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The effect of heating rate on the softening behaviour of a deformed Al–Mn alloy with strong and weak concurrent precipitation

K. Huang [a,b,*], K. Marthinsen [b]

[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, N-7491 Trondheim, Norway

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ABSTRACT

The interaction between recrystallization and concurrent precipitation is strongly dependent on the heating rate during annealing. In this study, the effects of heating rate on the grain structure, recrystallization texture and resulting mechanical properties upon annealing of a cold-rolled Al–Mn alloy under strong and weak concurrent precipitation conditions, resulting from different homogenization procedures, were investigated. It is clearly shown that increasing the heating rate leads to finer grain size, less elongated grain structure and reduced flow stress for both cases. In the case of strong concurrent precipitation, a sharp P-texture component is obtained after recrystallization, with its intensity decreasing with increasing heating rate. On the other hand, Cube is the major texture component when concurrent precipitation is weak, and its strength increases with increasing heating rate. The different effects brought about by the heating rate on the recrystallization texture of the two cases, which has not been addressed in the literature so far, are further discussed in view of the differences in precipitation behaviour.

1. Introduction

In many particle-containing alloys, second-phase particles are already present during deformation and subsequent annealing does not substantially change the particle structure. However, in materials such as Al–Mn alloys (AA3xxx), the supersaturated Mn may diffuse to create fine dispersoids during annealing after deformation, a process commonly termed as concurrent precipitation. Depending on how fast recrystallization completes, the recrystallization behaviour of these alloys can be divided into three regimes [1]: i) precipitation takes place before recrystallization; ii) precipitation occurs concurrently with precipitation; and iii) recrystallization completes before precipitation starts. If a slow heating rate is employed, sufficient precipitation may take place at high and low angle grain boundaries [2] to inhibit recrystallization even at high temperatures. On the other hand, if the recrystallization temperature is reached in a short time with a rapid heating rate, a fully recrystallized state can obtained before substantial precipitation takes place. Therefore, it is expected that the heating rate will have a strong effect on recrystallization of supersaturated Al–Mn alloys..

Al–Mn alloys are widely used in the beverage can body, in packaging and automobile heat exchanger industry due to their moderate strength and good ductility. Grain size and texture control is essentially important for these applications. For alloys with fixed chemical composition, it is the combined effect of the microchemistry state [3] in terms of solute level and second-phase particle structure, and the thermomechanical processing that determines the microstructure and thus the properties of the final products. The effect of microchemistry on the softening behaviour of Al–Mn(−Fe-Si) alloys has previously been extensively investigated (e.g. [3,4]), and it has been concluded that the recrystallization kinetics, as well as the final microstructure and texture can be strongly affected by the microchemistry state. In terms of the effect from different thermomechanical processing conditions, most investigations are focused on different deformation strains and annealing temperatures [2,3,5–7], while some of them also have emphasized the heating rate effect [8–13]. The influence of heating rate on the final microstructure, however, will be different even for the same material because of differences in the microchemistry evolution, an aspect which has not been addressed to the same extent in the literature so far. Different heating rates modify the sequence of recrystallization and concurrent precipitation and thus bring about different microchemistry states within the material, which finally leads to different grain structure and texture after recrystallization.

In this study, two variants of an Al–Mn (−Fe-Si) model alloy, with high and low precipitation potential respectively, are obtained from different homogenization procedures. All the samples of these two variants are then cold-rolled to a true strain of $\varepsilon = 3.0$ and subsequently heated to the same target temperature with identical holding time using three different heating rates. The microstructure and texture after annealing have been characterized by Electron backscatter diffraction (EBSD) in scanning electron microscopy (SEM). The effect of

* Corresponding author at: Thermomechanical Metallurgy Laboratory — PX Group Chair, Ecole Polytechnique Fédérale de Lausanne (EPFL), CH-2002 Neuchâtel, Switzerland.
E-mail address: ke.huang@epfl.ch (K. Huang).

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heating rate during recrystallization on the grain structure, mechanical properties and texture of the two supersaturated Al–Mn alloy variants, with different microchemistry states, is carefully analysed and discussed.

2. Experimental

The as-received material was in as-cast (direct chill) state, with the chemical composition (wt.%) of 0.390% Mn, 0.530% Fe, 0.152% Si with the balance Al. The detailed description of the as-cast material has been published previously in Ref [3]. The as-cast material (variant A) was subsequently homogenized in two steps, in an air circulation furnace with a temperature accuracy of ±2 °C, starting from room temperature (about 20 °C), to get a different level of Mn in solid solution and dispersoid density. The samples were first heated at 50 °C/h to 600 °C for 4 h, and then cooled at 25 °C/h to 500 °C where they were kept for another 4 h, which gave the B variant. Materials were water quenched to room temperature at the end of the homogenization procedure..

Samples of both variants were cold-rolled at room temperature in multiple passes to a true strain of $\varepsilon = 3.0$. The cold-rolled sheets were then heated in an air circulation furnace at different heating rates (50 °C/h and 200 °C/h) to 400 °C and were held for 10^5 s before water quench to ensure a fully recrystallized structure for each condition. Some additional samples heated at 50 °C/h were water quenched before reaching to 400 °C to follow the evolution of microstructure during heating. The fastest heating rate at 7.6×10^6 °C/h, is realized by quickly immersing the sample into a pre-heated salt bath. Owing to the inherent limit from the furnace, it is not possible to achieve another heating rate between 200 and 7.6×10^6 °C/h.

For all of the micrographs presented in this paper, the horizontal direction corresponds to rolling direction (RD) while the vertical direction is the normal direction (ND). Both hardness and electrical conductivity (EC) measurements were performed on the RD–TD plane of the samples in order to follow the softening and precipitation behaviours during annealing. For each reported value, eight different measurements on the examined plane were conducted and analysed, which give the average value and standard deviation. A load of 1 kg, a dwell time of 15 s and a loading speed of 100 μm s^{-1} were used for the hardness measurements. EC was measured by a Sigmascope EX 8 instrument at room temperature of about 293 K (20 °C). The microstructure and crystallographic textures of the sheets were measured by means of Electron backscatter diffraction (EBSD) on a Zeiss Supra/Ultra 55 scanning electron microscope (SEM) equipped with TSL software. The constituent particles and dispersoids were examined using the backscattered electron (BSE) detector mode of the same SEM. Orientation maps of both deformed and annealed samples, covering more than one thousand grains (except for the cases with extremely large grains), with step size of 1–2 μm, were used to study the orientation of the recrystallized grains and thus the texture after recrystallization. The grain size was measured as the equivalent circular diameter of grain sections in the RD–ND cross section. The texture was represented using orientation distribution functions (ODFs) determined by EBSD using the series expansion method. If the observed grain orientation was within a 15° scatter (in all directions) from the ideal components, it was classified as belonging to that specific component, including {112}<111> (copper), {124}<211> (R), {123}<634> (S), {113}<110> (M) [7], {001}<100> (Cube), {011}<566> (P) and {001}<310> (ND-rotated Cube). The remaining orientations are considered as random orientations.

3. Results and discussion

3.1. Microchemistry and microstructure before annealing

The microchemistry state of the two variants have been reported previously [3], but the main characteristics are recapitulated here to increase the readability of the present paper. No pre-existing dispersoids are present in the as-cast variant A, but it has a significantly higher concentration of Mn in solid solution, as listed in Table 1. On the other hand, a small number of dispersoids is observed for B, together with a lower level of Mn, reflecting a decreased potential for precipitation of Mn-containing dispersoids during subsequent annealing. In fact, the concurrent precipitation during annealing for variant A is found to be quite strong as opposed to variant B for which it is fairly weak [3]. The solid solution level of Mn was estimated based on the relationship between the conductivity and concentration of alloying elements in solid solution, details of which can be found in Ref. [14,15]. The increase of EC during annealing of the Al–Mn alloy is mainly because of the depletion of the supersaturated Mn in solid solution, i.e., it reflects the decrease of Mn solute content and the associated increase of concurrent precipitation.

Table 1
Electrical conductivity, concentrations of solute, diameter and number density of particles in the alloys studied [3].

	Electrical conductivity ($m/\Omega\ mm^2$)	Concentration of Mn (wt.%)	Constituent particles		Dispersoids	
			Diameter (μm)	Number density (mm^{-2})	Diameter (μm)	Number density (mm^{-2})
A	23.9	0.35	0.88	2.8×10^4	–	–
B	29.0	0.11	1.10	2.1×10^4	0.127	5.5×10^4

As an example, the deformed microstructure and texture of variant B are shown in Fig. 1. After deformation to a strain of $\varepsilon = 3.0$, the material shows a banded deformation structure with most of the high angle grain boundaries aligned with the rolling direction. A strong β fibre rolling texture was obtained after cold deformation, as illustrated in Fig. 1b.

3.2. Effect of heating rate for samples with strong concurrent precipitation

For the samples of variant A, the estimated concentration level of Mn (0.35 wt.%) is significantly above the level at equilibrium state which is less than 0.1 wt.% at 400 °C, suggesting a high potential for concurrent precipitation. Strong concurrent precipitation indeed takes place when annealing the deformed samples, as indicated by the increased EC both at the lowest and highest heating rate, as can be seen in Fig. 2.

In Fig. 2a, the variation of EC and hardness is plotted against the actual annealing temperature, which is directly connected to annealing time since a constant heating rate of 50 °C/h was used. Note that the variation during the holding time is not included. Our previous work has shown by EBSD micrographs that recrystallization started at ~350 °C for variant A during annealing [4]. At this lowest heating rate, discernible precipitation takes place before the onset of recrystallization, i.e., during static recovery, as indicated by the increase of EC before 300 °C. However, precipitation becomes distinctively more pronounced when the sample has reached to higher temperatures (e.g. 350 °C), due to the increased diffusion rate of solute elements, and strong interaction between precipitated dispersoids and the recrystallization process is thus expected. As an example, the precipitated dispersoid structure of the sample heated to 350 °C for variant A is shown in Fig. 3. A large number of fine dispersoids can be observed along the grain/subgrain boundaries, which are roughly aligned with the RD direction, in the deformed matrix (see Fig. 3a), confirming the strong precipitation indicated from the increase of EC. In Fig. 3b, it is clear that some dispersoids are located at the boundary of the growing recrystallized grain, but it is also clear that the recrystallization grain boundary/front has overcome the pinning effect from precipitated dispersoids (aligned along RD, as indicated by the light blue arrows), which are left behind the moving grain boundary. Recrystallization for this condition is completed at ~400 °C, as the hardness has dropped to the fully recrystallized state value at this temperature (see Fig. 2b).