

## THE INFLUENCE OF Al-Mg-Sc-Zr STRIPS PREPARATION ON MICROHARDNESS DURING ANNEALING AT ELEVATED TEMPERATURES

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### Abstract

Evolution of microhardness during isochronal annealing up to 600 °C was studied in strips processed from Al-Mg-Sc-Zr alloy. Specimens for all measurements were prepared from conventionally mould cast and hot rolled strips (CC) or from strips after twin-roll casting (TRC). Selected TRC specimens were further either hot rolled or constrained groove pressed. The role of strips preparation methods on microhardness and annealing response was established. It was shown that the formation of coherent Sc and Zr-rich particles at temperatures close to 300 °C results in a significant increase of microhardness in specimens that have not been exposed to elevated temperatures during the preparation of strips. The highest microhardness was observed in CC materials as a result of a combined effect of fine grains and strengthening particles. Above 350 °C a degradation of mechanical properties occurs in two stages in all materials independently on their previous history. The observed drop of microhardness was caused by the overaging of strengthening particles and withdrawal of the strengthening effect of the deformation structure.

**Keywords:** Aluminum alloys, metal sheets, preparation, severe plastic deformation, annealing

### 1. INTRODUCTION

Al-Mg-based alloys with an addition of scandium and zirconium are widely used in aerospace industry, especially thin plates for cockpits, the skins of aircraft, fuel tanks, etc. [1, 2]. The most common way of preparation of metal sheets is direct chill (DC) casting and subsequent hot and cold-rolling to reach the required thickness. However, increased demands on the material properties cannot be fulfilled by conventional methods (pronounced grain elongation and a pancake structure responsible for the exfoliation corrosion that proceeds along grain boundaries [3]). Therefore, new approaches of aluminium sheets production are in the center of an intensive research now.

Twin-roll casting (TRC) [4, 5] of a metal strip is a promising option which does not produce an undesirable fibre structure. In addition, TRC method produce directly the metal strips of requested thickness in one operation and following homogenization and rolling are not always necessary as in the case of DC-cast materials. Moreover, several methods of severe plastic deformation (SPD) can be used for further grain refinement and strengthening [6, 7] of the final metal strip.

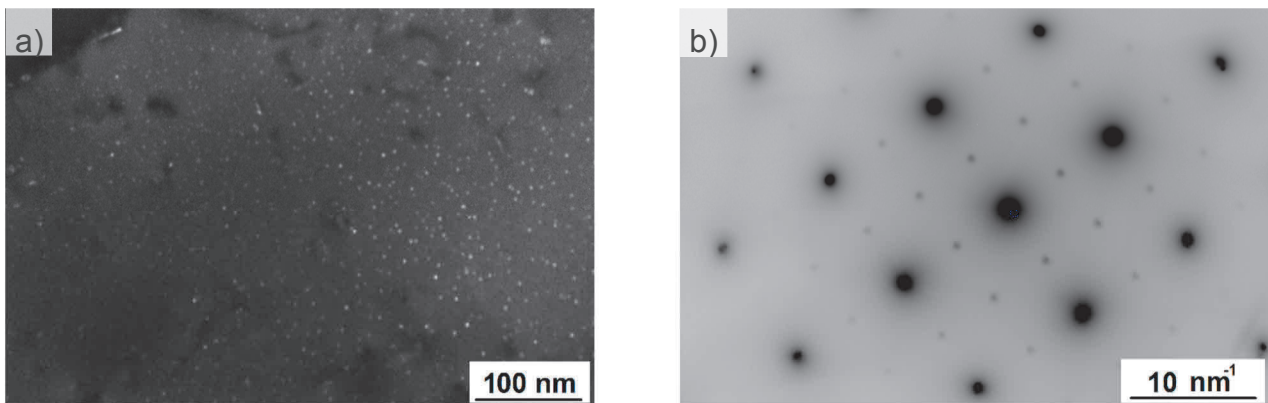
The aim of the present research is to compare the microhardness evolution during thermal exposure of strips prepared by a conventional way and TRC combined with a laboratory rolling or constrained groove pressing (CGP) [8, 9].

### 2. EXPERIMENTAL

Strips from Al-alloy with a composition shown in **Table 1** were:

- a) mould cast (specimens labelled as CC). Their original thickness after casting was 30 mm. The strip was cold rolled to the thickness 10 mm and then hot rolled at 300 °C to further reduce the thickness down to 5 mm.
- b) twin roll cast to final thickness 5 mm. Subsequently, the material was either hot rolled (300 °C) in a laboratory mill to a thickness of 3 mm or subjected to one complete CGP cycle at 250 °C.

Selected samples were annealed at 300 °C for 8 hours before the annealing experiment in order to form coherent particles  $Al_3(Sc,Zr)$  which harden the material and pin grain boundaries at higher temperatures [10],[11],[12]. These precipitates are depicted in **Figure 1**.



**Figure 1** The distribution of the coherent  $Al_3(Sc,Zr)$  particles in TRC sample annealed at 300 °C for 8 hours. (a) Dark field image near [100] Al zone axis, (b) the corresponding diffraction pattern

**Table 1** Chemical composition of Al-Mg-Sc-Zr alloy (wt.%)

Al	Mg	Zr	Sc	Mn	Si	Cu	Fe	Zn
Balance	3.24	0.14	0.19	0.16	0.11	0.024	0.21	<0.002

## 2.1. Experimental methods

Samples were annealed in an air furnace in an isochronal step-by-step heating scheme up to 600 °C with step of 50 °C/50 min. After each annealing and water quenching the microhardness was measured. Every measurement was carried out on a different sample, therefore the scatter of measured values is higher due to local inhomogeneities along the cast strip.

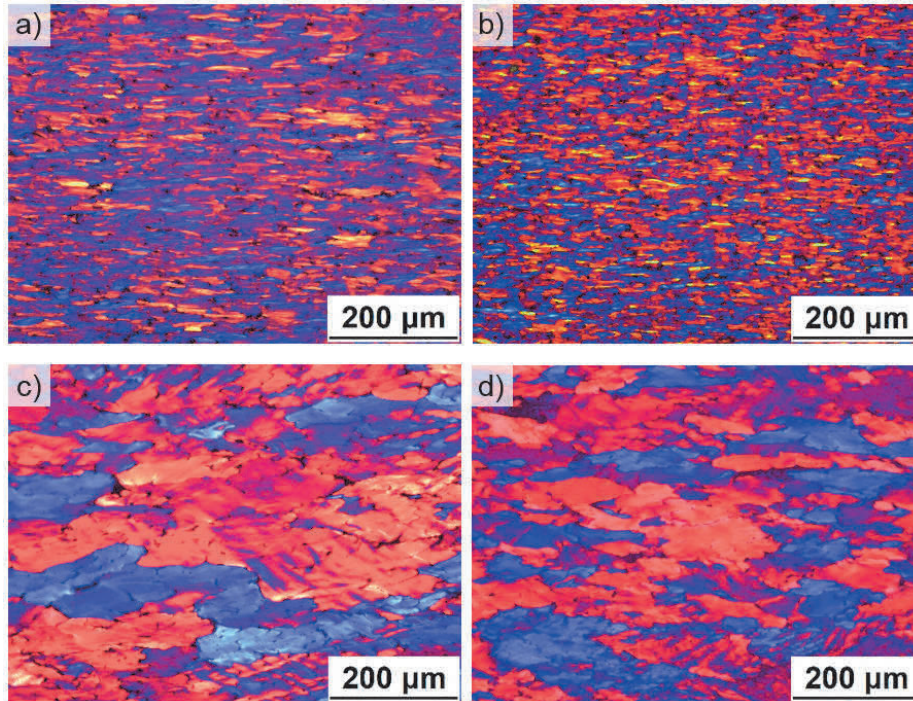
Samples for light optical microscopy (LOM) were mechanically ground by SiC papers and subsequently polished by diamond suspensions. Afterwards the samples were anodized with a Barker's reagent in Lectropol 5. Initial states of the material and final states after annealing at 600 °C were observed in polarized light in LOM.

## 3. RESULTS

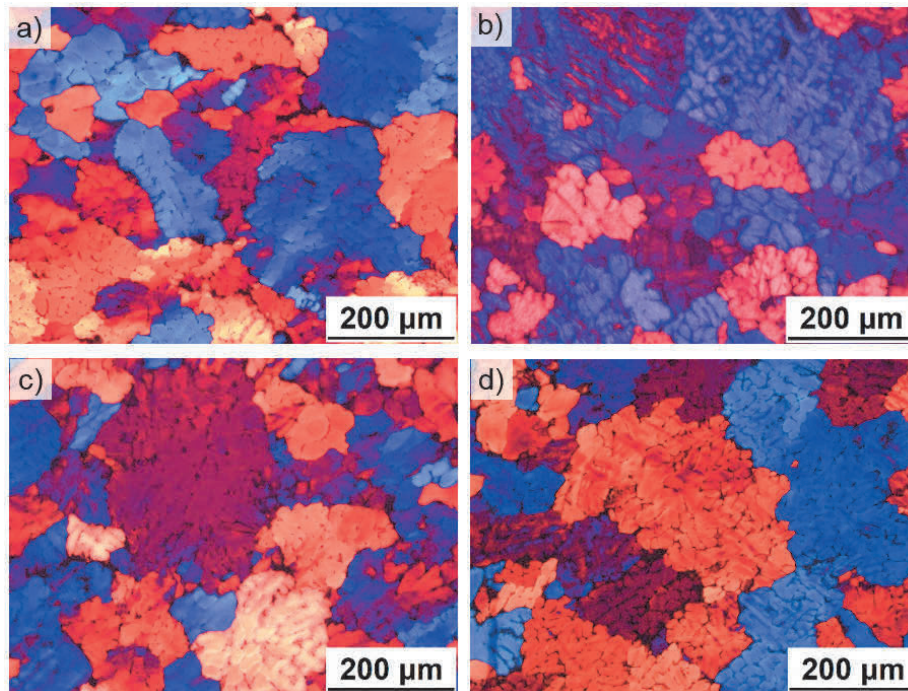
### 3.1. Initial state

The CC material is characterized by elongated grains about 40  $\mu m$  long and 5  $\mu m$  thick (**Figure 2**). On the contrary, the TRC material has a coarser structure. The grains are more equiaxed and their size in the central part of the strip is around 150  $\mu m$  (**Figure 3**). Additional hot rolling of the TRC material results in a partial flattening of the grains. However, the elongation is not as significant as in the case of the CC material. The average grain size is similar to the one in TRC specimen without rolling. Constrained groove pressing does

not have noticeable impact on neither the size nor the shape of the grains (**Figure 3**). CGP probably leads only to a fragmentation of the grains into subgrains [13].



**Figure 2** The microstructure of rolled materials in initial states. Comparison of the structure of mould cast sample after rolling by industrial mill (CC samples) and TRC sample rolled by a laboratory mill (TRC + hot rolling): (a) CC, (b) CC + 300 °C/8 h, (c) TRC + 300 °C/8 h + hot rolling, (d) TRC + hot rolling + 300 °C/8 h



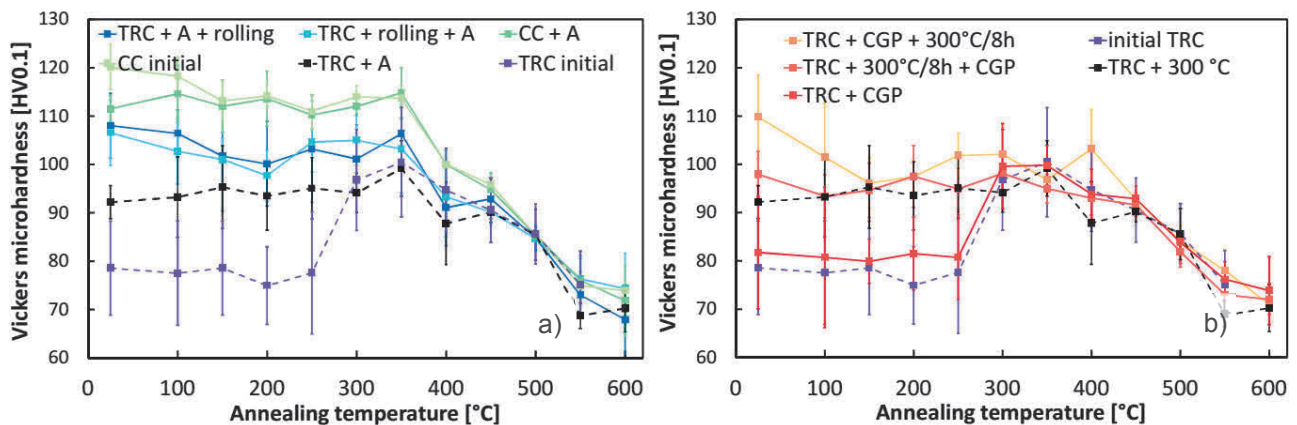
**Figure 3** The microstructure of TRC material after annealing at 300 °C and TRC material subjected to CGP: TRC + 300 °C/8 h, (b) TRC + CGP, (c) TRC + 300 °C/8 h + CGP, (d) TRC + CGP + 300 °C/8 h



### 3.2. Microhardness

The evolution of microhardness during exposure to elevated temperatures in all specimens can be seen in **Figure 4**. The CGP and TRC materials that have not been annealed at 300 °C/8 h evince lower microhardness in the initial state than the annealed ones. Initial states of non-annealed TRC and CPG materials differ from the pre-annealed materials by about 15 HV0.1.

Both rolled materials studied in this paper were annealed for 300 °C during their preparation, therefore they have higher microhardness already in the initial state without any successive annealing. When compared to the rolled material without any annealing [14], its microhardness is also lower.



**Figure 4** The evolution of microhardness: (a) in rolled materials (conventionally cast material rolled in industrial mill vs. TRC material and TRC material rolled in the laboratory mill), (b) in TRC material and TRC material subjected to CGP.

### 3.3. Isochronal annealing

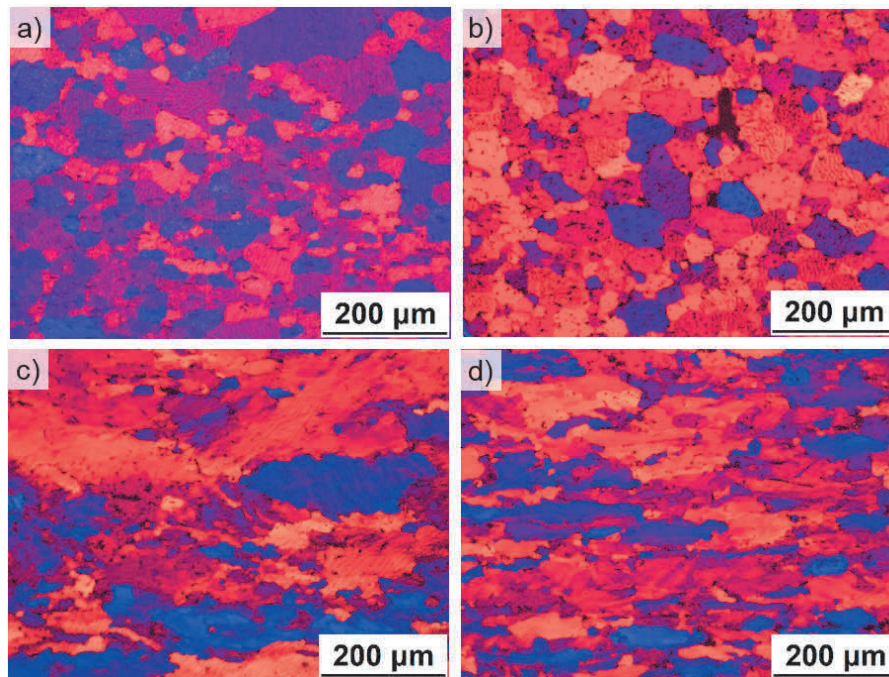
Up to 300 °C the most pronounced changes in microhardness occurred in TRC and CGP materials without the initial annealing. Above 250 °C the values of microhardness increase to the values observed in the pre-annealed materials. The values of microhardness of the remaining samples do not change significantly up to 350 °C. Above 350 °C, softening occurs in all materials (most probably in two stages) and at the highest annealing temperature the values of microhardness of all materials are the same within the experimental error. However, the differences in microstructure after annealing up to 600 °C persist. Equiaxed grains form in CC sample (**Figure 5**). On the contrary, the microstructure of TRC samples is not fully recrystallized and grains are larger (**Figure 6**) than in CC.

## 4. DISCUSSION

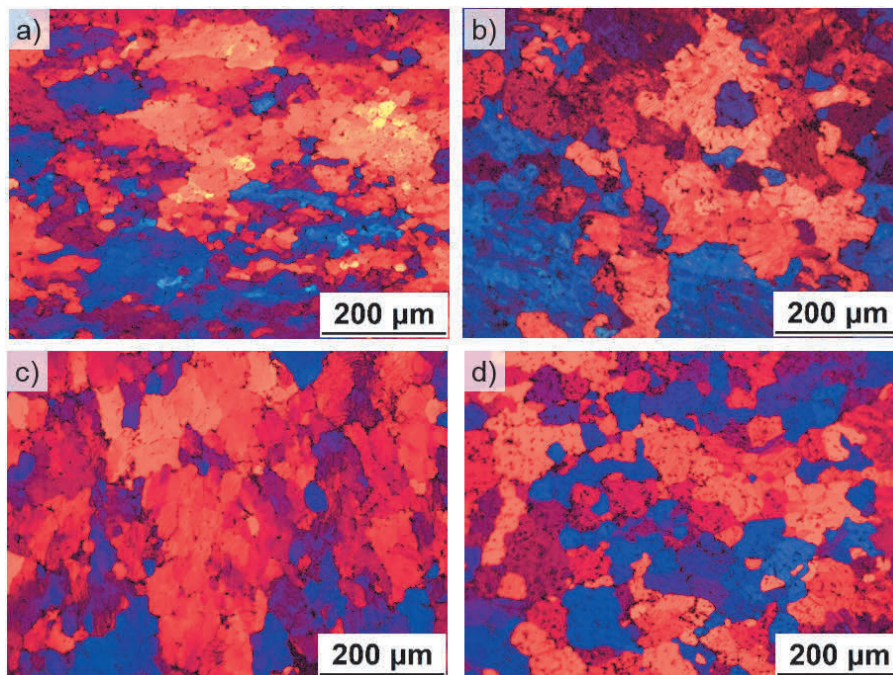
The influence of precipitation strengthening during isochronal annealing becomes evident particularly for TRC and CGP material. Remaining materials (CC and hot rolled) were exposed to higher temperatures during their preparation. Such annealing has already led to the precipitation of  $Al_3(Sc,Zr)$  particles in Al alloys with the addition of Sc and Zr which contribute to strengthening of the material. The most intensive precipitation usually appears at 300 °C. Therefore, the microhardness of TRC and TRC+CGP reaches the microhardness of pre-annealed samples not until this temperature. The remaining samples contain the particles from the beginning of the experiment and isochronal annealing below 300 °C does not have any significant effect on the microhardness. Markedly higher values of microhardness of CC samples are connected with the fine grain structure and hardening due to Hall-Petch relation [7].

The succession of deformation and annealing at 300 °C processes does not influence the evolution of microhardness significantly which differs from the observation on the same material processed by equal channel angular pressing [15]. The materials softening above 350 °C appears as a consequence of several

overlapping processes: most probably they are the dislocation recovery, polygonization, and overaging of  $Al_3(Sc,Zr)$  particles below 450 °C and recrystallization and partial dissolution of  $Al_3(Sc,Zr)$  particles above this temperature.



**Figure 5** The microstructure of rolled materials in final states after isochronal heating up to 600 °C: (a) CC, (b) CC + 300 °C/8 h, (c) TRC + 300 °C/8 h + hot rolling, (d) TRC + hot rolling + 300 °C/ 8h



**Figure 6** The microstructure of TRC material subjected to precipitating annealing at 300 °C/8 hours and TRC material subjected to CGP in final states after isochronal heating up to 600 °C: (a) TRC + 300 °C/8 h, (b) TRC + CGP, (c) TRC + 300 °C/8 h + CGP, (d) TRC + CGP + 300 °C/8 h

## 5. SUMMARY

The influence of the initial preparation method of AlMgScZr strips on the evolution of microhardness during isochronal annealing was studied. It was shown that the annealing of the as-cast and CGP specimens result in the formation of strengthening particles accompanied by a significant increase of microhardness. Above 350 °C the degradation of all materials occurs due to the overaging of strengthening particles and withdrawal of the strengthening effect of the deformation structure.

## ACKNOWLEDGEMENTS

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