Precipitation and recrystallisation in Al–Mn–Zr with and without Sc

Børge Forbord*, Håkon Hallem, Nils Ryum, Knut Marthinsen
Department of Materials Technology, Norwegian University of Science and Technology, Trondheim, Norway

Received 25 August 2003; received in revised form 10 September 2003

Abstract

The transition elements Zr and Mn form dispersoids in aluminium alloys and are extensively used in order to control the microstructure during heat exposure, i.e. to prevent recrystallisation and strength loss after deformation hardening. Additions of Zr have, through the formation of a dense dispersion of coherent and metastable Al₃Zr-dispersoids, proved to be beneficial in many alloys. In the Al–Mn–Zr-alloy studied here, TEM-investigations of homogenised material have shown that these phases are heterogeneously distributed. However, by adding Sc a high density of small and well-dispersed Al₃(Sc, Zr)-dispersoids is obtained during homogenisation, and the Sc-containing variant consequently displays a higher recrystallisation resistance after cold rolling.

Keywords: Aluminium alloys; Precipitation; Zirconium; Scandium; Dispersoids; Recrystallisation

1. Introduction

The transition element Zr forms dispersoids in aluminium alloys and is added in order to achieve a high recrystallisation resistance, i.e. to avoid strength loss during exposure at high temperatures. Recrystallisation involves the formation of strain-free nuclei/subgrains and the subsequent growth of these into the surrounding matrix. However, the formation of recrystallised grains will only occur if the subgrains are able to grow larger than a certain size, $R_C$. This is described mathematically by the Gibbs–Thomson relationship:

$$R > R_C = \frac{4\gamma_{GB}}{P_Z-P_D}$$

where $R_C$ is the critical radius for nucleation, $\gamma_{GB}$ the specific grain boundary energy and $P_D$ the stored deformation energy. $P_Z$ is the retarding force (usually referred to as the Zener-drag) that the dispersoids exert upon the subgrain boundaries. This should be as large as possible in order to achieve a high recrystallisation resistance, see Eq. (1). A widely used estimate for the Zener-drag is [1]

$$P_Z = \frac{3f\gamma_{CA}}{2\pi r}$$

where $r$ is the radius and $f$ the volume fraction of dispersoids. Eq. (2) shows that a high volume fraction of small dispersoids (high $f/r$-ratio) is necessary in order to achieve a large Zener-drag and a high recrystallisation resistance. It is also important that the dispersoids are homogeneously distributed in order to avoid areas with a low retarding force.

All these criteria were fulfilled when Auran et al. [2] added Zr to Al–Mg–Zn-alloys, as a high density of well-dispersed and coherent Al₃Zr-dispersoids with an average diameter of approximately 10 nm formed during heat treatment. Although no direct evidence was presented, Auran et al. believed that evenly distributed Zn₂Mg-precipitates acted as nucleation sites in Al–Mg–Zn. However, in, for instance, Al–Mn–Mg-alloys the effect of a Zr-addition is much smaller, see Riddle et al. [3], who found very few Al₃Zr-dispersoids after homogenisation. These results indicate that the precipitation of Al₃Zr is largely dependent on the alloying additions.

In this work an Al–Mn–Zr alloy with and without Sc has been studied. One of the main objectives has been to further investigate the precipitation of Al₃Zr in Mn-containing alloys. The influence of a Sc-addition on the precipitation of dispersoids and the subsequent effect on the recrystallisation resistance after cold rolling has also been examined.
Table 1
Chemical compositions of the alloys

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Zr</th>
<th>Si</th>
<th>Fe</th>
<th>Mn</th>
<th>Sc</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.15</td>
<td>0.15</td>
<td>0.21</td>
<td>1.01</td>
<td>–</td>
</tr>
<tr>
<td>2</td>
<td>0.13</td>
<td>0.15</td>
<td>0.20</td>
<td>0.91</td>
<td>0.17</td>
</tr>
</tbody>
</table>

2. Alloy selection and experimental work

2.1. DC-casting and machining of rolling slabs

Billets with diameters of 95 mm were DC-cast at Hydro Aluminium, Sunndalsora, see chemical compositions in Table 1. The furnace temperature was approximately 700°C and Al–5 wt.% Ti–1 wt.% B grain refiner was added at a concentration of 1 kg/ton. After casting rolling slabs of dimensions 11 mm × 60 mm × 200 mm were cut from the billets and grind in order to obtain a smooth surface before cold rolling.

2.2. Homogenisation, cold rolling and annealing

The rolling slabs were homogenised at 450°C in an air-circulating furnace for 12 h. A heating rate of 50°C/h was applied during the heat treatment. After homogenisation the slabs were quenched in water and subsequently cold rolled 90% to a thickness of approximately 1.1 mm. In order to investigate the structural stability, i.e. recrystallisation resistance, samples were cut from the cold rolled material and annealed (from RT) in an air-circulating furnace using a heating rate of 50°C/h. The samples were removed from the furnace at temperatures between 100 and 600°C and then quenched in water.

2.3. Hardness measurements

Vickers hardness measurements were carried out in order to follow the softening reactions during annealing.

Fig. 1. Dark field TEM-images showing areas with (a) a high and (b) a low density of Al3Zr in alloy 1. (c) The dense distribution of Al3(Sc, Zr)-dispersoids in alloy 2.

Fig. 2. Softening curves after homogenisation and cold rolling.

Fig. 3. The fully recrystallised structure of the cold rolled alloy 1 heated to 500°C.
hardness was determined as the average of 4 measurements at a load of 1 kg.

2.4. TEM/EELS/EDS

A Jeol 2010 operated at 200 kV was used in the TEM-investigations. EELS was used in order to measure the thickness of the TEM-foils. Particle sizes, densities and volume fractions were then determined from dark field images and analysed by the computer programs Adobe Photoshop and ImageTool. Five hundred to 1000 dispersoids were measured/ counted in each sample. The EDS-analysis was carried out by placing a focused nano-beam (spot size 5 nm) directly on the dispersoids. Analyses of the surrounding matrix were also collected in order to eliminate the contribution from the matrix to the dispersoid spectra.

2.5. Optical microscopy

Polarised light was used during the studies of the grain structure. The samples were investigated in the longitudinal section after cold rolling, and both the onset and completion of recrystallisation were identified.

3. Results and discussion

3.1. Homogenisation

Precipitation takes place during homogenisation, and in alloy 1 the Al<sub>3</sub>Zr-phase mainly precipitates continuously as coherent and spherical dispersoids. These have an average radius, r, of 11 nm and are heterogeneously distributed (Fig. 1a and b). In some areas the density of Al<sub>3</sub>Zr may be as high as 9 x 10<sup>20</sup> m<sup>-3</sup>, while in others the density of these dispersoids is very low. These observations indicate a low driving force for Al<sub>3</sub>Zr-precipitation in many areas of alloy 1.

Alloy 2, on the other hand, displays a dense distribution of homogeneously distributed Al<sub>3</sub>(Sc, Zr)-dispersoids with an average radius, r, of 6 nm, see Fig. 1c. The dispersoids are fully coherent with the matrix and are present at an average density of 7 x 10<sup>21</sup> m<sup>-3</sup>, i.e. eight times as high as the maximum number density in alloy 1. These observations indicate that the Sc-addition markedly increase the driving force for precipitation.

3.2. Structural stability after cold rolling

Softening curves for the cold rolled material are given in Fig. 2, and it is worth noticing that the strength drop associated with recrystallisation occurs at significantly lower temperatures in alloy 1 than in alloy 2. While alloy 1 is fully recrystallised at 500° C, alloy 2 displays a fibrous/unrecrystallised structure even at 550° C, see Figs. 3 and 4a, respectively. However, at 600° C alloy 2 is fully recrystallised as well (Fig. 4b).

This can be related to both the density/volume fraction and distribution of dispersoids, i.e. the f/r-ratio at various locations in the two alloys. This is described in Eqs. (1) and (2), which show that the Zener-drag/recrystallisation resistance becomes larger when the f/r-ratio increases. In alloy 2 the high density and the homogeneous distribution of dispersoids ensure that f/r is high everywhere. As a consequence this alloy displays a remarkable recrystallisation resistance. Alloy 1, however, displays areas with low dispersoid densities (low f/r) and is consequently more prone to recrystallisation.

4. Conclusions

In Al–Mn–Zr the Al<sub>3</sub>Zr-dispersoids mainly precipitates continuously as coherent and spherical dispersoids which
are heterogeneously distributed and in some areas virtually absent. However, they are heterogeneously distributed and in some areas they are virtually absent. However, when Sc is added a very dense and homogeneous distribution of dispersoids is obtained, as a result of an increased driving force for precipitation. Due to the more favourable dispersoid distribution, the Sc-containing alloy displays a far better recrystallisation resistance.

References