# The Effects of Dispersoids on the Recrystallization Behavior in a Cold Rolled AA3103-Aluminium Alloy

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#### Abstract

The effects of dispersoids present prior to softening vs. concurrent precipitation on the recrystallization process after cold rolling have been considered. The materials containing a large supersaturation of Mn and consequently a large precipitation potential, are strongly affected by precipitation taking place concurrent to the softening processes. Further, it has been shown that the recrystallized grain structure and texture is relatively unaffected by the dispersoids present prior to annealing unless the density becomes very high, and in these cases the effect becomes similar to the effects caused by concurrent precipitation, but weaker. In these extreme cases of precipitation, both nucleation and growth of the recrystallized grain structure and a strengthening of the texture.

#### 1. Introduction

A challenge during processing of aluminium alloys is the control of grain size and texture. In general, the grain size in a product is a function of many parameters such as initial grain size, thermomechanical history and the alloy composition. It is well established that second-phase particles have a strong effect on softening kinetics, final grain size and texture [1-6]. Deformation zones may develop around large particles (>1  $\mu$ m) during deformation, which may activate particle stimulated nucleation of recrystallization (PSN), whereas small, closely spaced dispersoids have the ability to retard (Zener drag) both low-and high-angle boundary motion [7]. The Zener Drag is usually given as  $P_Z=4f_{\gamma SB}/3r$ , where *f* is the volume fraction of particles of radius *r* and  $\gamma_{SB}$  is the subgrain boundary energy. This relationship assumes a random distribution of particles. The present paper investigates the effect of precipitation on the evolution of recrystallized grain size of a commercial AlMn-alloy. The main objective was to investigate the effects of dispersoids present prior to the annealing vs. precipitation during annealing, *i.e.* concurrent precipitation, on the recrystallization process after cold rolling.

#### 2. Experimental Procedure

2.1 Material Casting and Homogenization.

The material used in the present work was a commercially DC-cast AA3103 aluminium billet (0.57wt%Fe, 1.00wt%Mn, 0.12wt%Si). Small rolling slabs were cut out from a billet,

where the surface section was avoided due to the reverse segregation zone. The material was investigated both in the *as-cast.* (0-state) and homogenized condition (H-state). Homogenization was performed using a heating rate of 100°C/h to 610°C, held at temperature for 14 hours and finally water quenched.

## 2.2 Deformation and Pre-annealing Treatments

Two deformation processes were selected. One batch of material was cold rolled (CR) on a laboratory scale to a true strain of 0.5 (39% reduction). The cold rolled sheets were cut in two, where one of the parts was pre-annealed at 300°C for  $10^6$ s (~11<sup>1</sup>/<sub>2</sub> days), while the other part was pre-annealed at 350°C for 10<sup>6</sup>s. During pre-annealing, Mn is supposed to precipitate into dispersoids, and the precipitation rate will be enhanced by the existing deformation structure. It should be noted that no recrystallization were observed in the preannealed materials. Finally, the different pre-annealed sheets were cold rolled further to an accumulated strain of 3.0 (95% reduction). This process route was selected to obtain a material with the Mn precipitated as Mn-bearing dispersoids, hence, simulating an intended homogenization procedure leading to an extremely dense dispersoid structure. In the following this process route will be referred to as the two-step deformed material (T). The second batch was directly cold rolled to a true strain of 3.0 after casting or homogenization, *i.e.* giving a large supersaturation of Mn in solid solution. This deformation process is referred to as the single-step deformed materials. A total of six material variants were investigated, four two-step deformed variants and two single-step deformed variants. The specimen denotations are given in Table 1.

Specimen	Explanation	δ [MS/m], initial state	δ [MS/m], CR to ε=3	Disp. density [µm <sup>-2</sup> ]
S-3-0	As-cast variant, CR to $\varepsilon$ =3	17.71 (0.83)	17.64 (0.84)	~0
S-3-H	Hom. variant, CR to $\varepsilon$ =3	21.87 (0.52)	21.87 (0.53)	~0
T-3-0-300⁰C	As-cast variant, CR to $\varepsilon$ =5, pre-annealed at 300°C/10 <sup>6</sup> s and further CR to $\varepsilon$ =3	17.71 (0.83)	25.38 (0.35)	22.6
T-3-0-350⁰C	As-cast variant, CR to $\varepsilon$ =5, pre-annealed at 350°C/10 <sup>6</sup> s and further CR to $\varepsilon$ =3	17.71 (0.83)	30.75 (0.16)	21.3
T-3-H-300°C	Hom. variant, CR to $\varepsilon$ =5, pre-annealed at 300°C/10 <sup>6</sup> s and further CR to $\varepsilon$ =3	21.87 (0.52)	26.58 (0.30)	8.8
T-3-H-350°C	Hom. variant, CR to $\varepsilon$ =5, pre-annealed at 350°C/10 <sup>6</sup> s and further CR to $\varepsilon$ =3	21.87 (0.52)	31.27 (0.15)	6.2

Table 1: Specimen denotations with explanation and variation in electrical conductivity,  $\delta$  (MS/m), and Mn in solid solution (in parentheses, wt%) with process step for the single- and two-step deformed materials. The dispersoid densities ( $\mu m^{-2}$ ) in the final cold rolled materials are also included.

## 2.3 Softening:

The cold rolled sheets were cut into small specimens and annealed isothermally in salt baths at temperatures between 350°C and 500°C, for different times. The annealed specimens will be denoted by annealing temperature and time (*e.g.* 500°C-100 s).

## 2.4 Microstructure Characterization:

The microstructures have been characterized using the polarized light optical microscope (PLOM) technique and a high resolution FESEM equipped with a solid state backscatter detector and an EBSD system. The Oxford FEATURE software has been and to measure particle size distributions in 2D, while a modified Johnson-Saltykov method was applied to transform the distributions from 2D to 3D [8]. Dispersoid densities were measured from high magnification FESEM-micrographs.

## 3. Experimental Results and Discussion

#### 3.1 Characterization of the Starting Material As-cast and Homogenized Material

The *as-cast* microstructure consisted of equiaxed grains with a mean grain size of about 100 µm and a random texture. During casting, second phase intermetallic Al<sub>6</sub>(Mn,Fe) and some  $\alpha$ -Al(Mn,Fe)Si particles are formed on the grain boundaries. Microstructural investigations showed that the *as-cast* material contained few dispersoids. The homogenized material contained some dispersoids, but the density was quite low and they were heterogeneously distributed throughout the matrix. This is probably due to the high holding temperature and subsequent water quenching. The particle size distributions for the *as-cast* and homogenized structures are given in Figure 1, where it is seen that the size of the primary particles increases during homogenization. This is mainly due to decomposition of the Mn in supersaturated solid solution. It is clear that the *as-cast* state contains more small particles and less large ones as compared to the homogenized state.



Figure 1: 3D-cumulative particle size distribution of the AA3103-alloy in the *as-cast* and homogenized conditions, both for the un-deformed and deformed materials.

The variation of Mn in solid solution resulting from the different processing steps for the single- and two-step deformed materials has been monitored by electrical conductivity measurements and these results are included in Table 1. It should be noted that the measurements were performed on the materials after cold rolling to accumulated strains of 3. However, the conductivities after pre-annealing and final cold rolling of the two-step materials were quite similar. It may be pointed out that for the two-step materials the electrical conductivity increases considerably due to the combination of cold rolling and pre-annealing. The highest conductivities are found when pre-annealing at 350°C, which probably is due to a larger diffusivity compared to pre-annealing at 300°C. Table 1 also gives the respective concentrations of Mn in solid solution calculated with the Matthiessens rule assuming only Mn in solid solution [9].

#### 3.2 The Deformed State Single-step Deformed Material

A FESEM-micrograph from the deformed state of the single-step material (S-3-H) is given in Figure 2 (a). The micrograph shows a typical deformation structure consisting of deformation bands elongated in the rolling direction, with an interior subgrain structure. The subgrain size in the S-3-H material was measured by EBSD to be 0.26  $\mu$ m and 0.47  $\mu$ m in the directions parallel to ND and RD, respectively. The particle size distributions for the single-step deformed materials in the deformed conditions are included in Figure 1. It can be seen that there are only minor differences in the particle size distributions prior and after cold rolling, indicating no or little particle break-up. The single-step deformed materials in the *as-cast* and homogenized condition both contains a large supersaturation of Mn in solid solution (~0.8wt% and ~0.5wt%, respectively), giving a large precipitation potential during the subsequent softening (see Figure 2 (b)).



Figure 2: Micrographs showing (a) deformation structure in the S-3-H material and (b) concurrent precipitation heterogeneously on the deformation structure (S-3-0 softened at 350°C for 10<sup>4</sup>s).

## 3.3 Two-step Deformed Material

The deformation structures evolving from the two-step deformation route were quite similar to the deformation structures in the single-step deformed materials. The subgrain size was measured in the T-3-H-350°C material by EBSD to be 0.34  $\mu$ m and 0.57  $\mu$ m in the directions parallel to ND and RD, respectively. These are somewhat larger than the corresponding values for the single-step variant, which is probably due to the pre-annealing sequence after a strain of 0.5 giving considerable recovery in the two-step material, i.e. subgrain growth. In addition the amount of Mn in solid solution is considerable lower than in the single-step variants and this have been observed to give a lower hardness after the final cold rolling sequence. These observations indicate a somewhat smaller driving pressure for recrystallization in the two-step deformed materials.

The particle size distributions have been measured for the four two-step deformed materials as well, but they were guite similar to the corresponding variants of the singlestep material and are therefore not included in Figure 1. Microstructural observations showed that the dispersoids precipitated heterogeneously on the deformation structure during pre-annealing. The different dispersoid densities resulting from the pre-annealing sequence in case of the four different two-step variants after cold rolling to accumulated strains of 3.0 are included in Table 1. It is clear that the two as-cast variants contain the largest densities, while the variants pre-annealed at 300°C gives somewhat larger densities than pre-annealing at 350°C. These observations might be explained by a larger precipitation potential in the as-cast variants, and a larger diffusion rate at 350°C leading to some coarsening of the dispersoids. It can be stated that the goal of achieving materials with a high density of dispersoids and a low supersaturation has been reached. The Zener drag is stronger for large volume fractions and small dispersoids, *i.e.* it is clear that the ascast materials give the largest Zener drags. The conductivity were larger for the materials pre-annealed at 350°C, consequently it is reasonable to believe that the volume fraction of dispersoids are larger at 350°C. However, the dispersoids have been seen to be smaller when pre-annealed at 300°C, hence the difference in Zener drag between the materials pre-annealed at 300°C and 350°C becomes small due to the f/r-ratio.

### 3.4 Recrystallized Microstructures

The different microstructures resulting from softening of the single-step materials at high and low annealing temperatures are shown in Figure 3. It is observed that the microstructures resulting from high temperature annealing becomes fine grained. This is because the material is able to recrystallize before the precipitation begins, *i.e.* the precipitation *nose* is avoided [6]. This is however, not the case for lower temperature annealing. Now the grain structure becomes clearly affected by concurrent precipitation, leading to an inhomogeneous, coarse grained, pancake shaped grain structure. The dispersoids precipitated on the deformation structure during softening as seen in Figure 1 (b), and this heterogeneous precipitation has been shown to give a larger effective Zener drag than predicted by the classical Zener drag equation [4]. The dispersoids are retarding both nucleation (subgrain growth) and growth of recrystallized grains.



Figure 3: PLOM micrographs of the single-step deformed materials after isothermal annealing at high and low temperatures. (a) S-3-0/500°C-100s, (b) S-3-0/400°C-10<sup>4</sup>s, (c) S-3-H/500°C-10s and (d) S-3-H/350°C-10<sup>4</sup>s.



Figure 4: EBSD-maps (with Euler colours and grain boundaries) of the two-step deformed materials. (a) T-3-0-300°C/500°C-3x10<sup>5</sup>s, (b) T-3-0-350°C/500°C-3x10<sup>5</sup>s, (c) T-3-H-300°C/500°C-100s. The micron bar is 100µm.

The different microstructures resulting from annealing of the two-step materials at 500°C are shown in Figure 4. The two *as-cast* variants become coarse grained, i.e. the structures are clearly affected by the Zener drag resulting from the dispersoids present prior to annealing. The two homogenized variants become much less affected by the dispersoids, resulting in a relatively fine grained material. This shows that the softening processes are sensitive to the density of the dispersoids present prior to softening, but this density needs to be *very* high. The coarse grained materials in the two *as-cast* variants do however, not become as coarse as the single-step materials experiencing concurrent precipitation. The recrystallized grain sizes for all the investigated materials are given in Table 2, confirming

the observations from the micrographs in Figure 3 and 4. The texture strength for the recrystallized materials are also included in Table 2, showing that the texture sharpens when the softening processes becomes strongly affected by dispersoids (for more detail see [5]).

The explanation of the weaker effect on recrystallized grain structure and texture in the two-step deformed materials is believed to be a larger amount of mobile boundaries in the two-step materials resulting from the final cold rolling sequence, i.e. new mobile boundaries that are not pinned directly by dispersoids are introduced. Hence, the critical subgrain size for nucleation of recrystallization might be reached before the boundaries are pinned by dispersoids. However, the denser the dispersoid structure is the fewer nucleation sites can be activated. In the single-step materials affected by concurrent precipitation, the mobile boundaries are effectively pinned by dispersoids precipitating on the mobile boundaries and hence, the subgrain growth and nucleation of recrystallization becomes strongly retarded.

Table 2: Recrystallized grain sizes measured by PLOM and EBSD for the different material variants after final softening.

	Temp. Time [⁰C] [s]	PLOM		EBSD				
Specimen		[s]	RD	ND	Crit mis.	RD	ND	Texture strength
S-3-0	500	100	13.9	7.7	2°	10.1	5.7	4.4
S-3-0	400	10 <sup>4</sup>	151.3	32.8	2°	116.0	30.0	16.6
S-3-A	500	10	11.9	9.6	2°	8.8	6.1	2.2
S-3-A	350	10 <sup>4</sup>	89.8	21.1	2°	66.8	19.9	11.6
T-3-0-300°C	500	3x10⁵	56.6	12.9	2°	54.5	15.6	9.1
T-3-0-350°C	500	3x10⁵	38.0	10.3	2°	42.0	14.2	7.9
T-3-A-300°C	500	100	18.4	8.1	2°	12.8	7.1	3.2
T-3-A-350°C	500	100	10.3	5.8	2°	10.0	6.9	4.0

## 4. Concluding Remarks

It has been shown that the recrystallization process is strongly affected by concurrent precipitation than by dispersoids present prior to annealing. The effects of the dispersoids present prior to annealing are strongly dependent on the dispersoid density. For high densities the nucleation of recrystallization becomes pinned resulting in a coarse grained structure. The recrystallization textures are related to the Zener drag, *i.e.* the texture sharpens with increasing effect of the dispersoids on the recrystallization process.

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