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The Influence of Annealing on Microstructure and Microhardness of Twin-roll Cast Al-Mg-Sc-Zr Alloy

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Twin-roll casting is an effective and energy-saving method enabling direct preparation of strips of the requested final thickness. However, due to high solidification rates and higher solid solution supersaturation a significantly different microstructure is formed in comparison with conventionally cast alloys. Recently, the Sc and Zr containing aluminum alloys are considered to be new advanced materials for the transportation industry. The influence of Sc and Zr on mechanical properties of twin-roll cast Al-Mg alloy was investigated in the present study. A significant effect of Sc and Zr on hardening of the material after annealing in temperature interval between 250 °C and 400 °C was confirmed. Maximal hardness was obtained after two hours of annealing at 300 °C due to precipitation of a fine dispersion of Sc and Zr containing precipitates. Exposure of the annealed material to higher temperatures results in a partial degradation of mechanical properties due to changes of the strengthening effect of precipitates. However, a partially positive role of remaining precipitates, hindering recrystallization, persists even up to 550 °C.

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1. Introduction

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

There are two routes through which the mechanical properties can be improved. The first one is based on grain refinement and work hardening, which can enhance the strengths of the material through the grain boundary strengthening or dislocation strengthening. However, this positive effect is immediately lost during recrystallization. Another way of improving strength is by addition of elements which form strengthening particles through aging heat treatment.

Addition of scandium and zirconium to Al-Mg alloys offers attractive combination of properties. The precipitation of metastable $Al_3(Sc,Zr)$ particles during aging increases the strength levels of the alloy. The precipitates form at temperatures above 300 °C (typically 350 °C) and serve as highly effective obstacles for dislocation and grain boundary motion, inhibiting thus the recovery and recrystallization of the material [5–10].

However, alloys containing more than 3 wt.% Mg are susceptible to localized attack by intergranular corrosion and to exfoliation when they are exposed to temperatures ranging between 50–180 °C [11–14]. This susceptibility is predominantly linked to the precipitation of highly anodic β-phase (Al_3Mg_2) along the grain boundaries [15]. This phenomenon is often termed as sensitization. Moreover, the presence of stress along with a sensitized microstructure leads to a strong susceptibility towards intergranular stress corrosion. The continuous precipitation of β-phase leads to accelerated failure through anodic dissolution in the presence of detrimental chemical species like chloride ions.

The nucleation and growth of boundary precipitates were found to be strongly affected by the type of boundary [16] because of the correlation between the Mg segregation level and grain boundary energy [17]. In spite of the presence of precipitates at low-angle boundaries, stronger attack resistance is observed at grain boundaries with misorientation angle less than 20°. Strong β-phase continuous precipitation and subsequent corrosion attack is, therefore, often observed in layered pancake grain structures formed generally in rolled sheets.

Therefore, avoiding the formation of layered grains or their substitution by random network of low-angle boundaries may result in a significant limitation of β-phase formation in Al-Mg alloys and in improvement of their corrosion resistance. The above-mentioned type of microstructure could be achieved in conventional aluminum alloys prepared by direct-chill (DC) casting and subsequent rolling (which is necessary for obtaining a thinner sheet or strip) through controlled thermo-mechanical treatment, including recrystallization of the deformed material.

An alternative procedure eliminating the unfavorable recrystallization is the use of direct casting of sheets to the final thickness (typically 3–8 mm) by continuous casting methods (for instance twin-roll casting - TRC) without the necessity of using subsequent rolling steps.

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Grains formed during solidification of TRC strips are usually equiaxed containing low density of dislocations that are often organized in rare dislocation cell-walls or incorporated in low angle boundaries. Therefore, no additional recrystallization treatment is necessary in order to remove undesirable flat grains.

In TRC Al-Mg-Sc-Zr alloys the final strengthening can be simply achieved by artificial aging at approximately 350 °C and work hardening by deformation methods which do not change the thickness of the strip and also do not generate pancake grain structure – typically some SPD (severe plastic deformation) methods. 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.

In the present contribution the influence of annealing on microstructure and microhardness of a model Al-Mg-Sc-Zr alloy prepared by TRC is described. Properties are compared with a reference TRC Al-Mg alloy with a similar level of Mg addition. Al-Mg-Sc-Zr alloys have never been prepared by TRC before, and experimental data about their properties are not available in the literature. The present study describes the behavior of this new class of structural materials during annealing.

2. Experimental details

TRC alloys with the composition shown in Table I and thickness of 5 mm were studied. Twin-roll casting was realized with a vertical operation plane. No release agents were applied on the rolls surface before or during the process. The material was melted from the original DC cast and rolled strip (see Fig. 1) in a resistance furnace with the mechanical stirring of the melt.

TABLE I

Chemical composition of Al-Mg-Sc-Zr and Al-Mg alloys (in wt.%).

Alloy	Mg	Sc	Zr	Mn	Si	Cu	Fe	Zn	Al
Al-Mg-Sc-Zr	3.24	0.19	0.14	0.16	0.11	0.024	0.21	<0.002	bal.
Al-Mg	3.67	<0.005	<0.001	0.18	0.11	0.025	0.24	<0.002	bal.

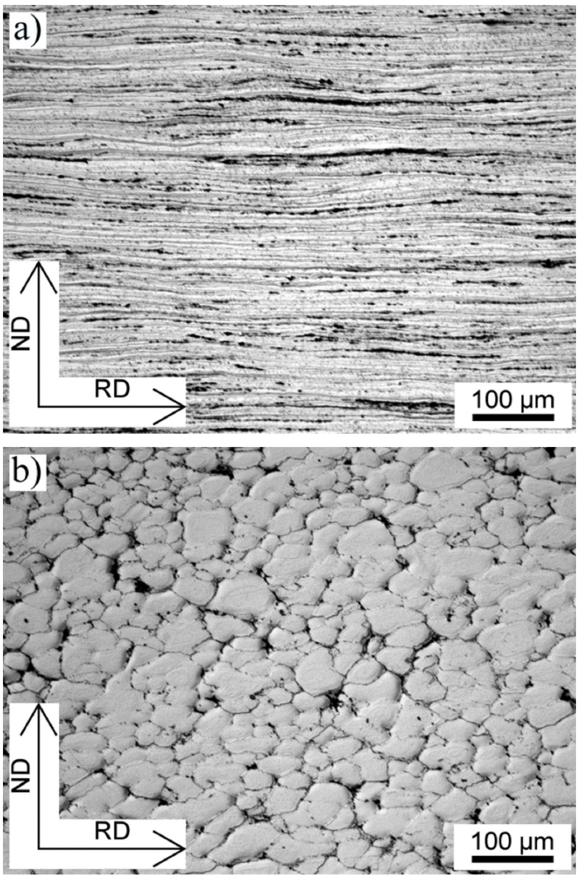

Fig. 1. Elongated flat grains in DC cast (a) and TRC (b) 5 mm thick strips prepared from Al-Mg-Sc-Zr alloy.

Casting was done at 655 °C with 2.75 m/min (2.65 m/min for Al-Mg alloy) casting rate. More detailed description of applied experimental equipment is given in [18]. In order to investigate the changes of mechanical properties and the microstructure at elevated temperatures, samples were annealed in an air furnace and quenched after each annealing. Annealing was carried out using step-by-step isochronal heating scheme up to 550 °C with steps of 50 °C/50 min or in an isothermal heating scheme at 300, 350 and 400 °C.

Vickers microhardness measurements HV 0.1 with the load of 100 g at QNess 10A were done after each step. Light optical microscopy (LOM) (Olympus GX51) and transmission electron microscopy (TEM) (JEOL JEM 2000FX) observations with the same heating scheme as the one employed during isochronal annealing, were used for the material characterization. Specimens for electron microscopy were electropolished in 30% HNO_3 solution in methanol at −15 °C.

3. Results and discussion

The TRC Al-Mg-Sc-Zr material contains slightly elongated grains 50–100 μm in size in the central part of the strip with the grain structure profoundly different from the one created by DC casting and subsequent rolling (Fig. 1). A comparison of TRC Al-Mg and Al-Mg-Sc-Zr alloys is shown in Fig. 2. A subtle grain elongation appearing in the central part of the TRC strip is typical when an additional deformation of already solidified central part of the strip occurs in the gap between the rolls, and grain coarsening is effectively inhibited by segregating primary phases rich in Fe, Si, Mg and Mn [19], which are always present in commercial purity Al alloys.

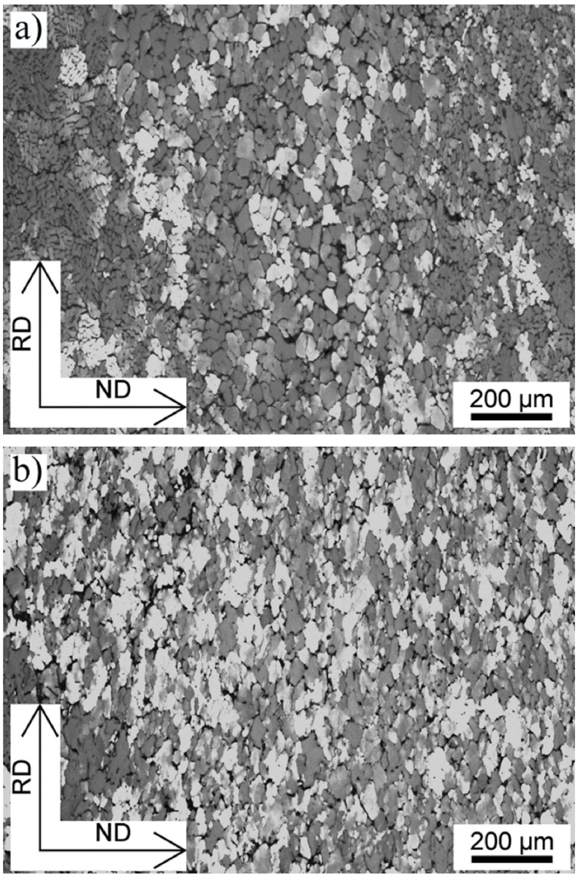

Fig. 2. Microstructure of TRC Al-Mg (a) and Al-Mg-Sc-Zr (b) alloys near the central part of the strip.

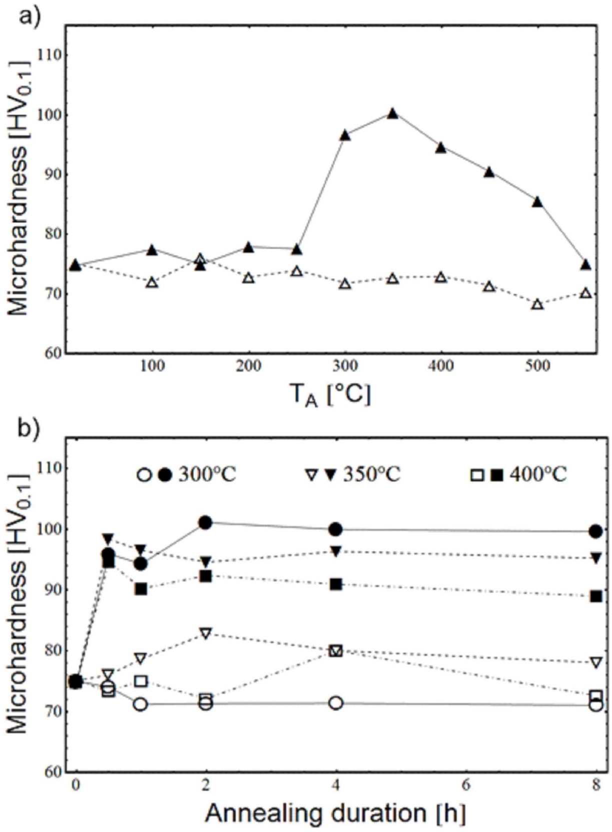

Fig. 3. Evolution of microhardness in TRC Al-Mg (empty symbols) and Al-Mg-Sc-Zr (full symbols) alloys during isochronal (a) and isothermal (b) annealing.

Coarser grains with the size of about 200 μm appearing in both alloys in the areas closer to the surface of the strip (well visible in the Al-Mg alloy in Fig. 2) reflect the inhomogeneity of the cooling of the material, which starts by a rapid solidification in the contact area between the melt and cooled rolls and propagates towards the central part. The central part is, therefore, always at higher temperature than the rest of the strip accommodating thus any deformation imposed by the rolls during casting. Negligible difference in the grain size between Al-Mg and Al-Mg-Sc-Zr alloys gives the evidence about the marginal role of Sc and Zr in the solidification process.

Figure 3a shows the evolution of microhardness in both TRC alloys during isochronal annealing. Only moderate changes are observed in the Al-Mg alloy during this treatment, which indicate that most probably only material homogenization associated with redistribution of Mg atoms, and recrystallization and grain coarsening might occur in the material.

On the other hand, a different evolution of microhardness was found in the Al-Mg-Sc-Zr alloy. A significant increase of microhardness appears in this material at annealing temperatures above 250 °C, reaching the peak hardness at 350 °C. Higher annealing temperatures result in a gradual reduction of microhardness to initial values.

Isothermal annealing confirms these trends (Fig. 3b). Considerably more pronounced changes of microhardness occur in the Sc and Zr containing alloy. The peak values of microhardness are reached in the Al-Mg-Sc-Zr alloy annealed at 300 °C already after two hours of annealing. Neither longer annealing times nor higher annealing temperatures result in the improvement of microhardness.

A comparable behavior is generally observed also in DC cast Sc and Zr containing aluminum alloys [10, 20, 21] treated in a similar manner. The evolution of microhardness is associated with the precipitation of $Al_3(Sc,Zr)$ particles. Usually the formation of fine dispersion of precipitates with the diameter of 5–10 nm results in hardening of the material, while their further coarsening is always connected with the material degradation and a loss of microhardness.

TEM observations performed on annealed specimens clearly prove that similarly in the TRC alloy it is the precipitation of $Al_3(Sc,Zr)$ particles that is responsible for the observed behavior of microhardness. Dispersion of fine $Al_3(Sc,Zr)$ precipitates was observed in the specimen annealed at 300 °C. A pronounced coarsening of the particles occurs above this temperature (Fig. 4). Although their growth to a large portion deteriorates mechanical properties, the positive role of

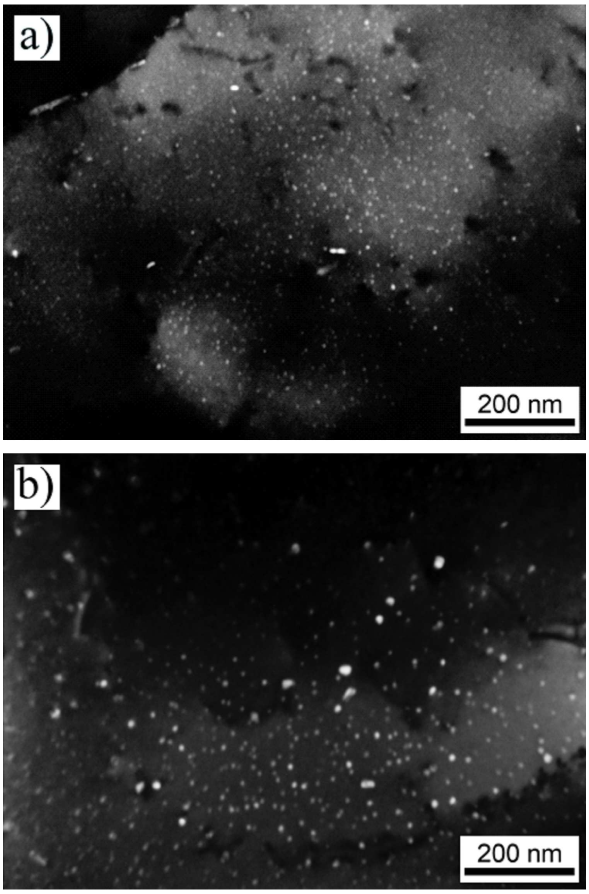

Fig. 4. Dark field TEM images of Al_3(Sc,Zr) particles viewed near the [100] Al zone axis in TRC Al-Mg-Sc-Zr alloy after isothermal annealing at 300 (a) and 400 °C (b) for 8 h.

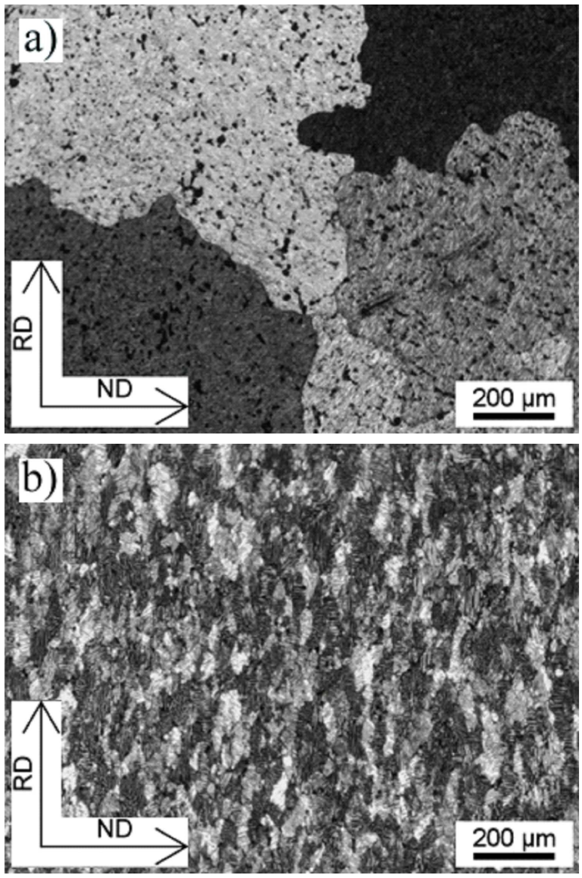

Fig. 5. Influence of Al_3(Sc,Zr) particles on grain coarsening during isochronal annealing up to 550 °C. Coarse grains in Al-Mg (a) and stable structure Al-Mg-Sc-Zr (b) alloys.

Al_3(Sc,Zr) precipitates as effective inhibitors of recrystallization is preserved even at highest annealing temperatures, as is documented in Fig. 5.

4. Conclusions

Twin-roll cast Al-Mg-Sc-Zr alloy was prepared. TRC enables preparation of strips of a necessary final thickness without additional rolling. In contrast to DC cast materials, where rolling is a necessary technological procedure, an equiaxed grain structure is formed by TRC. Maximal hardening of the TRC alloy by Al_3(Sc,Zr) particles is, similarly to DC cast materials, reached after two hours of annealing at 300 °C. Although the coarsening of Al_3(Sc,Zr) precipitates results in softening of the material, their positive role as inhibitors of recrystallization persists during the whole annealing process.

Acknowledgments

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