MICROSTRUCTURAL CHANGES DURING THE ANNEALING OF AA 8011 FOIL ROLLED FROM CAST STRIP

Anders Oscarsson[1], Börje Lehtinen[1], Bevis Hutchinson[1], Hans-Erik Ekström[2], Peter Bate[3], Lennart Häggström[4] and Amani M. Ghandour[4]
1 Swedish Institute for Metals Research
2 Gränges Technology Centre, Sweden
3 IRC, University of Birmingham, UK
4 University of Uppsala, Sweden

Abstract

Strip-cast aluminium alloy 8011 (0.67 % Fe, 0.63 % Si) has been cold rolled to a range of reductions in an industrial scale mill, and then subjected to a batch annealing process at a series of different temperatures. Mechanical properties and optical metallography show that there is a transition from normal discontinuous recrystallisation towards continuous recrystallisation as the level of deformation increases. The cold rolled state is characterised by dispersions of primary β–AlFeSi intermetallic particles and elemental silicon dispersed in a matrix having a high supersaturation of iron and a fine subgrain size. Following heavy rolling reductions this structure contains a high frequency of boundaries having large misorientations ($\omega > 15°$), as determined by application of the electron backscattering pattern (EBSP) technique.

Changes in this microstructure on annealing at successively higher temperatures have been monitored using SEM/BSE imaging to reveal the grain structures, as well as by STEM for the analysis of small particles and Mössbauer spectroscopy to measure the content of iron in solid solution. A computer model of coarsening for an analogous 2-dimensional structure has been constructed to help rationalise the annealing behaviour of the alloy. The model takes into account the effects of varying grain boundary misorientation distributions.

Introduction

Good combinations of strength and ductility can be achieved in aluminium foils if the substructure undergoes a continuous coarsening during the final annealing after cold rolling. This gradual coarsening has sometimes been called continuous recrystallisation (1). Occurence of large recrystallised grains will severely diminish formability and has to be avoided in practice. This study is a part of a larger investigation into the microstructures, textures and properties of different aluminium sheet alloys produced by strip casting, cold rolling and subsequent annealing. The aim of the present paper is to characterise the microstructural changes during the annealing of AA8011 which show a clear tendency for continuous recrystallisation after high cold rolling reductions.

Experimental

The material used was commercially processed AA 8011 containing 0.67 % Fe and 0.63 % Si. It was strip-cast to a thickness of 6 mm then cold rolled in a number of passes to 0.11 mm. Sheets of different thicknesses were annealed at different temperatures for two hours with a heating and cooling rate of 50 K/h. Examination was carried out using tensile testing, optical, scanning and transmission electron microscopy. STEM and Mössbauer spectroscopy were carried out on samples cold rolled to 0.11 mm thickness then heated with 50 K/h to different temperatures followed by water quenching. Misorientation distributions of (sub)grain boundaries after different degrees of cold rolling were measured on recovered samples using the electron back scattering pattern (EBSP) technique. These were compared with results from X-ray texture (ODF) analysis measured from the identical specimen. Computer modelling of coarsening for analogous 2-dimensional structures has been applied using varying grain boundary misorientation distributions both with and without pinning by second phase particles.

Results

Softening curves showing the effect of annealing after different degrees of cold rolling reduction are presented in figure 1. A transition in behaviour was observed at about 95 % reduction. For lower reductions the softening is moderate for temperatures up to 280°C followed by a sudden decrease in strength. At higher reductions the softening is more rapid at temperatures between 200 - 270°C but no sudden drop in strength occurs at any temperature and this results in a much more continuous softening process compared to the one for lower reductions.

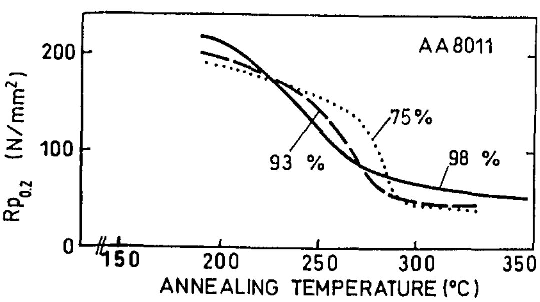


Figure 1
Variation of yield stress with annealing temperature for different cold rolling reductions.

Figures 2 and 3 show the grain structure evolution after low and high cold rolling reductions respectively. After low cold rolling reduction the recrystallisation proceeds in a normal discontinuous manner with some overall coarsening of subgrains during recovery followed by the growth of a few isolated grains into a relatively static matrix. The behaviour after high cold rolling reductions is quite different and involves only a gradual coarsening of crystallites even at higher temperatures, Fig. 3.

The cold rolled structure contains a great number of small, 0.1 - 1.0 μm, β-Al_5FeSi particles which precipitated during the strip casting process. Additionally, even smaller elemental Si-particles are precipitated in this stage and these occur often in agglomerates. During heating the β-Al_5FeSi particles remain apparently unchanged while the Si-particles coarsen. No fine Fe-rich particles (< 50 nm) were found despite persistent searching in the sample after heating to 280°C.

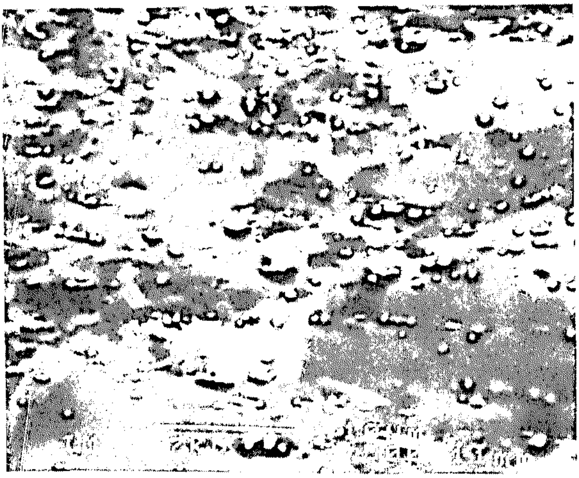
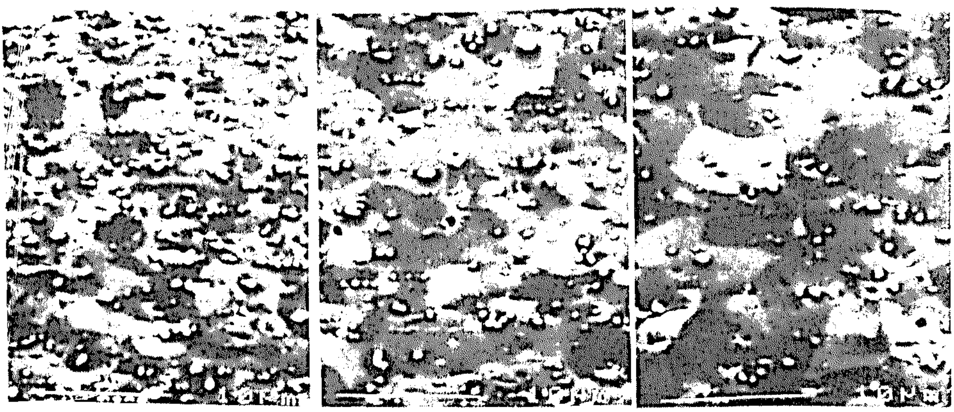


Figure 2
SEM/BSE micrograph showing discontinuous recrystallisation after 75 % cold rolling reduction and annealing to 290°C.

10 μm



a) 280°C b) 300°C c) 360°C

Figure 3 SEM/BSE micrographs showing continuous recrystallisation after 98 % cold rolling reduction and heating to various temperatures.

A few small Fe-rich particles were observed after heating to 300°C and they became more numerous at 320°C, see example in figure 4. Their sizes were 10 - 40 nm and analysis of their compositions suggested that most of them are either Al_6Fe or Al_3Fe types as only one out of fifteen analysed particles contained a small amount of Si. Mössbauer spectroscopy was applied to the 0.11 mm sheet after cold rolling and annealing to different temperatures. Figure 5 shows an example of such a spectrum together with calculated contents of Fe in solid solution. The value for the cold rolled sample is higher than previously reported (2), which may be explained by the fact that a different techique was used (wet chemical analysis by butanol dissolution/ atomic absorption). This discrepancy between different methods has been reported elsewhere (3). The conclusion drawn from the Mössbauer measurements is that very little of the Fe originally in solid solution precipitates during heating in this alloy which agrees well with STEM observations.

particle	Fe atom%	Al
1	27	bal.
2	17	bal.
3	17	bal.
4	13	bal.
5	16	bal.

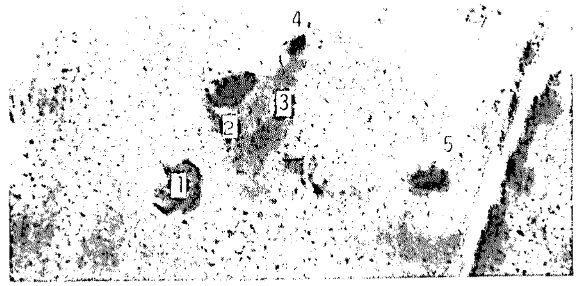


Figure 4 STEM micrograph showing small Fe-rich particles in the 98% cold rolled sample after heating to 320°C. Chemical compositions for each particle are given.

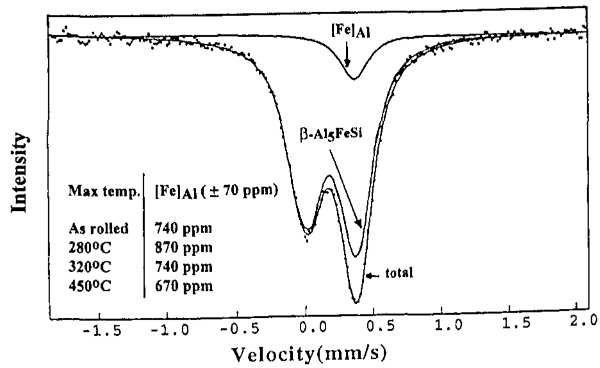
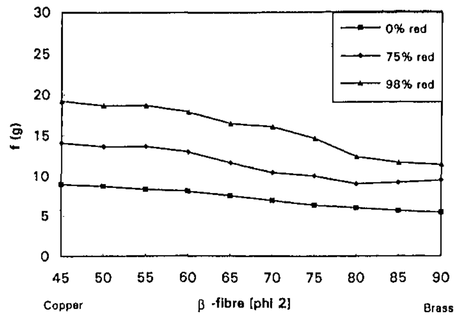


Figure 5 Example of Mössbauer spectrum of band heated to 450°C. The asymmetric doublet represents β-Al_5FeSi and the singlet represents the Fe solved in Al. Also given in the figure is the analysed Fe content in solid solution after heating to various temperatures.

The cold rolled sheets show the usual fcc rolling texture. The β-fibre intensities plotted in figure 6 show a rather well developed texture immediately after strip casting which can be attributed to the plastic deformation which takes place in the roll gap following solidification. An overall sharpening of the texture occurs during cold rolling but the shape of the β-fibre remains roughly the same. Annealing textures in the fully softened condition are markedly dependent on the prior rolling reduction. The cube recrystallisation texture dominates for low reductions but some β-fibre is also present. Following high reductions the β-fibre dominates completely, the Cu and R-components being especially strong. However, the texture is not invariant during annealing of the 98 % rolled sheet as seen in figure 7. There is a strengthening of the β-fibre between the Cu and R components at the expense of the Bs component.

The EBSP method was used to measure the distribution of minimum misorientation angles for grain or subgrain boundaries. Measurements were made on a central section parallel to the rolling plane along tracks parallel to RD and TD, corresponding to 350 boundaries per specimen. Figure 8 shows a simplified representation of these real misorientation distributions together with calculated ones from the X-ray ODF results for the same sample assuming that no spatial correlation of misorientations exists. For the uncorrelated misorientation distributions the



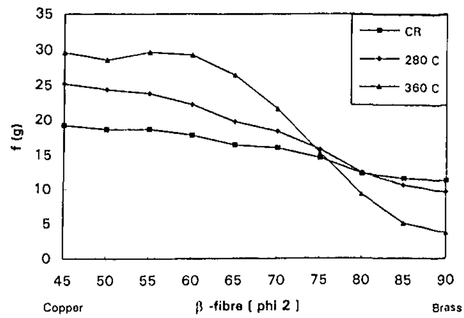
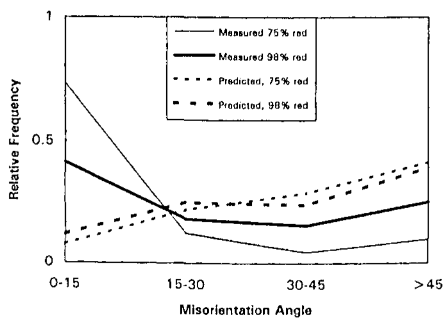


Figure 6 Orientation densities along the β-fibre after various stages of cold rolling reduction.

Figure 7 Orientation distributions along the β-fibre after 98 % cold rolling reduction and annealing to different temperatures.

proportion of high angle boundaries decreases due to the sharpening of the texture with increasing rolling reduction. The experimental misorientation distributions reveal an opposite trend with an increase in the proportion of high angle misorientations following heavier deformation, i.e. fewer subboundaries and more high angle boundaries. However, some degree of spatial correlation still persists even after 98 % reduction since the measured and predicted distributions are not identical.



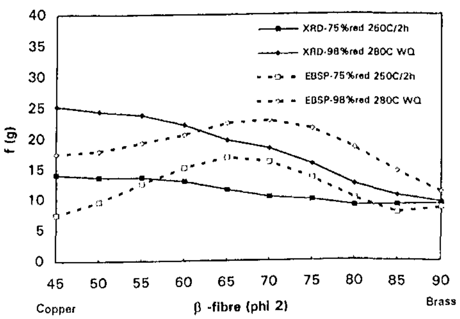


Figure 8 Misorientation distributions measured by EBSP (solid lines) and predicted from X-ray ODF results (dotted lines) after various stages of cold rolling reduction.

Figure 9 Orientation distributions along the β-fibre from X-ray ODF results (solid lines) and EBSP ODF results (dotted lines).

ODFs were also calculated from the data of the EBSP measurements. The results showed almost identical positions for the β-fibre but with different orientation densities along it compared to XRD measurements as seen in figure 9. The EBSP results show an underestimation of the Cu component and overestimation of the fibre between the R and Bs components. One explanation for the difference may be that the size of each grain has not been considered when calculating ODFs from the EBSP measurements. Another possibility is of course that the diffraction patterns of certain orientations are more difficult to recognise due to their smaller crystallite size or higher density of dislocations.

Discussion

Observations of microstructure and texture development as well as mechanical properties have shown that a change occurs in the nature of recrystallisation process as the degree of cold rolling is increased. At low reductions the recrystallisation proceeds abruptly in a discontinuous manner resulting in large grains and a dominating cube texture. At high reductions the process is more gradual, leading to a recrystallised texture similar to the deformed one, a behaviour which has often been termed continuous recrystallisation.

The phenomenon of continuous recrystallisation has often been associated with pinning of subboundaries by particles and their gradual release due to particle coarsening (1). A well developed subgrain structure is developed for both low and high rolling reductions after annealing at 250°C. The present alloy contains β–Al_5FeSi particles and some elemental Si after strip casting. There is some further precipitation of small Fe-rich particles and Si on heating but only a negligible amount appears at temperatures below 320°C. The Mössbauer results also confirm that a surprisingly high level of iron remains in solid solution. Furthermore, there is no obvious reason why the level of cold reduction should markedly affect the precipitation behaviour, and so the model based on particle coarsening cannot explain the transition from discontinuous to continuous recrystallisation satisfactorily.

Another proposed explanation (2) is that the recrystallisation behaviour is governed by the relative proportions of mobile and immobile boundaries present in the deformed and recovered substructure. Hillert (4) has shown that abnormal growth cannot occur when all boundaries have equal mobility if pinning particles are absent; growth will then tend towards continuous behaviour. Low angle boundaries (< 15°) and certain twin related boundaries are known to have very low mobility compared to high angle ones. It would then be expected that continuous recrystallisation would occur if all boundaries are either of low or high angle character.

The original grains are 10 - 15 µm across and they become flattened to the thickness of a single crystallite at about 95 % reduction (2). This implies a larger proportion of high angle boundaries at about this stage. Measurements in figure 8 have shown that the proportion of high angle boundaries (>15°) in the rolling plane increases from 27 % to 59 % when increasing the rolling reduction from 75 % to 98 %. A greater proportion of the boundary network is therefore expected to be mobile following heavy reductions. We believe this is the reason for the change in recrystallisation behaviour and one which we have sought to verify by computer modelling.

Computer simulations have been carried out using a 2-dimensional network in the manner described by Frost et al (5) and Humphreys (6). The assumed energies and mobilities as functions of misorientations are shown in figure 10. Computation has been limited to a starting texture with three different Gaussian spreads, (5°, 10° and 30°) around an ideal orientation. The different spreads resulted in initial misorientation distributions as seen in figure 11. The misorientation distribution are quite widely spread and tend to show a broad maximum at an intermediate value of ω. Although these distributions differ considerably in character from the ones measured by EBSP (Fig. 8), they do reproduce approximately the proportions of low and high angle grain boundaries. For example, in the model distribution having 10° Gaussian spread, the frequency of low angle boundaries ($\omega < 15°$) is 61 % while the measured value for the sheet cold rolled 75 % is 73 %. Increased cold reduction is equivalent to an increase in Gaussian spread.

Both the structure with only low angle boundaries (5^{o} Gaussian spread) and the one dominated by high angle boundaries (30^{o} Gaussian spread) grow in a continuous way as seen in figure 12. These observations are in agreement with Hillert's theory which predicts that growth will be normal if all boundaries have similar energy and mobility. The grain structures having only small angle boundaries grew very slowly in the model. In reality, a structure containing only low angle boundaries will never exist in such alloys even after low reductions due to the presence of pre-existing grain boundaries.

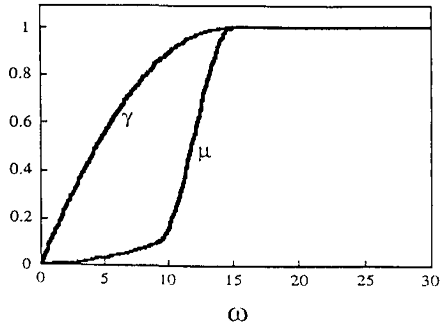
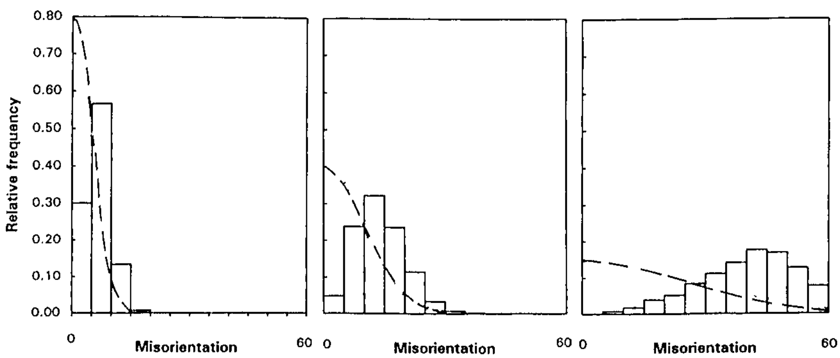


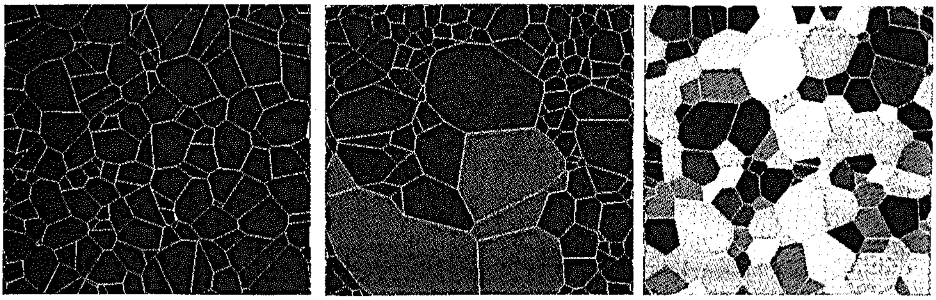
Figure 10
Assumed specific boundary energy (γ) and mobility (μ) as functions of misorientation angle (ω).

Calculations based on the model having 10° and 30° Gaussian spreads agree well in several regards with the behaviour of the 75 % and 98 % cold rolled sheets respectively. In the former case there is first some general coarsening, and then a small number of grains grow abnormally. These grains are ones of high misorientation and can be considered equivalent to the cube oriented grains which grow discontinuously in the 75 % cold rolled sheet (i.e. widely scattered away from the β-fibre). With the larger Gaussian spread of 30°, there is little change in the texture which develops in the model, and the growth is of a normal type, the grains having an almost constant relative size distribution. Such behaviour accords well with the 98 % cold rolled sheet which has a large proportion of high angle boundaries (60 % of boundaries have $\omega > 15°$). Growth occurs in a continuous manner and the β-fibre texture is retained at all annealing temperatures.



a) 5° b) 10° c) 30°

Figure 11 Initial misorientation distributions for simulated structures with different Gaussian spreads shown by the dashed lines.



a) 5° Gaussian spread b) 10° Gaussian spread c) 30° Gaussian spread

Figure 12 Simulated microstructures for different initial proportions of high angle boundaries after the number of grains has reduced from 1950 to 200.

Conclusion

Experimental measurements and computer modelling show that the transition from discontinuous to continuous recrystallisation in AA 8011 with increasing prior cold rolling reduction can be attributed to the change in misorientation distribution of the (sub)grain boundaries. Precipitated phases do not play an important role.

Acknowledgement

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References

1. E. Hornbogen, Met. Trans., 1979, 10A, 947.

2. A. Oscarsson, H.-E. Ekström, W. B. Hutchinson, Recrystallisation '92, Materials Science Forum, 1993, 113-115, 177.

3. A. Oscarsson et.al. Z Metallkd, 1988, 79, 600.

4. M. Hillert, Acta Met:, 1965, 13, 227.

5. H. J. Frost et al., Scripta Met., 1988, 22, 65.

6. F. J. Humphreys, Recrystallisation '92, Materials Science Forum, 1993, 113-115, 329.