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Effect of homogenisation treatment on precipitation, recrystallisation and properties of Al – 3% Mg – TM alloys (TM = Mn, Cr, Zr)

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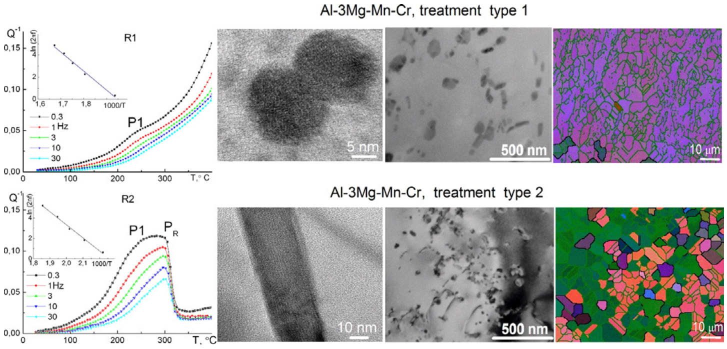
HIGHLIGHTS

- Coherent Mn-bearing particles are found after homogenisation annealing.
- Cr dissolved in the Mn-bearing phase as shown by point TEM-EDS analysis.
- The activation parameters of relaxation effects are estimated.
- Superplasticity is achieved after treatment with high temperature homogenisation.

GRAPHICAL ABSTRACT



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ABSTRACT

The influence of homogenisation regimes on precipitations of fine Mn-bearing dispersoids, recrystallisation kinetics, grain structure, mechanical properties and superplasticity of cold worked sheets of Al-3Mg alloys with Mn, Cr and Zr is studied. Precipitates of coherent compact and plate-like particles of Mn-bearing phase are found after homogenisation annealing. Activation parameters of the low-temperature internal friction, internal friction solute grain boundaries peak and dislocation nature thermally activated relaxation peak in the samples treated by different regimes are compared. High-temperature homogenisation at 460 °C provides a higher volume fraction and finer particles of Mn-containing phases after thermomechanical processing compared to low temperature treatment at 380 °C. Consequently, finer grains and higher superplasticity indicators are achieved in a recrystallised state. Polygonisation is observed during heating of the cold worked samples pre-homogenised at 380 °C: recrystallisation temperature is increased and mechanical strength at room temperature is improved.

1. Introduction

Al-Mg based alloys are widely used in airspace, shipbuilding, automotive and machine-building industries due to their good strength, corrosion resistance, easy formability, ability to superplastic forming and good weldability of such alloys [1]. High Mg-containing alloys, typically with from 4.5 to 6 wt.% Mg, exhibit high ultimate tensile strength, suchAl-Mg based alloys are also used for superplastic forming due to their finer grain size compared to low magnesium containing alloys. Decreasing of Mg concentration allows simplifying sheets manufacture, reducing costs and increasing corrosion resistance of Al-Mg based alloys. It is the main reason to study alloys with low concentration of Mg. This type of alloys cannot be strengthened by a heat treatment because

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coarse Al_3Mg_2 precipitates are formed after ageing. The most effective method to improve mechanical properties and superplasticity of Al-Mg based alloys is micro-alloying by transition metals such as Mn, Zr, Sc, Cr, Ti [2–4]. These elements form fine particles, also known as dispersoids, with size from 5 to 100 nm and allow achieving a high recrystallisation resistance which in turn helps to avoid strength loss during exposure at high temperatures [5–7]. At high annealing temperature, when recrystallisation occurs, the dispersoids pin grain boundaries (Zener pinning) and prevent a subsequent growth of the grains [6,8,9]. Grain refinement increases yield stress, elongation and fracture toughness at room temperature and leads to superplasticity of the alloys [10,11]. Dispersoids are formed at heat- and thermomechanical treatments due to a decomposition of the supersaturated solid solution of as-cast alloys [12–16]. A heat treatment regime significantly influences such parameters of the particles as type, size and volume fraction [17–20]. Fine coherence with the aluminium matrix $L1_2$-structured Al_3Zr, Al_3Sc or $Al_3(Sc, Zr)$ precipitates could be formed during Al-Zr, Al-Sc and Al-Sc-Zr solid solution decomposition [21–24]. These alloys require a low-temperature treatment to form the finest particles and to achieve maximum strengthening [25,26]. Sc and Zr-effects are widely studied in Al-Mg alloys. Al_3Sc or $Al_3(Sc, Zr)$ phases are formed in Al-Mg-Sc or Al-Mg-Sc-Zr alloys and improves both mechanical properties and superplasticity [27–29]. Zr-effect on the microstructure, mechanical properties and superplasticity of Al-Zn-Mg and Al-Cu based alloys is significant [30–33]; however, for Al-Mg-Mn alloys, the effect of a Zr-addition is much weaker, and very few Al_3Zr-dispersoids after homogenisation are identified in [34]. In contrast, high density of dispersoids of Al_3Zr phase is formed in Al-4Mg-1Zr alloy after severe plastic deformation (friction stir processing) and the alloy exhibits high strain rate superplasticity [35]. The homogenisation temperature and other processing parameters can influence the decomposition of aluminium solid solution and the formation of the Al_3Zr coherent phase. The processing temperature also influences the size of Mn containing dispersoids [36,37]. An increase in the homogenisation temperature is accompanied by an increase in the size of dispersoids [36]. I. Nikulin, et al. [38] concluded that a low temperature annealing at 360 °C provides the formation of fine Al_6Mn particles with a size of 25 nm after equal-channel angular pressing in an Al-Mg-Mn-Zr alloy, which is the main factor of improving the mechanical properties.

This study is focused on the analysis of the microstructure evolution, the mechanical properties and the superplasticity of sheets of Al-3 Mg based alloys containing of Mn with Cr and Mn with Zr. Sheets were produced by thermomechanical treatment using different homogenisation annealing temperatures. The results have been analysed and interpreted on the basis of various methods, such as a microstructural analysis and a mechanical testing including a mechanical spectroscopy method.

2. Materials and methods

2.1. Materials and processing

Al-based alloys with 3 wt.% Mg and different additions of transition metals (Table 1) are studied. The maximum of Mg solubility in Al solid solution at room temperature is 2.3% Mg [39]. Alloys with 3 Mg cross the solvus line at ≈185 °C and contain a low volume fraction ≈2% of the β (Al_3Mg_2) phase. The studied alloys contain low Mg concentration (3%) and high Mn concentration (1.2%).

Table 1
Chemical compositions of the studied Al – Mg – TM alloys.

Alloy	Composition, wt.%					
	Mg	Mn	Cr	Zr	Other less than	Al
1	3.0	1.2	0.10		0.01	Bal.
2	3.0	1.2	0.25		0.01	Bal.
3	3.0	1.2		0.26	0.01	Bal.

The alloys were melted in a Nabertherm S3 electric furnace with air atmosphere using graphite-fireclay crucibles. The 99.99 grade aluminium, the 99.95 grade magnesium, 'pre-alloyed' master compositions: Al-10% Cr, Al-10% Mn, Al-3.5% Zr were used to prepare the alloys. The sheets were obtained in the laboratory conditions using the following steps: 1) casting of the ingot into a water-cooled copper mould with the dimensions of $100 \times 40 \times 20$ mm^3 and the cooling rate of about 15 K/s, 2) 8 h of homogenization at 380 °C or at 460 °C, 3) hot rolling to 80% of deformation at a temperature 380 ± 5 °C, and finally 4) subsequent cold rolling with a 90% thickness reduction (true strain $e = -2.3$). A thermomechanical treatment regime with homogenisation at 380 °C was named R1 regime and with homogenisation at 460 °C was named R2 regime. The final sheets had 1 mm thickness.

The chemical composition of the alloys was controlled by SEM-EDS analysed after both homogenisation and cold rolling. Deviations from the nominal composition did not exceed 0.05 wt.%. The solidus temperature of all the alloys was approximately 615 °C. Solidus temperature was determined by a differential thermal analysis using "Setaram Labsys DSC 1600" at heating rate of 5 K/min in air atmosphere.

2.2. Microstructures analyses

The grains structure characterisations were carried out by means of a "Neophot 30" optical light microscope (OM) with polarised light. A Tescan-VEGA3 LMH scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectrometer (EDS) (X-MAX80, Oxford Instruments) and with an EBSD – HKL detector (NordlysMax EBSD, Oxford Instruments) was used for the microstructural studies. The electronic images were created at a voltage of 20 kV. The EBSD scan size was 400×400 μm^2, with a 0.3 μm scan step. Specimens for SEM investigations were prepared by mechanical grinding, polishing in colloidal silica based suspension, and anode oxidising in water solution of 10% (HF in H_3BO_4) in case of the analysis in polarised light.

The phase composition was examined by conventional X-ray diffractometry (XRD) using Brucker Advance D8 X-ray diffractometer with Cu-Kα radiation. Transmission electron microscopy (TEM) observations were carried out using a JEOL JEM–2000 EX and high resolution field emission gun JEOL JEM 2010 microscope operating at 200 kV and equipped with an energy dispersive X-ray spectrometer (EDS). Ellipse-shaped 3 mm samples with a major axis parallel to the stress direction were used. The discs were electrochemically thinned by twin-jet polishing using Struers Tenupol and the A2 electrolyte at a temperature range of (0 ± 2) °C. The average particles (dispersoids) size was determined by the random secant method, using >500 measures. The error bars were determined experimentally using a standard deviation and a confidence probability of 0.95.

2.3. Mechanical spectroscopy

A dynamical mechanical analyser DMA Q800 TA Instruments was used to study temperature dependent internal friction (TDIF) in the range of temperatures from 25 to 400 °C in the frequency (f) range of 0.3–30 Hz and the strain amplitude (ε_0) of 5×10^{-5}. The applied stress was represented as $\sigma = \sigma_0 \cos(\omega t)$, and the corresponding strain was $\varepsilon = \varepsilon_0 \cos(\omega t + \varphi)$, where $\omega = 2\pi f$ and φ was the loss angle. The internal friction (IF, denoted in most publications as inverse quality factor: Q^{-1} [40]) for the forced vibrations is equal to tan φ. Specimens for the damping tests were cut down along the rolling direction to a size of $1 \times 5 \times 60$ mm^3. The analysis of internal friction and the calculation of the activation parameters method are described in detail in our previous papers [40–42].