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# The Evolution of Microstructure during Annealing of Al–Mg–Sc–Zr Alloy Deformed by Equal Channel Angular Pressing

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The Al–Mg–Sc–Zr alloys exhibit reasonable mechanical properties (e.g. corrosion resistance, weldability, superplasticity) for application in many branches of the industry. They owe their strength mainly to coherent particles of Al<sub>3</sub>Sc or Al<sub>3</sub>(Sc,Zr) phase which form during heat treatment at 300 °C. The influence of such particles on mechanical behaviour and microstructure evolution was studied on a twin-roll cast alloy which was subjected to deformation by equal channel angular pressing. Three types of processing of the material were compared: (1) only deformation, (2) deformation and heat treatment and (3) heat treatment and deformation. The deformation by equal channel angular pressing caused fragmentation of the original grains into micrometric grains and subgrains regardless of the presence of Al<sub>3</sub>(Sc,Zr) particles. The subsequent isochronal annealing with heating rate 50 K/50 min led to both softening and hardening of the material, dislocation recovery and recrystallization of the grain structure, the later was similar in all the studied materials

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## 1. Introduction

Alloys from Al–Mg family find application in many branches of industry due to their perspective properties such as strength, corrosion resistance and possibility of superplastic forming [1, 2]. Several methods how to further improve their properties are under research in recent years. Their strength may be increased either by addition of suitable alloying elements such as scandium and zirconium [3] or by decreasing the grain size by severe plastic deformation [4]. The formation of  $\beta$ -phase Al<sub>3</sub>Mg<sub>2</sub> particles, which promote the exfoliation corrosion [5] may be suppressed by the use of casting procedures alternative to direct-chill (DC) casting and rolling, which produce layered and pancake grain structure [6].

One of the alternative casting techniques is twin-roll casting — TRC (for details see e.g. [7–9]) producing sheets with relatively equiaxed grains. However, the microstructure of the material after TRC is different (e.g. solid solution supersaturation, primary particles distribution, grain structure [10–12]) from the DC cast materials and processing routes established for the DC cast materials have to be modified in order to achieve comparable properties also in the TRC material.

Addition of scandium and/or zirconium to aluminium alloys enhances the high temperature stability of the ma-

terial [2], when dense distribution of metastable coherent Al<sub>3</sub>(Sc,Zr) with the size around 10 nm is present in the material. Such particles exert Zener drag on moving dislocations and grain boundaries and, thus, enhance the recrystallization resistance and strength of the material [13]. They form during annealing at temperature range 300–450 °C; nevertheless, at higher temperatures they may transform to stable phase which loses the strengthening effect [5].

Equal channel angular pressing (ECAP) is one of the most widely-known procedure for fabrication of ultra-fine grained materials with high strength [14] due to intense plastic shearing. During the ECAP processing, low angle grain boundaries and/or shear bands are generated in the original grain interiors resulting in grain subdivision and they are progressively transformed into high angle grain boundaries with further deformation, leading to a development of new (sub)microcrystalline grain structures at large strains [15].

The present work studies the influence of precipitation annealing, applied in order to provide a dense distribution of Al<sub>3</sub>(Sc,Zr) particles in the material, on the mechanical behaviour and microstructure of the material fabricated by twin-roll casting and processed by equal channel angular pressing.

## 2. Experimental

The studied material was prepared in laboratory conditions by twin-roll casting to thickness 5 mm. The vertical twin-roll caster with casting rate 2.75 m/min was used, the melt was preheated to 655 °C. The com-

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position of the material is as follows: 3.24 wt.% Mg, 0.19 wt.% Sc, 0.14 wt.% Zr, 0.16 wt.% Mn, 0.11 wt.% Si and 0.21 wt.% Fe.

Subsequently, the TRC material was subjected to severe plastic deformation by equal channel angular pressing at 250 °C, totally 4 passes by route  $B_C$  [14]. The sample dimensions were  $5 \times 5 \times 50 \text{ mm}^3$ .

In order to precipitate a dense distribution of fine coherent  $\text{Al}_3(\text{Sc,Zr})$  particles, the material was annealed at 300 °C for 8 hours — see [16] for details of the precipitates formation.

The annealing was applied either before or after the ECAP processing in order to compare the influence of the heat treatment at 300 °C and the resulting presence of  $\text{Al}_3(\text{Sc,Zr})$  on microstructure and mechanical properties evolution. Thus, three types of materials were studied — material which was first deformed and then annealed, materials first annealed and then deformed and material only deformed without annealing at 300 °C. After the deformation all three materials were isochronally heated in an air furnace with the step 50 K/50 min up to 600 °C in order to monitor the stability of the material.

The evolution of microstructure and mechanical properties were studied in scanning electron microscope

(SEM) FEI Quanta 200 FX FEG equipped with electron back-scatter diffraction (EBSD) detector, transmission electron microscope (TEM) JEOL 2000FX working at 200 kV and by measurement of Vickers Microhardness HV0.1 with a load of 100 g at QNess Q10A+ device.

### 3. Results and discussion

The twin-roll cast material contains equiaxed grains with average grain size  $\sim 50 \mu\text{m}$  and low dislocation density [12]. Two different treatments were applied to the TRC material — precipitation annealing and deformation by ECAP.

Annealing of the twin-roll cast material for 8 hours at 300 °C leads to precipitation of dense distribution of  $\text{Al}_3(\text{Sc,Zr})$  [17]. The optimal conditions for their formation were found in our previous work [16] — the highest increase in the microhardness was observed after annealing at 300 °C; furthermore, the precipitates diameter was smaller than after annealing at 350 or 400 °C which ensures higher pinning force acting against recrystallization. In the twin-roll cast material the Vickers microhardness increases from initial  $\sim 75 \text{ HV}$  for the as-cast material to  $\sim 100 \text{ HV}$  for the annealed one [16].

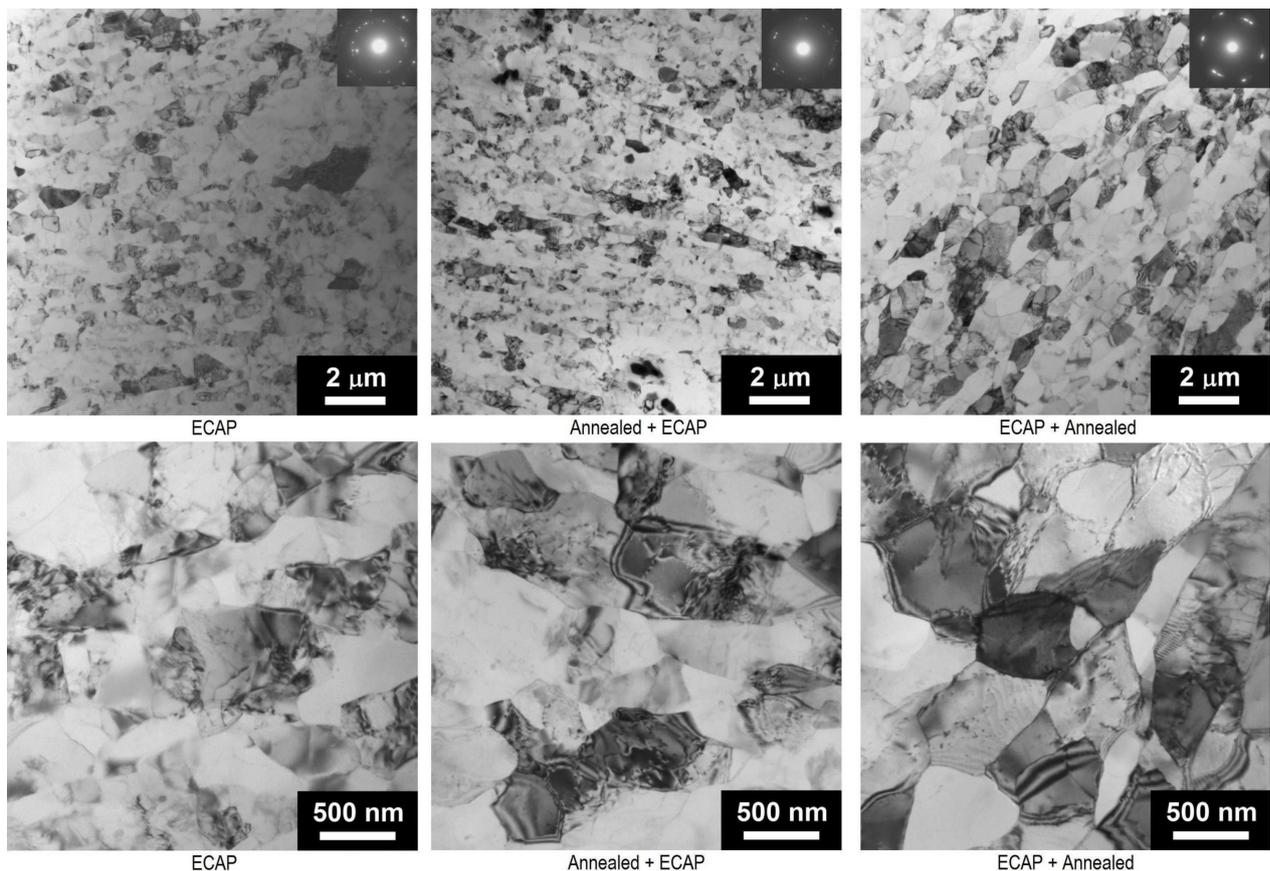


Fig. 1. The TEM image of the microstructure of the twin-roll cast material after deformation by ECAP (left), material which was annealed after the twin-roll casting at 300 °C for 8 hours and then deformed by ECAP (middle) and the TRC material which was first deformed by ECAP and subsequently annealed 300 °C/8 h (right).

Deformation by equal channel angular pressing of the twin-roll cast material causes fragmentation of the original grains to micrometric range — Fig. 1. However, the resulting grain structure is not uniform, the micrometric grains with high angle grain boundaries are often surrounded by elongated subgrains. This is in accordance with observations of Tanski and Sitdikov [15, 18, 19] who reported bimodal structure with shear bands in different Al–Mg alloys after various number of ECAP passes. The grain elongation is partially apparent in Fig. 1, more details can be found in [12, 20].

The precipitation annealing, applied either before or after ECAP, has no significant influence on the grain microstructure of the deformed material — see Fig. 1 for comparison. However, it slightly modifies the microhardness of the material (Fig. 2). Annealing after ECAP at 300 °C for 8 hours causes a slight decrease of the microhardness which may be connected with partial dislocation recovery; however, more pronounced microhardness decrease is most probably compensated by the precipitation of  $\text{Al}_3(\text{Sc,Zr})$  particles.

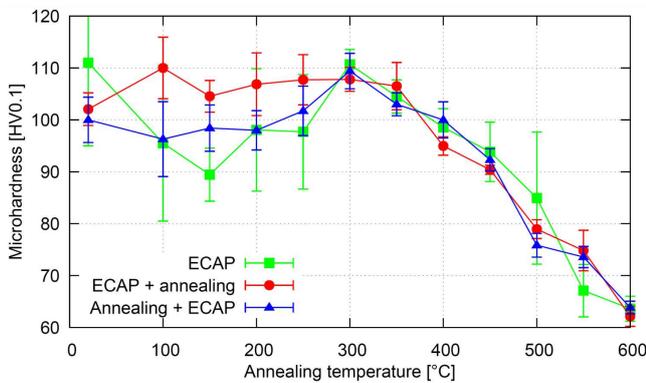


Fig. 2. Microhardness evolution during isochronal annealing 50 K/50 min from room temperature to 600 °C.

Concerning the material which was annealed before ECAP processing, its microhardness is also slightly lower than in the case of the material which was subjected only to ECAP. This is in accordance with work on other type of aluminium alloys [21] which showed that pre-ECAP annealing led to lower dislocation density after ECAP and lower microhardness. Nevertheless, it is necessary to mention that these differences in microhardness are subtle within the experimental scatter.

The difference caused by the pre-annealing at 300 °C becomes significant during isochronal annealing. In the material pre-annealed at 300 °C after ECAP, the microhardness remains almost constant, as both the precipitation and recovery took place during the post-ECAP annealing and the repeated application of heat treatment has no significant influence on the material.

On the other hand, in the material pre-annealed at 300 °C before ECAP deformation the microhardness slightly increases at temperatures above 200 °C, reaching the peak at 300 °C. It has been reported for different ma-

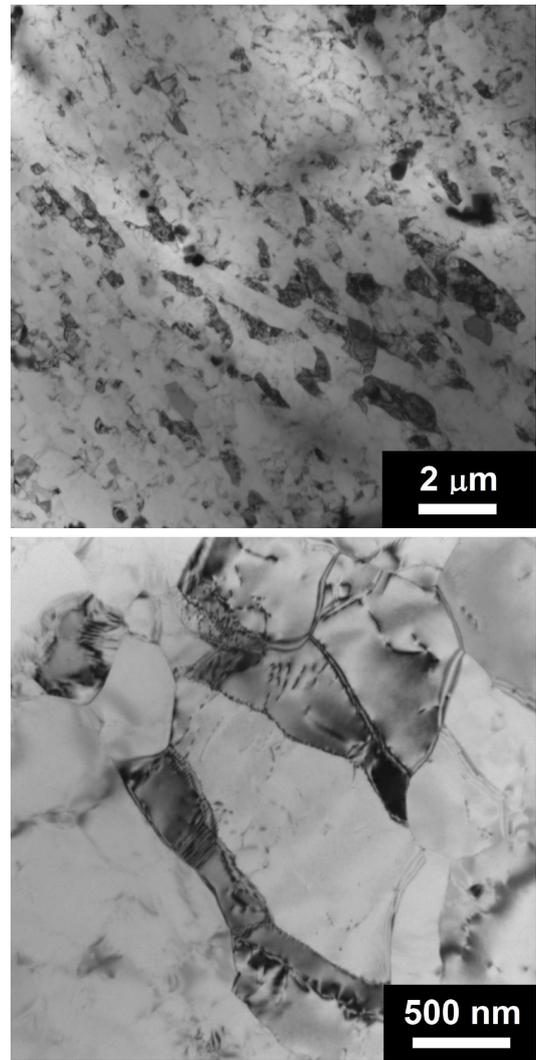


Fig. 3. The TEM micrograph of the twin-roll cast material after deformation by ECAP and isochronal annealing to 150 °C.

terials [22–24] that severe plastic deformation may lead to partial dissolution of metastable strengthening particles. If some  $\text{Al}_3(\text{Sc,Zr})$  precipitates dissolved during the ECAP processing, their reprecipitation at temperatures around 300 °C may have led to further strengthening of the material and increase of the microhardness, masking eventual softening caused by a recovery of the dislocation substructure.

The material which was processed only by ECAP exhibits the most pronounced changes of the microhardness below 300 °C. Firstly, the microhardness drops to a local minimum at 150 °C. Well recovered substructure after annealing to 150 °C is depicted in Fig. 3. Most probably recovery of the residual dislocation substructure occurs in this temperature range. On the contrary, microhardness increase is observed at higher annealing temperatures up to 300 °C. This pronounced hardening can be attributed to precipitation of coherent  $\text{Al}_3(\text{Sc,Zr})$  particles, which

has stronger influence on the microhardness than the recovery. Similar behaviour was observed by Vlach [25] in conventional Al–Sc–Zr alloy after cold rolling.

Further annealing up to 600 °C brings no general difference between the materials; the microhardness gradually decreases, reaching values around 65 HV at 600 °C. The microhardness evolution above 300 °C is similar in both pre-annealed materials, suggesting that the distribution of Al<sub>3</sub>(Sc,Zr) particles is comparable in these materials. On the other hand, the material without pre-annealing shows faster drop of microhardness already at 550 °C followed by a subtle decrease at 600 °C indicating considerable degree of recrystallization at 550 °C.

The comparison of the grain distribution at 550 and 600 °C (see Fig. 4) reveals extensive recrystallization between these two temperatures and full replacement of subgrains by large grains with the size ranging from 10 to 200 μm in all materials. The final grain size is significantly larger in the material which was not pre-annealed, confirming partial recrystallization at 550 °C followed by a grain coarsening at 600 °C. The origin of this effect comes from a possible different distribution of Al<sub>3</sub>(Sc,Zr) particles, which were not formed during annealing at 300 °C for 8 hours. Their pinning effect thus could be limited resulting in more progressive recrystallization and grain coarsening.

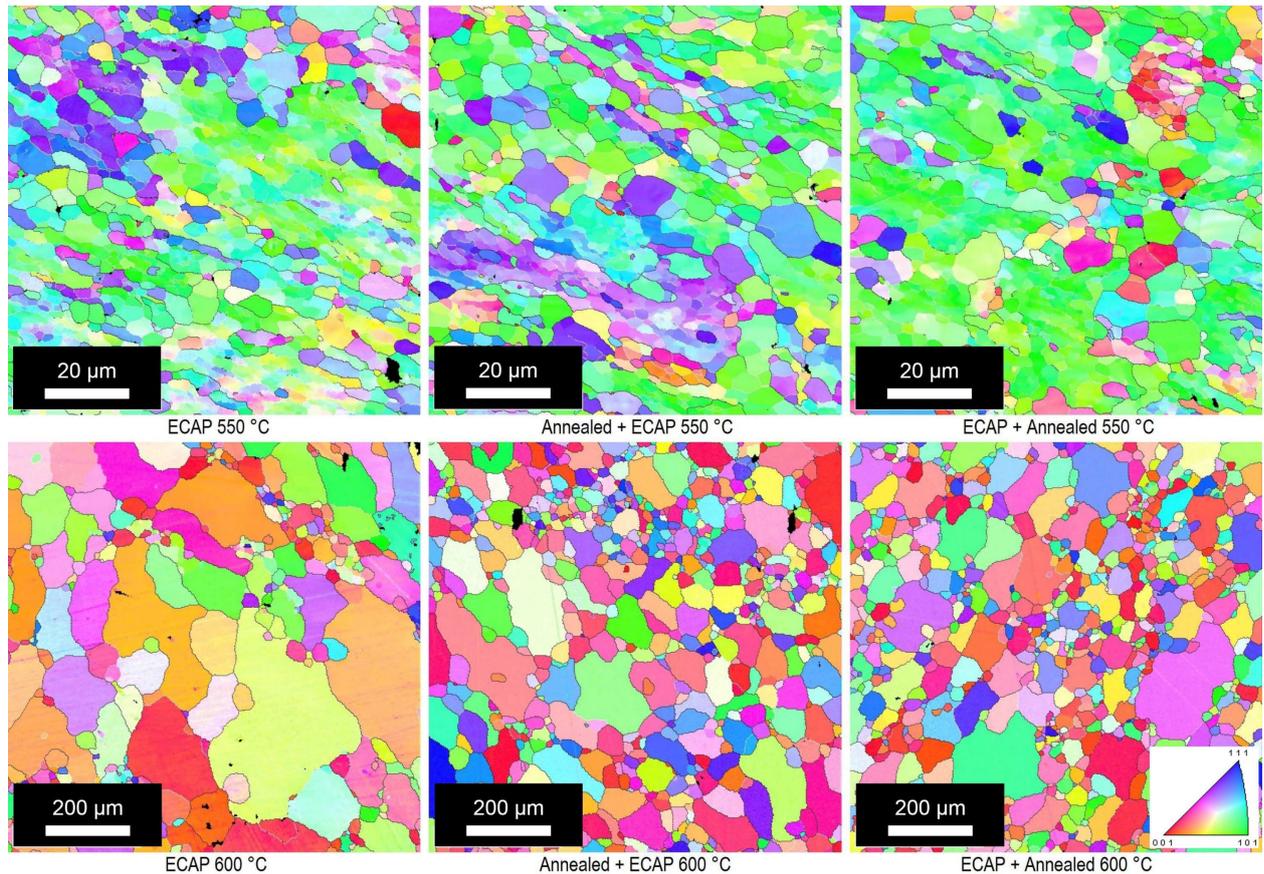


Fig. 4. The EBSD orientation map of the microstructure of the materials annealed to 550 °C (top) and 600 °C (bottom). The twin-roll cast material after deformation by ECAP (left), material which was annealed after the twin-roll casting at 300 °C for 8 hours and then deformed by ECAP (middle) and the TRC material which was first deformed by ECAP and subsequently annealed 300 °C/8 h (right).

#### 4. Conclusion

The role of annealing at 300 °C for 8 hours, which induces high density of Al<sub>3</sub>(Sc,Zr) precipitates into the aluminium matrix, on the deformation by equal channel angular pressing was studied. The annealing did not influence the grain structure after the deformation. The non-annealed material, which had the highest microhardness after ECAP deformation, exhibited the largest changes of microhardness during post-ECAP isochronal annealing up to 300 °C — firstly a drop connected with dislocation recovery was observed, secondly hardening of the

material caused by precipitation of Al<sub>3</sub>(Sc,Zr) followed. At higher temperatures the behaviour of all materials was comparable, the microhardness gradually decreased and the recrystallization took place between 550 and 600 °C. In the specimen without heat pretreatment significant grain growth was observed at 600 °C.

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