Al-Mg-Sc ALLOYS PROCESSED BY EQUAL-CHANNEL ANGULAR PRESSING

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Abstract

Twin-roll cast Al-Mg-Sc alloy was processed by equal-channel angular pressing in order to produce ultrafine-grained structure. The microstructure was studied by the means of scanning electron microscopy, electron back-scatter diffraction and transmission electron microscopy. During in-situ heating partial recrystallization occurred in the bulk of the material. Near the edge of the sample observed in transmission electron microscope the deformed microstructure was retained, as in the 2D foil processes of recrystallization proceed slower and at higher temperatures compared to the 3D materials.

Keywords: Aluminum alloys, equal-channel angular pressing, in-situ annealing, thermal stability

1. INTRODUCTION

Aluminum alloys belong to the most widely used metallic materials in many branches of industry. Al-Mg type of alloys finds its application mostly in the aerospace and ship-building industries because of their adequate corrosion resistance, good weldability [1-2] and possibility of superplastic forming [3-4].

One of the drawbacks of such materials is the susceptibility to localized attack by intergranular corrosion and to exfoliation in temperature range 50-180 °C [5-7], which occur mainly in materials with flat grains; such are materials prepared by direct-chill casting (DC) which are processed to the final thickness by hot- or cold-rolling which forms a pancake-like structure of the grains.

The DC technique can be substituted by twin-roll casting (TRC) which produces sheets with thickness in order of mm. During the TRC molten material is directed through a nozzle between two water-cooled rolls, where it solidifies. The microstructure with nearly equiaxed grains is formed when compared to the DC material rolled to the respective thickness [8-11].

In TRC Al-Mg-Sc-Zr alloys the final strengthening can be achieved by aging at temperatures around 350 °C and work hardening by deformation methods [12-13], which do not change the thickness of the strip and also do not generate pancake grain structure - methods of severe plastic deformation (SPD). The combination of TRC with artificial aging and SPD prior or after aging can thus generate Al-Mg based alloy with adequate mechanical properties and improved resistance to intergranular corrosion and exfoliation [7].

One of the most accessible SPD methods is equal-channel angular pressing (ECAP) [14]. During ECAP the billet of material is pressed through a die which consists of two channels of equal cross-section intersecting at an angle Φ, usually 90° [15]. 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. As a result microstructure with submicrometric grains is formed.

The twin-roll cast material consists of fairly equiaxed grains with average grain size in order of 100 µm subdivided into a few subgrains [16].

In the recent paper we focused on microstructure evolution during in-situ heating of TRC Al-Mg-Si alloy subjected to SPD by equal-channel angular pressing.

2. EXPERIMENTAL

Twin-roll cast aluminum alloy with thickness 5 mm was prepared at University Paderborn with composition 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. This alloy was subsequently subjected to severe plastic deformation by equal-channel angular pressing at 250 °C at Charles University with channel dimensions 5 x 5 mm^2, pressing speed 10 mm/min and 4 passes by route B_c [14] in total. To evaluate the evolution of the microstructure during exposure to elevated temperatures, the sample was annealed in-situ in transmission electron microscope JEOL 2000FX working at 200 kV and equipped by a heating holder. The grain structure evolution was observed by scanning electron microscope FEI Quanta 200 equipped with detector for electron back-scatter diffraction (EBSD). The microhardnees was measured on Qness A10+ device with load 100 g for 10 s.

3. RESULTS AND DISCUSSION

3.1. Ecap

The processing by 4 ECAP passes leads to a significant change of the microstructure - **Figure 1**. The EBSD orientation map shows that the microstructure is bimodal - the original grains of the twin-roll cast material are fragmented into structure which consists of a large number of small grains with average size around 1 µm which are embedded into one larger grain with only moderately changing orientation and few subgrain boundaries. Due to the size of the sample before ECAP processing and, furthermore, due to the fact that after each ECAP pass a small piece of the sample may break away and the head and tail of the sample possess a bit different microstructure than the bulk, the processing by more ECAP passes in order to ensure fully ultra-fine grained structure was not auspicious.

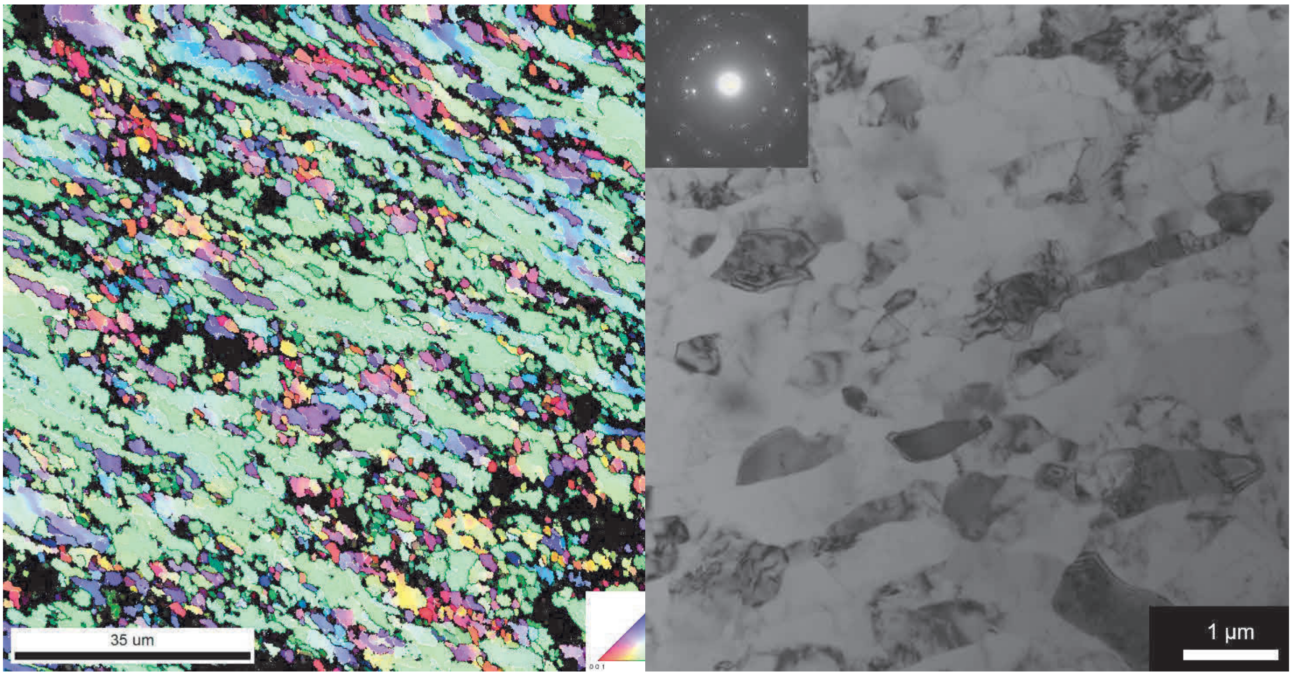


Figure 1 The microstructure of the Al-Mg-Sc alloy after 4 passes of ECAP. Some of the grains are elongated in the rolling direction, some new equiaxed micrometric grains with high-angle grain boundaries are formed

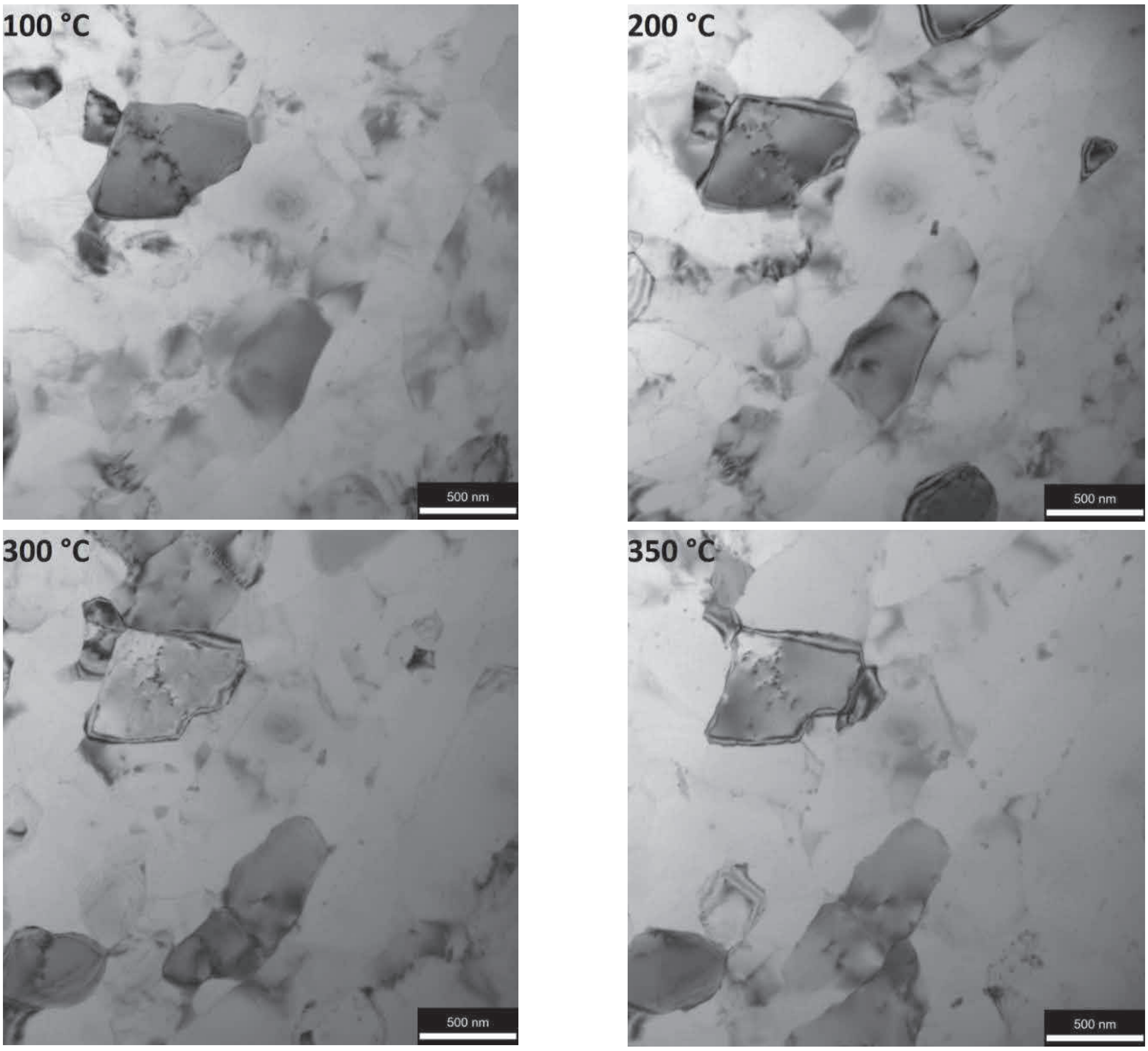
The TEM observations (**Figure 1**) confirmed the presence of micrometric grains surrounded by high angle grain boundaries in the matrix. The elongation of some grains is apparent. The selected area diffraction pattern shows that the structure is formed by individual grains with different orientation rather than by fluent change of orientation inside the grains.

This is in contrast to the microstructure created by cold-rolling performed on the same material [16], where only the shape of the grains was modified, their average size was retained and they were subdivided into subgrains with fluently changing misorientation. The dislocation density inside the grains was fairly higher.

The fragmentation of the original grains into micrometric constituents is in accordance with the increase of Vickers microhardness from original value of 78 HV for the twin-roll cast material to 111 HV after ECAP processing. The fact that the lower is the grain size of the material the higher is its strength was first described by Hall and Petch and the derived Hall-Petch relation has been proven for many materials since then - see e.g. [17].

3.2. In-situ TEM

In order to monitor the microstructure changes of the deformed material, in-situ annealing with heating rate 50 °C/10 min in TEM up to 550 °C was performed. The TEM micrographs from the same area for different temperatures are depicted on **Figure 2**.



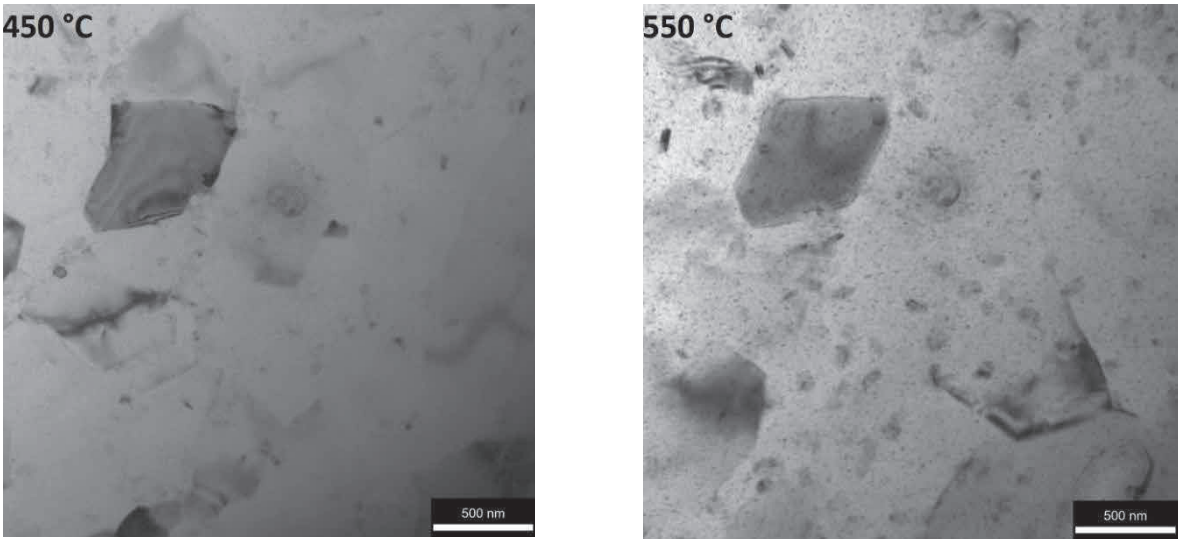


Figure 2 In-situ annealing in TEM 50 °C/10 min to 550 °C. No significant changes in the grain structure were observed.

During the heating the grain size distribution in the observed area did not evince any significant changes. The shape of the grains after reaching 550 °C was more round, the grain boundaries were more straight and the edges of the grains got more round - small particles which pinned the grain boundaries dissolved during annealing to this temperature; thus, it was feasible for the grain boundaries to change their shape to the one which is more favourable from the point of view of equilibrium energy. Most of the dislocations located inside the micrometric grains disappeared during annealing around 450 °C.

At temperature around 300 °C particles of α-AlMnFeSi phase [13,18] started to precipitate. During further annealing their diameter slightly increased and the smaller ones dissolved back to the solid solution around 450 °C.

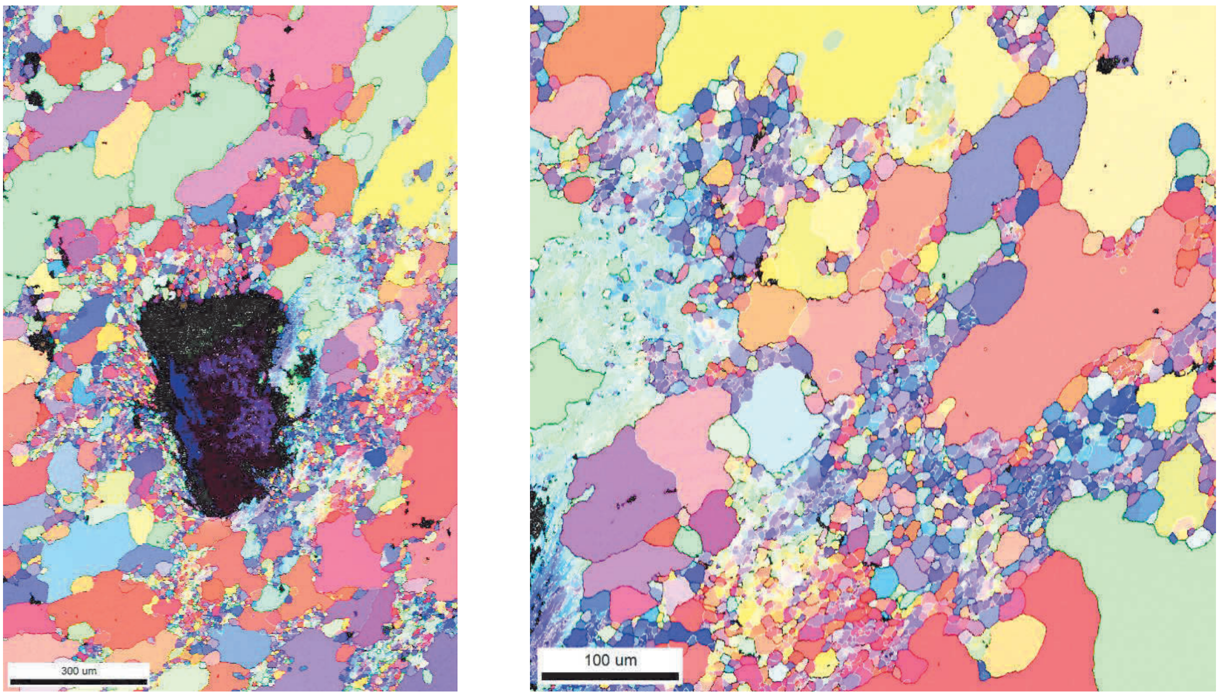


Figure 3 EBSD image of the sample after in-situ annealing to 550 °C. The deformed microstructure is retained mainly near the edge of the TEM foil

The grain structure after in-situ annealing to 550 °C is visualized by EBSD on **Figure 3**. The inhomogeneity of the grain size distribution is apparent - near the edge of the TEM foil small grains with size around 10 μm prevail, some parts of the material retained the deformed microstructure. Regions further from the edge are dominated by big recrystallized grains with size over 100 μm supplemented by clusters of smaller grains.

It was proven by our previous research [19] that the 2D nature of the TEM foil influences the processes of recovery and grain recrystallization in that way that the recrystallization proceeds in the thin foil at higher temperatures and to lower extent as compared to the bulk material.

4. CONCLUSION

The processing by equal-channel angular pressing leads to modification of microstructure and creation of micrometric grains embedded in the original matrix. In-situ annealing in transmission electron microscope leads to dislocation recovery and precipitation. However, the recrystallization during the in-situ annealing proceeds inhomogeneously - the areas near the edge of the foil resist recrystallization more considerably than the bulk of the material

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