECAP passes causes dynamic recovery of the matrix and creation of smaller subgrains or grains with much lower dislocation density as compared to the material after 1 pass. Thanks to higher stored deformation energy the materials after 4 ECAP passes exhibit higher initial values of Vickers microhardness. The stored deformation energy is connected with the driving force for recovery and recrystallization. Thus the continuous drop of HV0.1 at the first stage of annealing resulting form recovery is steeper and recrystallization which is responsible for the HV0.1 reduction in the second stage starts at lower temperatures for the materials subjected to 4 ECAP passes.

5. CONCLUSION

The addition of zirconium to the AA 3003 alloy and the annealing at 450 °C lead to precipitation of fine Al₃Zr particles. These precipitates enhance significantly the recrystallization resistance of the alloy during
annealing at high temperatures and reduce the grain growth. The ECAP results in a significant reduction of the grain size.

ACKNOWLEDGEMENTS

This work was financially supported by grants GAUK 1428213, SVV-2013-267303 and GAČR P107-12-0921.

REFERENCES


