Analysis of Softening Behaviour of AA1200 Alloys Using Gallium Enhanced Microscopy

H.-E. Ekström, O.V. Mishin and L. Östensson

Sapa Technology, SE-61281 Finspång, Sweden

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Abstract

The softening behaviour during annealing has been investigated in cold rolled AA1200 alloys. It is found that the evolution of boundary spacing determined using gallium enhanced microscopy gives a very good representation of the softening behaviour. Analysis of the microstructural evolution and thermoelectric and resistivity measurements reveal that iron is precipitated in the course of grain growth. It is suggested that when material is annealed at low temperatures, solute drag of iron atoms holds up the grain boundaries. After precipitation of the iron, boundaries can move again.

1. Introduction

The microstructure changes during annealing of a deformed metal are commonly described in terms of recovery and recrystallisation. In heavily deformed aluminium, the recovery process often consumes most of the energy stored during deformation [1]. It has been shown [2] that the evolution of the flow stress during recovery can be described as a sum of the friction stress due to elements in solid solution and constituent particles, σ_i , and contributions related to the dislocation density within cells, ρ_i , and from the cell/subgrain size, δ

$$\sigma(t) = \sigma_i + \alpha_1 MGb \sqrt{\rho_i(t)} + \alpha_2 MGb / \delta(t)$$
(1)

where α_1 and α_2 are constants, M is the Taylor factor, G is the shear modulus and b is the Burgers vector. From the results of Furu et al. [1] it follows that the evolution of the cell size during recovery should be both time- and temperature-dependent:

$$1/\delta(t) = 1/\delta_0 - (RT/A) \ln(1+t/\tau), \tau = CTexp((U-A/\delta_0)/RT)$$
 (2)

where δ_0 is the initial cell/subgrain size, U is the activation energy for recovery, A is proportional to the distance between the solute atoms, C is a constant and R is the universal gas constant.

This framework is used in the present work, in which a new technique called gallium enhanced microscopy (GEM) [3] is employed for quantitative characterisation of the microstructure evolution during annealing of two AA1200-type alloys. In contrast to the EBSD technique used in our previous study [4], the GEM technique allows observations of dislocation boundaries with misorientations of less than 1° [3]. The results obtained here are used for modelling of the flow stress.

2. Experimental

2.1 Materials.

A laboratory cast alloy with 0.12 wt% Si and 0.48% Fe was produced from high-purity aluminium 99.999% by casting in iron moulds. A thermomechanical treatment was designed to reduce the amounts of iron and silicon in solution to the lowest possible levels. The ingot was cold rolled from 25 mm to 12 mm thickness, heat treated at 320°C for 48 hours, cold rolled again to 9 mm thickness, heat treated for 120 hour at 200°C, and finally cold rolled 88% (sample FS-L [4]). Another sample was prepared from a commercial AA1200 alloy that had concentrations of the main alloying elements similar to those in the laboratory cast sample. A 2.8mm thick hot-rolled strip was annealed for 24 hours at 250°C to decrease the amount of iron in solution. It was then cold-rolled 90% (sample Com1200).

2.2 Back Annealing.

The annealing temperatures that enabled appropriate softening after 10 hour exposure were chosen to be 219°C for the Com1200 sample and 110°C for the FS-L sample. The samples were heated up to the selected temperature with a rate of 50°C per hour. Another series of specimens was heated up to the same temperatures, but then these specimens were quickly transferred into a different furnace and annealed at temperatures either 10°C higher or 10°C lower than the original temperatures. Following the thermal exposure all these samples were cooled in air.

2.3 Measurements and Tests.

The microstructural investigations were performed in the longitudinal section that contained the rolling and normal directions (RD-ND). One side of the 10mm long specimens was mechanically polished. The opposite side was dipped into a droplet of liquid gallium for 30 minutes. The samples were then analysed using a Philips XL30S FEGSEM in the BSE mode. These GEM images were used for measuring the boundary spacing and for evaluation of the fraction recrystallized. For some conditions, area fractions recrystallized were estimated from optical micrographs. The solute content was evaluated from resistivity data and thermoelectric power [4]. Tensile tests were performed at room temperature on specimens with the tensile axis parallel to RD.

3. Results

The microstructure of the commercial alloy annealed at 219°C is shown in Figures 1,2. In this sample, subgrains in the subsurface layer were found to be much smaller than those in the sheet centre. In the samples annealed for less than to 8.1 hours, only recovery and subgrain growth took place. Discontinuous recrystallisation occurred in the sample annealed for 21.6 hours. It is evident that recrystallized grains grow faster in the central layers than in the subsurface, Figure 2. However, very small crystallites were still present in the mid-thickness. After further heating such small crystallites were not observed.

The grain structure in the laboratory cast sample had a more streaky appearance with very long and large recrystallized grains extended along the rolling direction. Large

recrystallized grains appeared at 2% to 25% depth already during heating up to the annealing temperature, 110°C. Recrystallisation was slowest in the mid-thickness region. The GEM images from the subsurface, quarter thickness and central layers were used for determining the boundary spacing in the sheet normal direction. The mean boundary spacing for the commercial material as a whole was calculated as the arithmetic mean of the boundary spacing at three different depths, see Table 1. A comparison of the GEM data with the results of the previous EBSD-analysis [6] shows that the latter method gives larger values.



Figure 1: Microstructures at 5% depth below the surface in the Com1200 sample: (a) as cold rolled, (b) annealed at 219°C for 5.9 h (c) for 21.6h.



Figure 2: Microstructure from surface to midthickness of the Com1200 sample annealed at 219°C for 21.6h

Time at 219°C (h)	Subsurface (µm)	1/4-depth (µm)	Mid-thickness (µm)	Mean (µm)	EBSD Mid-thickness (µm)
As cold rolled	0.58	0.49	0.44	0.50	0.5
Heated to 219°C	0.74	0.63	0.66	0.68	0.8
0.51	0.76	0.69	0.69	0.72	-
2.0	0.80	0.74	0.83	0.79	1.0
5.9	0.79	0.83	0.97	0.86	-
8.1	0.83	0.86	0.98	0.89	1.3
21.6	0.81	2.03	3.04	1.96	-
36.1	7.90	4.66	5.81	6.12	-
449.1	5.98	8.91	8.51	7.80	-

Table 1. Boundary spacing along ND for the commercial AA1200 sample heat-treated at 219°C.

A somewhat different method was used for the laboratory cast alloy. In this case, the boundary spacing was determined separately for recrystallized and non-recrystallized areas, see Table 2. The fraction of recrystallized areas was also determined from the GEM images, except for the subsurface layers annealed for 0.5h and 2.2h. In this case, optical micrographs were sufficient for distinguishing between recrystallized and recovered areas.

Table 2. Boundary spacing and fraction recrystallized for different depths of the laboratory cast FS-L sample heat treated at 110°C. Fractions recrystallized were estimated either from optical micrographs (marked by asterisks) or from GEM images.

Parameter	Depth from	Heated	Time at 110°C (h)				
	surface	to 110°C	0,50	2,2	8,2	30,8	124,5
	0 – 14%	25.6*	43.2*	53.1	64.8	89.5	88.6
	14 – 28%	3.0*	9.0*	98.0*	97.5	99.7	100
Recrystallized fraction (%)	28 – 42%	0	18.1	2.1	58.6	89.4	100
	42 – 58%	0	10.1	14.0	44.4	73.1	100
	Mean	8.2	21.5	45.8	69.5	90.0	96.7
	0 – 14%	10.5	12.7	8.7	8.3	10.0	10.6
	14 – 28%	8.3	8.9	9.5	12.2	12.1	11.5
Recrystallized grain size (µm)	28 – 42%	No recr.	7.1	4.8	9.5	10.4	11.1
	42 – 58%	No recr.	11.2	8.2	12.3	10.2	23.8
	Mean	9.7	9.8	7.7	10.3	10.8	12.9
	0 – 14%	0.63	0.61	0.58	0.81	0.72	0.75
	14 – 28%	0.65	0.65	0.61	0.75	0.56	0.79
Subgrain size (µm)	28 – 42%	0.50	0.58	0.65	0.67	0.82	0.61
	42 – 58%	0.58	0.56	0.58	0.62	0.54	0.72
	Mean	0.57	0.58	0.60	0.71	0.66	0.72

Figure 3 shows the variation of the 0.2% proof stress in the commercial AA1200 alloy during annealing at three different temperatures. Equation (2) fitted to the data, for which the proof stress was greater than 40MPa, gives U=186kJ/mole. Furthermore, the concentration of iron in solid solution appears to be dependent on the boundary spacing (see Figure 4).

For the laboratory cast alloy discontinuous recrystallisation commenced during heating at a very low temperature. Its activation energy for the softening was found to be low, 135 kJ/mole [4].





Figure 3: Tensile data for the Com1200 sample. The curves represent a fit of Equation 2 to the data points (see text).

Figure 4: Concentration of iron in solid solution versus boundary spacing δ for the Com1200 sample annealed at 219°C.

4. Discussion

Equation 1 that describes the evolution of the flow stress during recovery gives a good fit to the tensile test data for $R_{p0.2}$ >40MPa for the commercial AA1200 alloy. This implies that recovery is the dominant process during the softening of this material and it is responsible for more than half of the total reduction in the yield stress.

The activation energy for recovery in the cold rolled FS-L alloy (135 kJ/mole) having a very low amount of iron in solution, 10 ppm [4], is close to that for self-diffusion in aluminium, 121 kJ/mole [5]. On the other hand, the activation energy for the commercial AA1200 alloy with 30ppm of iron in solution is much higher, 186 kJ/mole. This value is close to that obtained by Furu et al. [1] for cold-rolled commercial purity aluminium, 175 kJ/mole. It has been suggested that such a high energy is due to solute drag of iron that controls boundary migration in aluminium [6]. As follows from Figure 4, the amount of iron in solution decreases when the boundary spacing increases, which suggests precipitation of iron in the course of subgrain and grain growth. If subgrains and grains grew uniformly in all directions, the amount of iron in solution would be expected to be proportional to $1/\delta^3$. However, Figure 4 clearly demonstrates a $1/\delta^2$ -dependence, which implies growth only in two directions.

To obtain the relationship between the flow stress and the boundary spacing in the commercial AA1200 alloy, the proof stress can be plotted versus $1/\delta$ (see Figure 5). The data points fall close to a straight line expressed by the following function:

$$R_{p0,2} = 24.5 \text{ MPa} + 60.6/\delta$$
 (3)

A comparison with Equation (1) suggests that the contribution from internal dislocations to the flow stress is negligible in these heavily deformed samples. Assuming M=3, G=26 MPa and b=0.286 nm, we obtain α =2.7, which is a reasonable value. Similar results have been reported for the AA8006 alloy [7].

The experimental proof stresses in the cold rolled and annealed conditions of the Com1200 sample are further compared with the proof stresses calculated from Equation 3 (see Figure 6). The results for the FS-L sample are also given in Figure 6. For the FS-L sample Equation 1 was modified to take into account different boundary spacings in the recrystallized and non-recrystallized areas:

$$R_{p0.2} = 24.5 \text{ MPa} + 60.6[(1-X)/\delta + X/D]$$
 (4)

where X is fraction recrystallized, δ is the subgrain size and D is the mean size of recrystallized grains.

It appears that Equation 4 gives a good description of the softening behaviour of the laboratory cast alloy (see Figure 6), especially considering uncertainties in the evaluation of the recrystallized fractions.



Figure 5: 0.2% proof stress versus the inverse of the boundary spacing for the Com1200 sample.



Figure 6: Experimental and simulated proof stress in the as cold rolled conditions and after annealing either at 219°C (Com1200) or at 110°C (FS-L).

5. Concluding Remarks

The GEM technique allows statistical analysis of the microstructure over large sample areas and measurements of the boundary spacing with relatively little effort. The evolution of the boundary spacing determined from the GEM images very well describes the evolution during softening of the AA1200 alloys used in this experiment. Results obtained suggest that the assumption of negligible influence of the internal dislocations on the flow stress is reasonable. The softening of the commercial alloy seems to be mostly due to recovery. The amount of iron in solution decreases as a function of $1/\delta^2$. It is suggested that during annealing at low temperatures the boundary mobility is restricted until iron at the boundaries starts to precipitate.

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