

## ROLLED Al-Mg-Sc ALLOY STUDIED BY TRANSMISSION ELECTRON MICROSCOPY

KŘIVSKÁ Barbora<sup>1</sup>, ŠLAPÁKOVÁ Michaela<sup>1</sup>, BAJER Jan<sup>1</sup>, GRYDIN Olexandr<sup>2</sup>,  
CIESLAR Miroslav<sup>1</sup>

<sup>1</sup>Charles University, Faculty of Mathematics and Physics, Prague, Czech Republic, EU  
[slapakova@karlov.mff.cuni.cz](mailto:slapakova@karlov.mff.cuni.cz)

<sup>2</sup>University Paderborn, Faculty of Mechanical Engineering, Lehrstuhl für Werkstoffkunde Microscopy,  
Paderborn, Germany, EU

### Abstract

Twin-roll cast Al-Mg-Sc alloy was submitted to deformation by cold-rolling. The influence of heat treatment 300 °C / 8 h, which was applied either before or after the deformation, on the deformation microstructure was studied by the means of scanning and transmission electron microscopy and by microhardness measurements. The cold-rolling leads to increase of dislocation density and fragmentation of the grains into subgrains. Annealing both before and after deformation causes precipitation hardening of the material. The in-situ annealing in transmission electron microscope leads to rearrangement of dislocations and partial recrystallization of the microstructure.

**Keywords:** Al-Mg alloys, Al<sub>3</sub>(Sc,Zr) precipitates, electron microscopy, heat treatment

### 1. INTRODUCTION

Al-Mg based alloys are widely used in ship-building and aerospace industries because of their good corrosion resistance, weldability [1, 2] and possibility of superplastic forming [3, 4]. However, one of the disadvantages of Al-Mg alloys is their lower strengths for structural applications.

There are two common routes through which mechanical properties can be improved. The first one is addition of elements which form strengthening particles through heat treatment. Additions of scandium and zirconium to Al-Mg alloys offer attractive combination of properties. The precipitation of metastable Al<sub>3</sub>(Sc,Zr) particles upon aging delivers contribution from precipitation hardening increasing thus the strength levels of the alloy [5-7]. The precipitates form at temperatures between 300 and 450 °C and serve as highly effective obstacles for dislocation and grain boundary motion increasing thus recovery and recrystallization resistance of the material [8, 9].

Another way of improving strengths is by a grain refinement and work hardening, which can enhance the strengths of the material through the grain boundary strengthening or dislocation strengthening [10]. Nevertheless, this positive effect is immediately lost during recrystallization. The use of twin-roll casting (TRC) method for producing the material is an alternative method to direct-chill casting which casts the strips to the final thickness and thus leads to energy and time savings and, moreover, the recrystallization annealing is no more required during the strips production [11-13].

In the present work we focused on the investigation of the properties of twin-roll cast Al-Mg alloy with addition of small amount of Sc and Zr and the role of this alloying elements on microstructure evolution during deformation by cold-rolling.

### 2. EXPERIMENTAL

Aluminum alloy 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 was twin-roll cast in laboratory conditions to thickness 5 mm. Subsequently it was cold-rolled to

thickness 3 mm. Annealing in air furnace at 300 °C for 8 hours was applied either before or after the cold-rolling. The mechanical properties of the material were tested by Vickers microhardness (HV) measurement with a load of 100 g at QNess 10A with at least 10 measured indents for each value. The microstructure was observed by scanning electron microscope (SEM) FEI Quanta 200 equipped with electron back-scatter diffraction (EBSD) detector and by transmission electron microscopes (TEM) JEOL 2000FX and 2200FS. Specimens for electron microscopy were electropolished in 30% HNO<sub>3</sub> solution in methanol at -15 °C.

### 3. RESULTS AND DISCUSSION

Twin-roll cast material contains nearly equiaxed grains with size in order of 100 μm - **Figure 1a**. The grains are subdivided into subgrains with low average misorientation. For more details about the as-cast material see [14].

The material was subjected to annealing at 300 °C for 8 hours in order to support precipitation of Al<sub>3</sub>(Sc,Zr) phase. It has been shown in previous work that this annealing temperature is sufficient for formation of high density of these particles in the given material [15].

In order to evaluate the role of the annealing at 300 °C three states of the material were considered - firstly material only cold-rolled, secondly material which was annealed after the cold-rolling and finally material which was annealed before the cold-rolling.

#### 3.1. Rolling

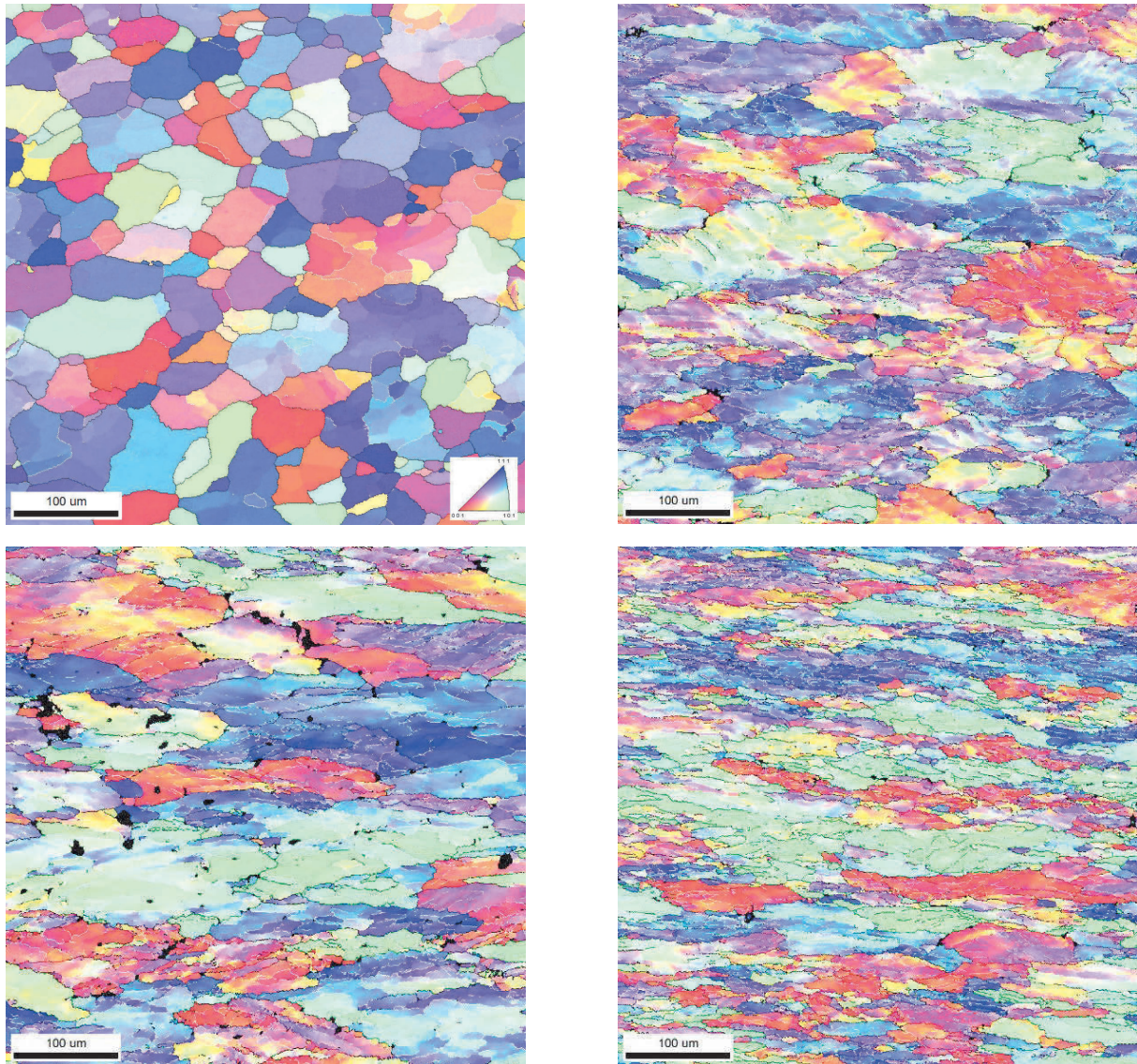
After cold-rolling both the microstructure and mechanical properties of the materials evince a change in all the studied materials.

Concerning the results from electron back-scatter diffraction the average grain size was not influenced by the deformation, only the shape of the grains was modified - they become slightly elongated in the rolling direction (**Figure 1b, 1c and 1d**). However, in the interior of the grains the misorientation increased significantly and the grains are now fragmented into high number of subgrains.

The microstructure images from the transmission electron microscope give evidence of substructure with high density of dislocations - **Figure 2a, 2b and 2c**. Elongation of the subgrains is also apparent. No significant difference in the dislocation substructure was caused by the annealing undertaken before or after the cold-rolling. The selected area electron diffraction images (insets in **Figure 2**) prove that the depicted areas contain only one grain (divided to subgrains) with fluently changing orientation. This is in contrast to the microstructure created by equal-channel angular pressing performed on the same material [16], where a lot of distinct grains formed on comparable area.

The microhardness of the as-cast material was 78 HV and increased after the rolling. The final microhardness was determined by the presence of annealing - in the non-annealed material the microhardness reached a value of 90 HV. In the material annealed before the cold-rolling the microhardness reached 105 HV and in the case where the thermal treatment was applied after the deformation, the value was 102 HV. In all the materials the HV values fluctuated and the standard deviation is around 5 HV.

The annealing for 8 hours at 300 °C leads to precipitation of Al<sub>3</sub>(Sc,Zr) and increase of microhardness to 100 HV [15]. This means that the main increase of the microhardness in both annealed materials was caused mainly by precipitation hardening, only slight increase is attributed to deformation hardening by dislocations. No strengthening particles are present in non-annealed material thus the microhardness augmentation is caused by the increased dislocation density.

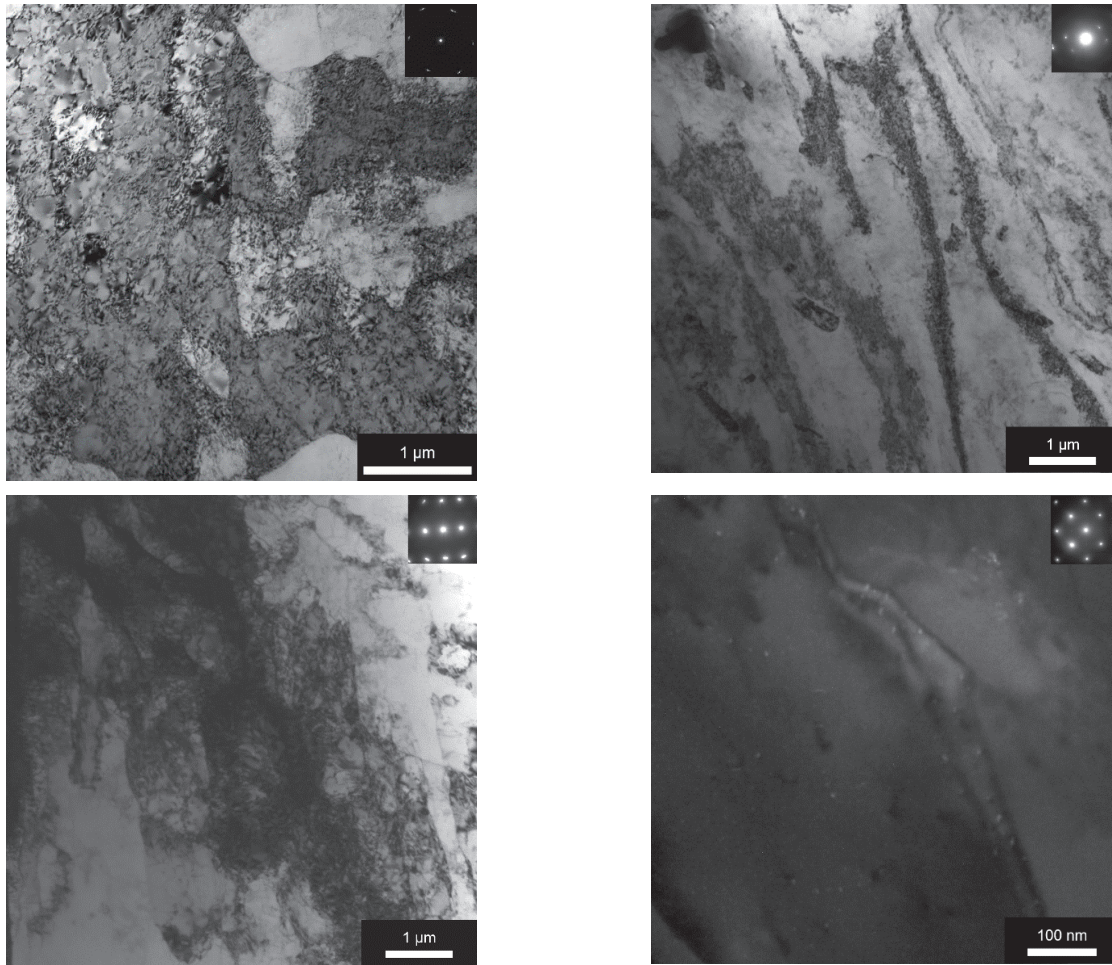


**Figure 1** Electron back-scatter diffraction maps - a) twin-roll cast material, b) cold-rolled material, c) material annealed at 300 °C for 8 hours and subsequently cold-rolled and d) material cold-rolled and annealed 300 °C/8 h after the deformation. Cold-rolling lead to fragmentation of the grains into subgrains

### 3.2. $\text{Al}_3(\text{Sc,Zr})$

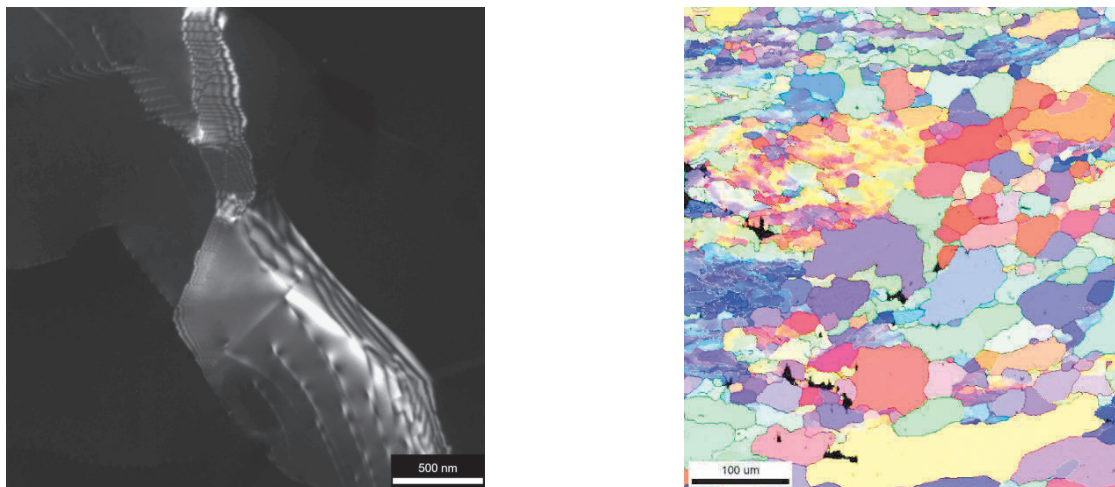
It was reported in literature that cold-rolling has an influence on precipitation of the  $\text{Al}_3(\text{Sc,Zr})$  particles. Both the cases were described - either the precipitation is facilitated [7] or hindered [17] by the cold-rolling. The main difference seems to lie in the composition of the material and the casting technique - twin-roll casting compared to direct chill casting.

In the material studied in this paper the  $\text{Al}_3(\text{Sc,Zr})$  formed in the cold-rolled material after annealing at 300 °C for 8 hours - this is documented in **Figure 2d** taken in  $[001]_{\text{Al}}$  zone axis. Additional diffraction spots from  $\text{L}_{12}$  phase are apparent in the selected area diffraction image in the inset. The density of  $\text{Al}_3(\text{Sc,Zr})$  particles is comparable with the twin-roll cast material annealed under the same conditions [15].



**Figure 2** Transmission electron micrographs of the cold-rolled materials with high dislocation density. a) cold-rolled, b) annealed and cold-rolled, c) and d) cold-rolled and annealed. d) Detail of  $\text{Al}_3(\text{Sc,Zr})$  precipitates in  $[001]_{\text{Al}}$  zone axis. Corresponding selected area diffraction patterns in inset.

### 3.3. Annealing



**Figure 3** Microstructure after in-situ annealing in TEM up to 550 °C  
a) Dislocations forming low-angle grain boundaries. b) EBSD grain structure with partially recrystallized matrix and partially retained deformed grains near the foil edge (located on the left).

In order to evaluate the microstructure evolution during heat treatment, the cold-rolled material was submitted to in-situ annealing in transmission electron microscope. The temperature during the observation was increased by 50 °C every 10 minutes. During the annealing precipitates of  $\alpha$ -Al(Fe,Mn)Si phase formed at temperatures around 300 °C, which is in accordance with post-mortem observations [18]. The dislocation substructure recovered and low-angle grain boundaries formed - **Figure 3a**.

After reaching the temperature of 550 °C the grain structure was inhomogeneous - part of the microstructure retained in the deformed state with high number of subgrains, in other parts of the matrix new fully recrystallized grains formed - see **Figure 3b**. The deformed microstructure prevailed near the hole in the TEM specimen - similar behavior was described in another type of Aluminum alloy during in-situ annealing because near the hole the two-dimensional nature of the TEM foil prevails and recrystallization and dislocation recovery proceed there slower than in the bulk material [19].

#### 4. CONCLUSION

The cold-rolling of Al-Mg-Sc alloy results in elongation of the grains and increase in the dislocation density. The microhardness of cold-rolled material is influenced by annealing at 300 °C for 8 hours which is conducted in order to precipitate coherent Al<sub>3</sub>(Sc,Zr) particles. During in-situ annealing in TEM the dislocations recover and form low-angle grain boundaries near the edge of the TEM foil, in the bulk of the material new recrystallized grains appear.

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