Equal-channel angular pressing is a method of severe plastic deformation, which implies high deformation into the material and leads to grain size reduction and strength increase. It was applied on a twin-roll cast Al-Mg-Sc alloy, which contained Al_{1}(Sc,Zr) particles formed during annealing at 300 °C for 8 hours. The evolution of the microstructure and mechanical properties after deformation was studied during isochronal heating 50 K/50 min. Strengthening occurred during annealing at lower temperatures and was followed by prominent microhardness drop, which was connected with formation of new grains and recrystallization. The presence of Al_{1}(Sc,Zr) particles in the matrix is accounted for the high temperature stability.

Keywords: Aluminium alloys, Equal-channel angular pressing, Al_{1}(Sc,Zr), Thermal stability, Recrystallization

1 Introduction

Al-Mg type of alloys finds its application mostly in the ship-building and aerospace industries thanks to their adequate corrosion resistance, good weldability [1-2] and possibility of superplastic forming [3-5].

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 [6-8], which occur mainly in materials with flat grains like those prepared by direct-chill casting (DC) where a pancake-like structure of the grain is formed during final processing by hot- or cold-rolling.

The DC technique can be substituted by continuous casting methods [9] like twin-roll casting (TRC) [10-13] which produce 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. When compared to the DC material, microstructure with nearly equiaxed grains is formed [14].

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_{1}(Sc,Zr) particles upon aging delivers contribution from precipitation hardening increasing thus the strength levels of the alloy [10,15-16]. The precipitates form at temperatures between 300 and 450 °C and serve as highly effective obstacles for dislocation and grain boundary motion [17-18] which shifts the recrystallization temperature to higher values.

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 [19]. One of the methods how to achieve this and not to generate pancake grain structure are 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 [8].

One of the most accessible SPD methods is equal channel angular pressing (ECAP) [20]. During ECAP the billet of material is pressed through a die which consists of Ti–Al–Si alloys produced by reactive sintering. In: Journal of Alloys and Compounds, Vol. 504, No. 2, pp. 320-324.


of two channels of equal cross-section intersecting at an angle $\Phi$, commonly 90° [21]. The possibility of repeating the pressing several times to induce a required level of strain into the material is the main advantage of this technique. As a result microstructure with submicrometric grains is formed.

The recent paper deals with the Al-Mg alloy containing $\text{Al}_3(\text{Sc,Zr})$ strengthening particles which was subjected to SPD by ECAP and its thermal stability at elevated temperatures.

2 Experimental

An aluminium 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 to thickness of 5 mm. This alloy was annealed at 300 °C for 8 hours and subsequently subjected to severe plastic deformation by equal-channel angular pressing at 250 °C with channel dimensions 5 x 5 mm$^2$, pressing speed 10 mm/min and 4 passes by route Bc [21] in total. The microstructure of the material was observed by transmission electron microscope (TEM) JEOL 2000FX working at 200 kV. To monitor the thermal stability the material was subjected to isochronal annealing with step 50 °C for 50 min up to 600 °C. The microhardness was measured on Qness A10+ device with load 100 g for 10 s for each annealing temperature and the final grains size and distribution was depicted by Light optical microscope (LOM) in polarized light after electrolytical etching by Barker solution.

3 Results and discussion

The twin-roll cast material was subjected to annealing at 300 °C for 8 hours in order to precipitate high density of $\text{Al}_3(\text{Sc,Zr})$ particles. The suitable time and temperature for this precipitation was found in work of Cieslar [20]. The precipitates are evenly distributed in the matrix (Figure 1) and they are easily distinguishable (due to their coherency with the matrix) by selected area diffraction, where they add small intensity to the diffraction spots, which are forbidden for pure aluminium.

The application of equal channel angular pressing leads to fragmentation of the original grains and formation of high density of high angle grain boundaries (Figure 1). However, the resulting microstructure is not fully equiaxed as would be required and is commonly observed in Mg alloys [22], but the grains are slightly elongated in a direction inclined to the pressing direction of ECAP [23].

![Fig. 1 TEM micrograph of the material after annealing at 300 °C for 8 hours (left) – dark field image of Al$_3$(Sc,Zr) precipitates, $B=[001]_\text{Al}$, $g=[100]_{\text{Al}_3(\text{Sc,Zr})}$. Material after ECAP processing (right) with slightly elongated micrometric grains with high angle grain boundaries](image)

The ECAP processing caused a significant increase of the microhardness to 100 HV which is a well described phenomenon in materials deformed by such procedure [21]. Further annealing led to changes of the microhardness – see Figure 2. The microhardness slightly increased between 200 and 300 °C. Such hardening may be contributed to the precipitation hardening by additional formation of $\text{Al}_3(\text{Sc,Zr})$ particles. This corresponds to observations on the same material process by different methods where the peak hardness was achieved between 300 and 350 °C [20,24-25].

At temperatures above 300 °C the trend of microhardness changes and fluent decrease of the microhardness takes place. Such drop is in aluminium alloys commonly connected with recovery and recrystallization. Concerning the grain microstructure, the evolution is depicted in Figure 3 from light optical microscope. At 500 °C the grains are still fragmented into small constituents not distinguishable by LOM, which is similar case to the state just after ECAP processing.

After further annealing to 550 °C small grains with size around 10 µm are already distinguishable, several
larger grains of 100 µm are also present. The most significant change in grain structure can be observed after annealing to 600 °C when the newly formed grains grow significantly up to average size in order of 100 µm causing further decrease in microhardness.

Such high recrystallization resistance of the material (over 500 °C) may be attributed to the presence of \( \text{Al}_3(\text{Sc}, \text{Zr}) \) particles which are known to serve as recrystallization inhibitors. In this type of alloys their influence is higher compared to e.g. Al-Mn type of alloys, where they shift the recrystallization temperature to the region around 450 °C [26].

![Microhardness evolution of the deformed material during isochronal annealing 50 °C/50 min.](image)

### 4 Conclusion

The microstructure with micrometric grains which formed after equal channel angular pressing gradually changes during isochronal annealing. Firstly, slight increase of microhardness is caused by precipitation strengthening by \( \text{Al}_3(\text{Sc}, \text{Zr}) \) particles. Further on, at temperatures above 300 °C recovery and recrystallization take place and new grain structure is formed with the size of the grains in order of 100 µm. The presence of \( \text{Al}_3(\text{Sc}, \text{Zr}) \) particles in the matrix successfully increases the recrystallization temperature above 500 °C as compared to other materials.

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### References


