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Controlling grain structure and texture in Al-Mn from the competition between precipitation and recrystallization

K. Huang [a, b, *], K. Zhang [b], K. Marthinsen [b], R.E. Logé [a]

[a] Thermomechanical Metallurgy Laboratory – PX Group Chair, Ecole Polytechnique Fédérale de Lausanne (EPFL), CH-2002, Neuchâtel, Switzerland
[b] Department of Materials Science and Engineering, Norwegian University of Science and Technology, Trondheim, N-7491, Trondheim, Norway

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ABSTRACT

The recrystallization behaviour of Al-Mn alloys (AA3xxx series alloys) is affected by randomly distributed dispersoids present before annealing, by dispersoids precipitated at grain/subgrain boundaries before the onset of recrystallization, and by dispersoids concurrently precipitated during recrystallization. In this study, the effects of these three populations of dispersoids on the recrystallization behaviour of a cold rolled AA3xxx alloy were analysed and compared using four temperature-time paths to different target temperatures. Changing the temperature-time path modifies the extent of recovery, the dispersoid structures, as well as the absolute recrystallization temperature, which then influences the final grain structure and recrystallization texture. In particular, an in-depth investigation on how different populations of dispersoids affect the main recrystallization texture components of AA3xxx alloys, i.e., P{011}⟨566⟩, ND-Cube {001}⟨310⟩, and Cube {001}⟨100⟩, has been carried out. The results clearly show that, as compared to isothermal annealing, annealing with more elaborate heating and annealing schedules (temperature-time paths) all lead to increased strength of the P texture component and decreased intensities of both the Cube and ND-rotated Cube texture components. The increase of P texture strength and average grain size is most significant when recrystallization occurs concurrently with precipitation. The controlling mechanisms behind this behaviour and the possibility to use them to tailor the grain structure and texture of similar alloys are further discussed.

1. Introduction

Aluminium AA3xxx-series alloys (i.e. alloys with Mn as their main alloying element) are widely used in packaging, beverage cans and the automobile heat exchanger industry, with different requirements on grain structures and textures. The supersaturated Mn may precipitate as fine dispersoids due to its limited solubility in Al [1], e.g., during homogenization and back annealing (i.e. annealing after cold/hot deformation), thus changing the microchemistry of the alloy in terms of solute level and particle structure [2]. Three types of dispersoids, including randomly distributed dispersoids formed during homogenization (type I), dispersoids precipitated on (sub)grain boundaries before recrystallization (type II), as well as dispersoids precipitated on (sub)grain boundaries during recrystallization (type III), are usually involved during the processing of these series of alloys. The heterogeneous precipitation of type II and III dispersoids is hence different from that found in precipitation hardening alloys such as Al-Cu where precipitation can occur homogeneously throughout the microstructure during annealing [3]. Since the grain size and texture can change significantly when the microchemistry of the material, as well as the thermo-mechanical processing conditions change [2,4], there is a strong scientific and industrial interest in better understanding and controlling the combined effect of these three types of dispersoids on recrystallization of the non-heat treatable Al 3xxx alloy products.

The fine dispersoids precipitated out during homogenization after casting (type I) are mainly randomly distributed and their effect on the subsequent recrystallization behaviour after deformation is relatively well understood. The effect of this type of dispersoids has often been analysed by annealing the deformed materials at high temperature to avoid the other two types (type II and III) of dispersoids. This usually leads to an equiaxed grain structure with Cube as the major texture component [2,5].

* Corresponding author. Thermomechanical Metallurgy Laboratory – PX Group Chair, Ecole Polytechnique Fédérale de Lausanne (EPFL), CH-2002, Neuchâtel, Switzerland.
E-mail address: huangke0729@hotmail.com (K. Huang).

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Cold or hot deformation after homogenization introduces more heterogeneous nucleation sites for precipitation. At low annealing temperatures of the deformed material, dispersoids can heterogeneously precipitate on grain/subgrain boundaries before possible recrystallization initiates (type II) or concurrently with recrystallization (type III) [4–6]. The precipitation of large numbers of type II and/or III dispersoids at lower annealing temperatures can retard or even totally suppress recrystallization [5–7]. In addition to slow recrystallization kinetics and a resulting coarse elongated grain structures, less common texture components like ND-rotated cube {001}⟨310⟩ and P {011}⟨566⟩ are often observed when precipitation occurs during annealing [2,4–6,8–13]. The effect of dispersoids of type II and III on the development of the P-orientation is usually not separately investigated, since they are collectively considered as "concurrent precipitation". The individual contribution from type II and type III dispersoids therefore requires further investigation.

Annealing with different temperature-time paths modifies the microchemistry evolution, it can thus determine which type of dispersoids interacts with recrystallization, an aspect which has not been systematically investigated so far. Cold-rolled AA3xxx-type alloy specimens were annealed by Schäfer and Gottstein [14] using different temperature-time paths, however, the effect of dispersoids along grain/subgrain boundaries before recrystallization (type II) was mostly left unexploited. In another recent work, the effect of type II dispersoids on the recrystallization behaviour of an *as-cast* AA3xxx model alloy, which has negligible type I dispersoids, was investigated [15]. However, annealing of the deformed as-cast AA3xxx alloys leads to a less frequently observed sharp M {113}⟨110⟩ component [4,12,15], which shares some similarities with, but which is still distinctively different from the P- and ND-rotated cube texture components and makes the analysis more complex. The influence of "concurrent precipitation" (both type II and III according to the current definition) on the recrystallization behaviour of the same material has recently been *quantitatively* investigated in terms of nucleation and growth behaviour of the P-orientation [16]. Computer simulations have also been performed to study and probe the complex interactions between dispersoids and recrystallization. In these conditions, physically based numerical models usually fail to predict the correct recrystallization kinetics [7,17]. The different grain structures and recrystallization textures obtained at different annealing temperatures are usually not described, see e.g. Refs. [18,19]. In other words, there is currently no numerical model that can successfully predict the different grain structures and recrystallization textures during annealing with different temperature-time paths of cold deformed AA3xxx alloys. To the best knowledge of the authors, there is no detailed analysis focusing on the combined effect of these three populations of dispersoids on recrystallization, even though each of them, to a different extent, does play a role in determining the recrystallization kinetics, final grain structure and texture.

In this study, a homogenized AA3xxx model alloy, which contains a large number of randomly distributed fine dispersoids, was cold rolled to a true strain of $\varepsilon = 3.0$. These deformed samples were then heated with four carefully designed temperature-time schedules to various target temperatures to induce dispersoid precipitation preferentially located at grain/subgrain boundaries, before and during recrystallization, respectively. The main objective is to explore the effect of these three different populations of dispersoids on the microstructure and crystallographic texture of the AA3xxx alloy. The results obtained in this paper provide new understanding for microstructure design through thermo-mechanical processing of particle-containing metallic materials, as well as a guide to its associated numerical modelling. The different temperature-time paths are expected to have limited effect on the variation of large constituent particles at the investigated target temperatures [20,21].

2. Experimental

The examined material was a DC-cast AA3xxx-type model alloy supplied by Hydro Aluminium, with the chemical composition (wt.%) of 0.152% Si, 0.530% Fe, 0.390% Mn. Previous experiments have shown that about 0.35 wt % of Mn is still in solid solution after casting, the other small part was precipitated out as large constituent particles of ~0.88 μm in diameter [2]. The as-cast material was homogenized in an air circulation furnace where the samples were heated at 50 °C/h from room temperature to 450 °C and then kept for another 4 h before water quenching. It has previously been shown that in this condition 0.16 wt % of Mn was still left in solid solution [2], i.e., 0.19 wt % of Mn was consumed either such as to produce randomly distributed fine dispersoids with average diameter of 50 nm and number density of 1.3×10^6 mm^{-2} (particles smaller than 20 nm are not counted), or contribute to the growth of constituent particles which reached about 0.96 μm in diameter.

The homogenized materials were cold rolled with good lubrication conditions at room temperature from slabs of a thickness of 30 mm−1.5 mm by multiple passes to an accumulated true strain of $\varepsilon = 3.0$. The rolled sheet was water cooled after few passes to avoid deformation heating. These deformed samples were then annealed according to the four different temperature-time paths illustrated in Fig. 1. One set of the deformed samples were directly annealed in a pre-heated salt bath at: i) 500 °C for 5s; ii) 350 °C for 10^5s and iii) 400 °C for 10^5s, all referred to as isothermal annealing samples as shown in Fig. 1a. Another set of samples were subjected to a two-step annealing where the samples were isothermally pre-annealed at 300 °C for 10^4s before the final annealing at 350, 400 and 500 °C, respectively, as shown in Fig. 1b. The samples subjected to three-step annealing, as shown in Fig. 1c, were heated at 50 °C/h to 300 °C and held for 10^4s before the final isothermal annealing step. In addition, some samples were annealed with a constant heating rate of 50 °C/h all the way to the target temperatures, followed by isothermal annealing for 10^5s, as illustrated in Fig. 1d, hereafter just termed as "slow annealing". Finally, a few supplementary tests slowly annealed to 325 °C and 335 °C, which will be detailed later in the discussion part when relevant results appear, were also conducted. All samples were quickly water quenched after the heat treatments.

The evolution of electrical conductivity (EC) was measured in order to estimate the precipitation behaviour, with eight measurements conducted on the rolling direction (RD) - transverse direction (TD) section for each condition, even though only the averaged values and associated standard deviations are shown in this work. The RD- normal direction (ND) cross sections of the samples were polished according to standard metallographic procedures. Particles were observed with a backscattered electron (BSE) detector and grain structure and orientation were obtained by Electron backscatter diffraction (EBSD) in a Zeiss Supra 55 or FEI XLF 30 field emission gun scanning electron microscope (FEG-SEM), at the Norwegian University of Science and Technology (NTNU) and Ecole Polytechnique Fédérale de Lausanne (EPFL), respectively. Inverse pole figure (IPF) maps of the annealed samples were obtained and analysed with the TSL or HKL softwares, using a scanning step size of 0.25–2 μm, to study both the grain structure and recrystallization texture. In all micrographs, the horizontal direction corresponds to the RD direction while the vertical direction is the ND direction. Due to the large grain size in most of the investigated conditions, a large area (~2–4 mm^2) was scanned by EBSD to give a statistically reliable representation of the texture and average grain size, although only a small area is usually presented in the micrograph. The grain size was measured as the equivalent