THE INFLUENCE OF ECAP ON MECHANICAL PROPERTIES OF A TWIN-ROLL CAST Al-Mn-Fe-Si-Zr ALLOY

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Abstract

The Al-Mn-Fe-Si-Zr alloy was twin-roll cast and a part of the material was after casting annealed at 450 °C for 8 hours in order to allow the precipitation of the Al_3Zr phase. Both modifications were further processed by equal channel angular pressing (ECAP) at room temperature. The influence of increasing number of ECAP passes on the microstructure was studied using transmission electron microscopy. The character of interfaces formed during ECAP was studied using electron back-scatter diffraction. The room temperature mechanical properties were investigated by microhardness measurements and by tensile tests. It was found that the highest increase in strength characteristics occurs already during the first ECAP pass when the dislocation density is enormously increased. A partial recovery of dislocation structure and formation of high angle boundaries were observed during following ECAP passes. Consequently, the increase in strength characteristics reveals a saturation effect especially in the annealed material.

Keywords: Aluminium alloys, TRC, ECAP, microstructure, strength

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 when compared to conventional casting methods. Additionally, the high solidification rate around 500 °C/s during TRC [1] results in microstructure and phase composition modifications - finer grain size, higher solid solution supersaturation, and finer dispersion of secondary particles [2] which can improve the mechanical properties of these materials.

The grain size is one of the principal tools for controlling mechanical properties of polycrystalline materials because the reduction in grain size results generally in a strength increase at lower temperatures and a formability enhancement at elevated temperatures. Severe plastic deformation (SPD) of bulk billets achieves the ultra-fine grained structure by imposing a very high plastic strain [3]. The most common SPD technique is equal channel angular pressing (ECAP) where the sample is pressed through a die consisting of two channels of equal cross-section that intersect at an angle usually 90° [4]. The main advantage of this technique is the possibility of repeating the pressing several times to induce a required level of strain into the material.

It is well known that the addition of small amount of zirconium or scandium leads to the enhancement of mechanical properties of aluminium alloys (e.g. [5-7]). After heat treatment at 450 °C [8] coherent precipitates of cubic Al_3Zr or Al_3Sc phase form in the aluminium matrix. These particles pin moving grain boundaries, and thus refine the grain size and shift the onset of recrystallization to higher temperatures. This effect has been observed in both conventionally cast and twin-roll cast Al-Mn-based alloys after various methods of deformation and also after cold-rolling [9].

The influence of $Al_3(Zr,Sc)$ particles is very important especially in alloys prepared using ECAP with ultrafine-grained structure. The stability of this structure at very high temperatures allows superplastic behaviour (e.g. [10, 11]). Much less information is available on the influence of these particles on the course of ECAP process and on the resulting microstructure and strength characteristics. The results of such investigation performed on the Al-Mn-based alloy are presented in this paper.

2. EXPERIMENTALS

We studied 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) with the addition of 0.16 wt.% of Zr. A part of the twin-roll cast material was 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 is known to lead to the precipitation of Al_3Zr particles [8, 12]. Simultaneously, particles of the α-$Al_{15}(Mn,Fe)_3Si_2$ phase are formed and the content of Mn in Al matrix decreases. Samples with the cross section of 10x10 mm^2 were afterwards processed by ECAP at room temperature to 1, 2, and 4 passes using route B_c and pressing speed 10 mm/min. At the annealed material, even 8 passes were possible. On the other site, the non-annealed material exhibited cracking at higher number of passes and the preparation of the material wit 8 passes was not possible.

Because of very fine microstructure, the grain structure was not studied by the means of light microscopy. The information on the microstructure was therefore obtained from transmission electron microscopy. Additional information on the grain shape, grain size distribution, and on the character of interfaces was obtained from electron back-scatter diffraction (EBSD) experiments. The samples were prepared by electro-polishing in nitric acid in methanol solution. The images were taken in the Y plane [4] after ECAP.

The mechanical properties were studied at room temperature as a function of the number of ECAP passes by Vickers microhardness measurement (load 100 g) and by uniaxial tensile test at the strain rate of 10^{-3} s^{-1}. The flat samples with the thickness of 1 mm were cut using a diamond saw parallel to the Y plane of ECAP pressings, so that the specimen tensile axis was parallel to the pressing direction.

3. RESULTS

3.1 Mechanical properties

Fig. 1 shows the results of tensile tests. It is clear that the increasing number of ECAP passes shifts the stress vs. strain curves to higher stresses. Simultaneously, a drop in formability occurs. It follows form **Fig. 1** that the main change in deformation behaviour occurs already during the first ECAP pass. Further ECAP passes cause only moderate strength increase and do not influence significantly the ductility.

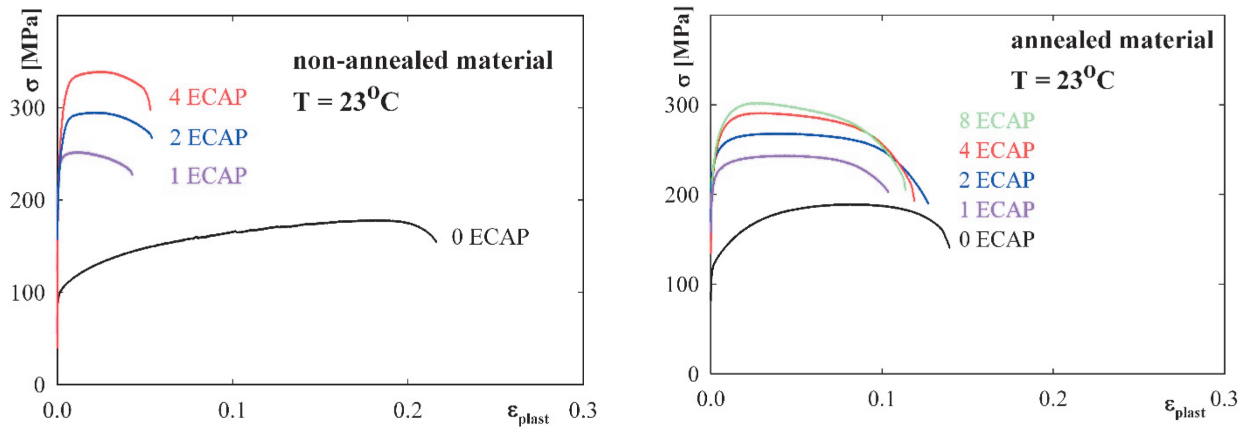


Fig. 1 The influence of the number of ECAP passes on the stress vs. strain curves for non-annealed (left) and annealed (right) material

Fig. 1 documents also the differences in the behaviour of non-annealed and annealed materials. The higher strength of the annealed material prior to ECAP results from the precipitation hardening especially by Al_3Zr particles. However, an opposite effect was observed already after the first ECAP pass. The strength increase caused by ECAP is significantly higher in the non-annealed material so that the non-annealed material becomes stronger. This behaviour can be seen also in **Fig. 2** where the microhardness values (left) and ultimate tensile strength (right) are plotted as a function of the number of ECAP passes. Both dependences are qualitatively very similar and document the tight interconnection between both these mechanical characteristics. The higher strengthening effect of ECAP in the non-annealed material can be explained by the higher content of Mn remaining in the solid solution. Mn atoms are very effective in hindering dislocation motion and suppression of recovery processes. Therefore, the deformation energy stored in the non-annealed material is higher than in the annealed one.

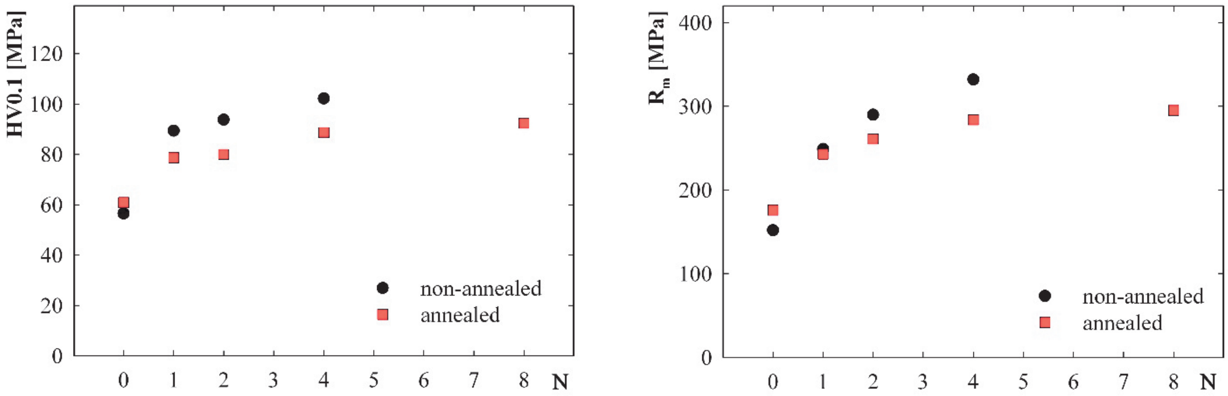
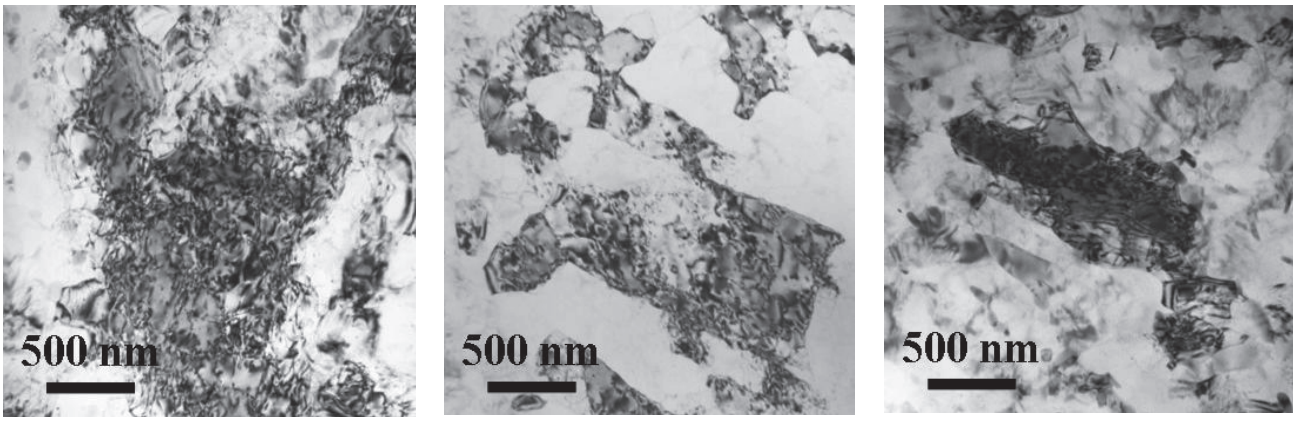


Fig. 2 The influence of the number of ECAP passes on microhardness values (left) and ultimate tensile strength (right) for both materials

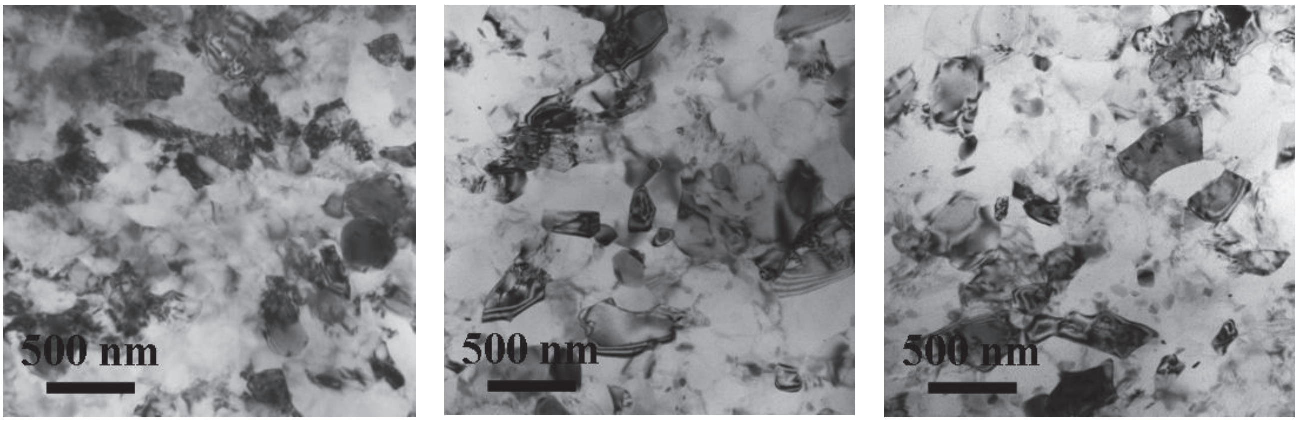
3.2 Microstructure after ECAP

The initial structure of both non-annealed and annealed materials contains elongated grains formed during TRC [12]. The main difference between both materials is the presence of Al_3Zr and α-$Al_{15}(Mn,Fe)_3Si_2$ particles in the annealed material. The initial structure is gradually destroyed during ECAP. The TEM micrographs of both materials after different numbers of ECAP passes are shown in **Fig. 3**. **Fig. 3**a documents severely deformed microstructure with very high density of dislocations after 1 ECAP pass. Only a very limited number of newly formed grains of the sub-micrometer size can be found. No significant differences were observed between both materials. Figures 3b and 3c show the microstructure after 2 ECAP passes. Both the regions with high dislocation density and some newly formed grains, especially in the annealed material, are present. The replacement of severely deformed regions by ultrafine-grained structure proceeds during further ECAP passes (**Figs. 3d and 3e** for 4 ECAP passes). This microstructure development seems to be faster in the annealed material. Finally, the microstructure after 8 ECAP passes consists mostly of ultrafine grains with the grain size deeply below 1 μm (**Fig. 3f**), however, it is not fully recrystallized.

The EBSD orientation maps of the annealed material are shown in **Fig. 4**. As expected, the initial state prior to ECAP (**Fig. 4a**) is characterized by large elongated grains. The first ECAP pass changes this structure only in some regions where the bands of new grains or subgrains are formed (**Fig. 4b**). The orientation map of the same sample taken at higher magnification reveals the presence of numerous low angle boundaries within the elongated grains (**Fig. 4c**). The orientation map of the sample after 4 ECAP passes is completely different. The map taken at lower magnification (**Fig. 4d**) reveals that the microstructure is not fully uniform. Numerous regions of the size approximately 5 μm consisting of subgrains are still present. The map taken at higher magnification (**Fig. 4e**) reveals the mean grain size of approximately 0.6 μm, some low angle boundaries are still present. The structure of the non-annealed material is very similar, only the grain size is slightly lower. Finally, the map of the sample after 8 passes shows a mixture of regions with very small equiaxed grains and regions with elongated grains of the size over 1 μm (**Fig. 4f**). Small angle boundaries were detected especially in the elongated grains dividing them into subgrains.

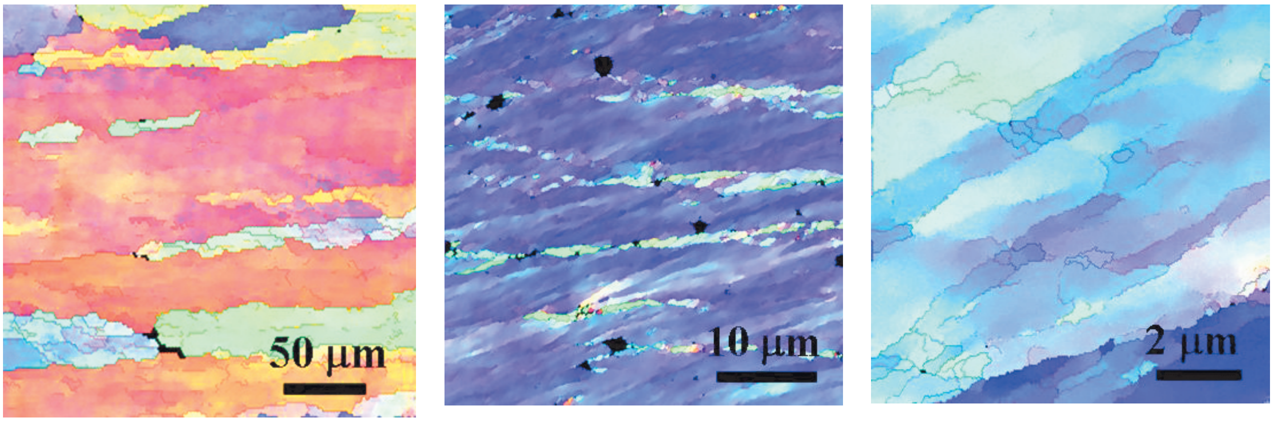


(a) Annealed, 1 ECAP pass (b) Non-annealed, 2 ECAP passes (c) Annealed, 2 ECAP passes

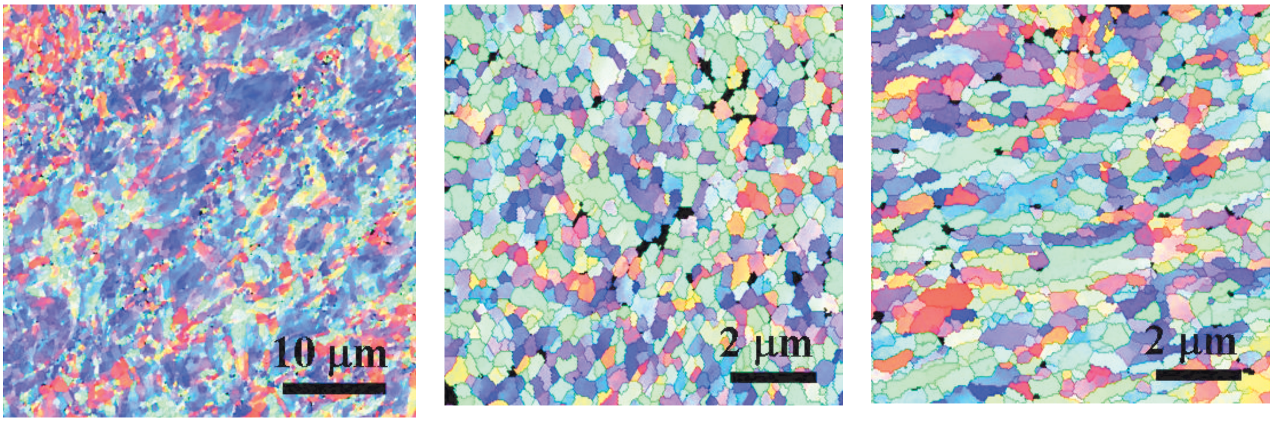


(d) Non-annealed, 4 ECAP passes (e) Annealed, 4 ECAP passes (f) Annealed, 8 ECAP passes

Fig. 3 The microstructure after various number of ECAP passes



(a) Initial state prior to ECAP (b) Annealed, 1 ECAP pass (c) Annealed, 1 ECAP pass



(d) Annealed, 4 ECAP passes (e) Annealed, 4 ECAP passes (f) Annealed, 8 ECAP passes

Fig. 4 The EBSD orientation maps after various number of ECAP passes, small angle boundaries (< 15°) in red, high angle boundaries (> 15°) in black

4. DISCUSSION

Similarly to other Al-based alloys, the equal channel angular pressing is an effective tool for the microstructure refinement also in the TRC Al-Mn-based alloy. The grain size after 4 ECAP passes is close to 0.5 µm, i.e. it is lower in comparison with ECAP Al-Zn-based [11] or Al-Mg-based alloys [13]. The explanation can be found in a lower ECAP temperature in the Al-Mn-based alloy which does not allow grain coarsening at higher number of ECAP passes. The development of an ultrafine grained structure is completed during 4 ECAP passes. Further increase in ECAP deformation leads to elongation of newly formed grains. A similar microstructure development during ECAP was observed in a chill cast Al-Mn-Sc-Zr alloy with a reduced Fe+Si content [14]. Such process does not significantly influence the deformation characteristics and both the strength and ductility are very close in samples after 4 and 8 ECAP passes, respectively. Similarly to [14], the main increase in strength characteristics occurs already during the first ECAP pass. The microstructure analysis documents clearly that not the ultrafine grain size but very high dislocation density it responsible for this effect.

The Al$_3$Zr particles are introduced into Al-based alloys in order to retard recrystallization and grain growth. The annealing used in our research resulted additionally in the precipitation of the α-Al$_{15}$(Mn,Fe)$_3$Si$_2$ particles and thus in the reduction of Mn atoms dissolved in the Al-matrix. Both the microstructure development and changes in strength characteristics during ECAP reveal that especially the dissolved Mn atoms retard the process of recovery and recrystallization. Consequently, the strength characteristics of non-annealed material after ECAP are higher in comparison with the annealed one.

CONCLUSION

The ECAP processing of the TRC Al-Mn-based alloy enhances its strength characteristics and slightly reduces its formability. The main change occurs already during the first pass and is connected with an enormous increase in dislocation density. The ultrafine-grained structure is formed during 4 ECAP passes. However, the structure is not fully recrystallized and contains numerous low angle boundaries. The Mn atoms remaining in the Al solid solution in the non-annealed materials seem to have a greater stabilizing effect than the Al$_3$Zr particles present simultaneously with the α-Al$_{15}$(Mn,Fe)$_3$Si$_2$ particles in the annealed material.

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