EFFECT OF STRAIN LEVEL ON RECRYSTALLISATION RESPONSE OF AA8006 AND AA8011 THIN STRIPS

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Abstract

Aluminium sheets manufactured from twin-roll continuous cast AA 8006 and AA 8011 alloys are used in heat exchangers as fins of complex configuration. The strips are delivered in the quarter-hard H22 and soft O conditions obtained by annealing after cold rolling to the final thickness. Finstock materials must exhibit high formability combined with good strength and detailed understanding of the structure evolution during thermo-mechanical processing is therefore necessary in order to obtain optimum sheet properties. Previous investigation results indicated that the increase of rolling reduction applied prior to the final annealing results in an increase of the yield strength $R_{p0.2}$ of annealed sheets. The paper presents the results of a research aimed at understanding the physical-metallurgical causes of this effect. The influence of further factors affecting deformation restoration during annealing such as composition, second phase dispersion and heating rate are discussed too.

1. INTRODUCTION

Aluminium sheets produced from twin-roll cast materials are largely used in heat exchangers as fins of complex design. The materials for such applications (called finstocks) must exhibit high formability combined with good strength. Among the most frequently used finstock materials are the aluminium alloys AA8006 (Al-Fe-Mn-Si) and AA8011 (Al-Fe-Si). Finstock sheets are delivered in the quarter-hard H22 and soft O conditions obtained by annealing after cold rolling. Our previous works [1,2] indicated that, when sheets of different thickness are annealed at the same conditions, thinner sheets (0,10 mm gauge) are less recrystallised and exhibit higher yield stress than thicker sheets (0,17 to 0,28 mm gauge). This surprising phenomenon was observed AA8006 and AA8011 alloys subjected to different thermo-mechanical treatment prior to the final annealing.

It is normally found that an increasing plastic strain promotes recrystallisation on subsequent annealing. However, there are situations where a highly stained metal can resist recrystallisation [3]. In metals deformed to very large strains at room temperature a fine-grained structure developed by accumulation of high angle grain boundaries (HAGB) during straining was observed (see references in [3]). It was found that at a critical strain the HAGB spacing is reduced to the dimension of the subgrain size, the HAGBs begin to touch, and a new microstructure of mainly high angle boundaries is effectively formed. This process is usually classified as "continuous recrystallisation". Either continuous recrystallisation can occur during deformation or when a low temperature recovery anneal is subsequently given.

The paper presents the results of an investigation aimed at studying the stability of AA8006 and AA8011 microstructures during annealing after large strains, especially the effect of strain level on microstructure capability to resist recrystallisation. The effect of some further factors affecting deformation restoration kinetics such as alloy composition, second phase dispersion and heating rate was also evaluated.
2. EXPERIMENTAL

Thin strips prepared from twin-roll continuously (TRC) cast strips (provided by HZB) were used as experimental material. Materials of different compositions: two AA8011 alloys (Al-Fe-Si) differing by the ratio of iron and silicon content and a AA8006 alloy (Al-Fe-Mn), were investigated. Materials with different particle dispersion level, prepared either using homogenisation treatment at various conditions or without homogenisation, were compared. Material composition and pre-treatment are summarised in Tab. 1. In order to assess the impact of strain level, sheets of thickness in the range from 0,09 to 0,28 mm were prepared by cold rolling reductions from 96 to 99%. The corresponding strains \( \varepsilon \) are in the range from 3,9 to 5,3, i.e. they are very large and can be classified as severe plastic deformation.

Annealing tests were carried out using both high heating rate/short duration treatments and slow heating rate/long duration treatments (simulating industrial procedure). Isochronal annealing treatments of high heating rate and duration from 1 to 16 minutes were carried out using salt bath pre-heated to temperatures 280°C (materials A, B and C) and 300°C (material D). Samples for mechanical tests were prepared by annealing for 5 minutes in a furnace pre-heated to different temperatures in order to prepare both partially (270-300°C) and fully recrystallised (350-380°C) microstructures. High heating rate and short annealing time are not feasible in real industrial conditions unless the sheet producer is equipped with special facility for continuous annealing where high heating rates and short annealing times can be achieved. Usually, the annealing of thin sheets in industrial conditions is carried out in coils large up to 2 m in diameter, which are put to the furnace and the temperature is gradually increased to the required value. As the heating rate of the outer and inner coil layers are very different, the coils are held at temperature for long time so as to ensure the appropriate annealing duration for the whole coil volume. In order to simulate the industrial procedure slow heating rate/long time annealing tests were performed in furnaces with gradual temperature increase during 10 hours and isothermal annealing for 8 hours. Several temperatures from 250 to 350°C were used for AA8011 alloy strips, whereas the AA8006 samples were annealed at 270°C and 380°C.

### Table 1.

<table>
<thead>
<tr>
<th>Material</th>
<th>Alloy</th>
<th>Fe</th>
<th>Si</th>
<th>Mn</th>
<th>All</th>
<th>Fe/Si</th>
<th>Homogen.</th>
<th>V_{V/r} [1/mm²]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>[wt. %]</td>
<td>temper. [°C]</td>
<td>8 h.</td>
<td>18 h.</td>
</tr>
<tr>
<td>A</td>
<td>AA8011</td>
<td>0.87</td>
<td>0.43</td>
<td>0.04</td>
<td>1.34</td>
<td>2.0</td>
<td>no</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>AA8011</td>
<td>0.68</td>
<td>0.67</td>
<td>0.01</td>
<td>1.36</td>
<td>1.0</td>
<td>no</td>
<td>-</td>
</tr>
<tr>
<td>C</td>
<td>AA8011</td>
<td>0.72</td>
<td>0.67</td>
<td>0.01</td>
<td>1.40</td>
<td>1.1</td>
<td>550</td>
<td>-</td>
</tr>
<tr>
<td>E</td>
<td>AA8006</td>
<td>1.30</td>
<td>0.18</td>
<td>0.42</td>
<td>1.90</td>
<td>7.2</td>
<td>550</td>
<td>139</td>
</tr>
<tr>
<td>F</td>
<td>AA8006</td>
<td>1.50</td>
<td>0.16</td>
<td>0.40</td>
<td>2.06</td>
<td>9.4</td>
<td>580</td>
<td>114</td>
</tr>
<tr>
<td>D</td>
<td>AA8006</td>
<td>1.50</td>
<td>0.16</td>
<td>0.40</td>
<td>2.06</td>
<td>9.4</td>
<td>580 (ind.)</td>
<td>-</td>
</tr>
</tbody>
</table>

For the microstructural characterisation of the sheets optical microscopy and image analysis were employed. The particle dispersion level of materials A, B and C was assessed only qualitatively and it was found that it is significantly lower in the homogenised material. Our investigations [4] indicated that the change of particle dispersion is due to intensive particle dissolution and coarsening occurring during homogenisation. The particle dispersion level in samples F, E and D (homogenised at different temperatures and durations - Tab. 1) was evaluated quantitatively and the results are in Tab. 1. The grain structure was observed in...
3. RESULTS

3.1 Short Annealing after Quick Heating

In the as-deformed state a typical banded microstructure, which is characteristic of rolled sheets, was observed in all materials. Observations in polarised light showed that after annealing the specimens contained markedly differing recrystallised fractions. As the level of deformation restoration (recrystallised fraction) is characterised also by sample hardness (decreases with increasing restoration level), the hardness HV0,05 of all annealed specimens was measured. Fig. 1 shows the results for materials AA8011 in terms of the evolution of hardness with increasing annealing time, the results for material D (AA8006) are in Fig. 2.

When comparing AA8011 strips annealed for the same time, samples prepared from material A were the less recrystallised, samples C were the more recrystallised (after 4 minutes almost fully), samples B were between these two levels. When comparing samples of the same material rolled to different strains and annealed for the same time it was observed (Fig.1) that the more strained samples were recrystallised to lower extent than the less strained ones. This was observed in materials A and C but not for material B. In material B the recrystallisation rate does not depend on the strain imposed before annealing. The same effect of strain on the restoration kinetics was observed also in material D sheets.

With increasing annealing time, the recrystallised fraction increased in all samples. In all samples A only a few new recrystallised grains were found after annealing for 16 minutes. Samples B contained a recrystallised fraction smaller than 10% after 16 minutes anneal. Fully recrystallised structure was observed in both C sheets annealed for 16 minutes at 280°C. Only a small (less than 10%) non-recrystallised fraction remained in samples D annealed for 16 minutes at 300°C.
Fig. 3 shows the change of yield stress $R_{p0.2}$ in materials AA811 after isothermal annealing for 5 minutes at 300°C (270°C for samples C) and at 380°C (350°C for samples C). The yield stress of the AA8006 samples as a function of annealing temperature for the same short annealing regime as above is plotted in Fig. 4. The microstructure evaluation of the annealed samples in polarised light (optical microscopy) showed that the level of restoration of the deformed microstructure is correlated with yield stress: higher $R_{p0.2}$ values correspond to lower recrystallised fractions.
Fig. 3. Yield stress $R_{p0.2}$ of AA8011 samples as a function of annealing temperature $T$ (a) and grain structures corresponding to different values of $R_{p0.2}$ (b,c).

b) B, $\varepsilon = 4.4$; annealed at 300°C

c) C, $\varepsilon = 4.7$; annealed at 270°C

Fig. 4. Yield stress $R_{p0.2}$ of AA8006 samples as a function of annealing temperature $T$ (a).

b) D, $\varepsilon = 4.6$; annealed at 300°C

c) C, $\varepsilon = 4.7$; annealed at 270°C
function of annealing temperature $T$ (a) and grain structures corresponding to different values of $R_{p0.2}$ (b,c).

c) D, $\varepsilon = 3.9$; annealed at 300°C
3.2 Long Annealing after Slow Heating (Industrial)

The variation in yield stress of AA8011 sheets, cold rolled to strains $\varepsilon$ in the range from 4 to 5.3 and then annealed according to an industrial type regime at four different temperatures, as a function of temperature is given in Fig. 5. The variation in yield stress of AA8006 sheets, rolled to $\varepsilon$ from 3 to 4.6 and annealed at two temperatures, as a function of the level of particle dispersion $V_p/r$ is in Fig. 6.
As in the case of short annealing regimes, it was found that the yield strength of annealed AA8011 sheets is affected by alloy composition, particle dispersion level (homogenised versus non-homogenised samples) and cold rolling strain. After annealing at the same temperature the samples of material A have the highest $R_{p0.2}$ values, whereas the C samples have the lowest, the yield stress of samples B is between (Fig. 5a). For the low-temperature anneal, also the heating rate and duration influence significantly the yield strength of the alloys (Fig. 5b).

A significant effect of both particle dispersion level and strain $\varepsilon$ on sheet recrystallisation response (expressed by yield stress differences) was observed also in AA8006 sheets (Fig. 6).

4. DISCUSSION

The kinetics of restoration of the deformed metal microstructure during annealing is affected mainly by: 1) the stored deformation energy, which provides the driving force for the restoration phenomena (recovery, recrystallisation), 2) the solid solution content, 3) the second-phase (particle) dispersion level [5]. The initial (thin sheet) states of the materials under investigation differed both by solute content and particle dispersion. Electrical conductivity measurements indicated that the AA8011 materials have different concentrations of iron and silicon in super-saturated solid solution. The conductivity $\kappa$ of materials A and B was 31.6 and 32.2 m$\Omega$.mm$^2$, respectively, the value of $\kappa$ of the homogenised material C with the same composition as B was 32.8. These results indicate that in material A the solute concentration is the highest, in material C – the lowest. The value of $\kappa$ for AA8006 materials D, E and F are 30, 31.8 and 31, respectively, which is indicative for decreasing solute content in the order E, F and D. The particle dispersion level of the materials was also significantly different (Tab. 1 and text in §2.1). As in TRC sheets cold rolling does not change neither solid solution content nor particle size, the same differences exist between the as-rolled thin sheets.

Solute atoms have enormous effect on subgrain and grain boundary migration. By building an atmosphere at moving boundaries, solute atoms can exert a drag effect on them. The drag effect depends on the driving force and solute concentration: at higher driving forces and low concentrations there is a transition to a high velocity regime in which boundary velocity does not depend on solute content. At high concentrations the mobility decreases with increasing solute content. In our Al alloys Fe, Mn and Si atoms can be in solide solution and they have strongly different drag effect due to their different diffusion rate. Fe and Mn have low diffusion rate, Si mobility is higher. The drag effect of solute elements can influence both dynamic restoration occurring during cold rolling and the processes at low temperature anneal. Solute drag becomes less effective at high temperatures [5]. The dramatic effect of iron in solute solution on the softening response of Al-Fe-Si alloys was clearly demonstrated.

<table>
<thead>
<tr>
<th>Particle Dispersion Level $V_v/r$ [1/mm$^2$]</th>
<th>$R_{p0.2}$ [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>4.6</td>
</tr>
<tr>
<td>60</td>
<td>4.0</td>
</tr>
<tr>
<td>80</td>
<td>3.6</td>
</tr>
</tbody>
</table>

Table 1: Yield stress $R_{p0.2}$ of AA8006 sheets annealed according to industrial type regime as a function of particle dispersion level $V_v/r$ (materials homogenised at different conditions).
by Furu *et al.* in [6]. The authors showed that by increasing Fe solid solution content by 0.01 wt% the recrystallisation is significantly slowed (recrystallisation curves are shifted in time an order of magnitude). They found that the effect of increased Fe solute content is even stronger on the recovery part of the softening, especially at lower anneal temperatures. This strong effect was ascribed to the precipitation of Fe-bearing particles at temperatures < 375°C.

Particles have also an important influence on recovery and recrystallisation: large (>1μm) particles may provide sites for originating recrystallisation (by the mechanism of Particle Stimulated Nucleation - PSN), while small and closely spaced particles tend to pin both subgrain and grain boundaries.

The Al thin sheets subjected to different anneal regimes have significantly different microstructures and it is thus clear that the observed differences in the deformation restoration are due to differences in the effect of above cited factors. When comparing A8011 samples, regardless of the magnitude of strain ε induced by cold rolling, it is evident that the deformation recovery is strongly affected by solute content and particle dispersion. In all annealing experiments (Fig. 1, 3 and 5) samples A exhibit the highest strength (HV or $R_{p0.2}$), which means that the restoration phenomena are the most inhibited in the material with the highest solute content and higher particle dispersion level. When comparing the samples with the same composition, but differing in solute content and particle dispersion due to homogenisation (samples B and C), the effect of solute atoms and particle size and spacing is even more pronounced. The higher fraction of large particles in samples C promotes recrystallisation (PSN mechanism), i.e. it can occur at lower temperatures (fig. 3 and 5) and needs less time to be completed (Fig.1). The inhibiting effect of solute atoms is, as expected, higher at lower temperatures (Fig. 3), while at higher temperatures (380°C and more) almost all materials are fully recrystallised after for 5 minutes anneal. Nevertheless, the differences in solute content and particle dispersion affect grain size and homogeneity: for example - very large surface grains are observed in samples B but not in samples A and C.

The results of conductivity measurements and our previous works [4] clearly showed that the solid solution super-saturation of as-cast AA8006 strips is significantly higher than that of A8011 materials (κ = 24,1 m/Ω.mm² for AA8006 alloy versus more than 31,5 m/Ω.mm² for both A8011 alloys under investigation). Besides Fe and Si, AA8006 twin-roll cast alloys can have also Mn atoms in solid solution. Due to the high solute content substantially inhibiting recrystallisation, the insertion of a homogenisation treatment in the processing procedure of AA8006 thin sheets is always necessary. Different solute concentrations and particle dispersions can be obtained by varying homogenisation parameters as in the case of materials D, E and F. The results of restoration response of these materials during industrial type annealing summarised in Fig. 6 clearly show that effect of solid solution content and second phase particle dispersion on the kinetics of recovery and recrystallisation has the same character as in A8011 alloys. In AA8006 sheets the fraction of large particles capable of originating recrystallisation by PSN is noticeable higher than in A8011 non-homogenised materials, thus the contribution of this mechanism to softening is larger. From Fig. 6 is evident that when particle dispersion level increases, which is connected with a decrease in the fraction of large particles, the yield stress of the annealed samples increases too, i.e. the recrystallised fraction for a given temperature and strain level decreases. Serrated flow was observed during tensile test of AA8006 samples annealed at 300°C but not at 380°C. As serrated flow is ascribed to the effect of solute atmosphere drag on mobile dislocation and their release at a critical local strain, this phenomenon indicates that the solute content in the samples annealed at low temperature (long term anneal) is higher than in the samples annealed at 380°C. The effect of precipitation and its interaction with the restoration processes need further investigation.
One of the most important factors affecting restoration kinetics during annealing of sheets is the level of plastic strain $\varepsilon$ induced by cold rolling. When comparing the restoration response of the sheets prepared from the same material by rolling to different strains (Fig. 1 to 6) it can be seen that in the majority of cases more strained samples resist more effectively deformation restoration. Exceptions of this trend are the samples prepared from material B.

Our previous investigations [4] indicated that the microstructure of thin AA8006 and AA8011 sheets in the as-rolled condition is comprised of well-developed subgrains with very low dislocation density inside the grains. A relationship exists between the flow (yield) stress $\sigma$ of a material and its subgrain structure (subgrain size $D$) expressed by the equation

$$\sigma = \sigma_0 + c_3 D^{-m}. \quad (1)$$

$m = 1, 1/2$ or something in between [5]. As it is reasonable to suppose that that effects on flow stress of other factors such as solute content and particle dispersion in samples prepared from the same initial sheet (Tab. 2) are the same ($\sigma_0$ and $c_3$ are equal), according to equation 1 the subgrain size in the as-rolled samples is noticeably affected by cold rolling.

Table 2.
Tensile properties of as-rolled sheets.

<table>
<thead>
<tr>
<th>Material</th>
<th>$R_{p0.2}$ [MPa]</th>
<th>$R_m$ [MPa]</th>
<th>$A_{50}$ [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>5,3 4,4-4,7 4,0</td>
<td>5,3 4,4-4,7 4,0</td>
<td>5,3 4,4-4,7 4,0</td>
</tr>
<tr>
<td>A</td>
<td>241 228 217</td>
<td>270 252 247</td>
<td>6,2 8,5 7,1</td>
</tr>
<tr>
<td>B</td>
<td>206 195 -</td>
<td>261 251 -</td>
<td>5,1 8,3 -</td>
</tr>
<tr>
<td>C</td>
<td>163 170 -</td>
<td>206 206 -</td>
<td>4,9 5,2 -</td>
</tr>
<tr>
<td>D</td>
<td>-    217 193</td>
<td>248 219</td>
<td>3,6 3,4</td>
</tr>
</tbody>
</table>

Using the new analytical approach to the stability and growth of cellular microstructures (subgrain and grain structures) developed by Humphreys [3] it can be shown that in heavily cold worked metals the grain boundary spacing $D_i$ is related to the initial grain size $D_0$ by

$$D_i = D_0 \exp(-\varepsilon). \quad (2)$$

The high angle boundary structure developed at large strains can be approximated to plates of thickness $D_i$ and the area of HAGB per unit volume $A_{hagb} = 1/D_i$. The subgrain size $D_s$ do not change significantly with strain and the area of low angle grain boundary (LAGB) per unit volume $A_{lagb} = 3/D_s$. Therefore, the fraction of HAGB will increase with strain. According to Humphrey’s model [3], the microstructure becomes less susceptible to discontinuous growth (recrystallisation) as the mean misorientation of subgrains increases and the structure will therefore be stabilised by an increasing fraction of high angle boundaries. The critical strain for microstructure stability decreases with initial grain size [3]. Oscarsson et al. (see ref. 11 in [3]) showed that the critical strain above which stable fine-grained microstructure is formed on annealing of Al sheets is produced by rolling reductions $> 95%$. All materials used in our annealing experiments were produced by cold rolling reduction higher than 95% and their strain level is therefore above the critical limit.

The character of grain structures of AA8006 and non-homogenised AA8011 sheets containing large fraction of diffuse grain boundaries (observed in polarised light) indicates that deformation recovery occurs (at least) partially by discontinuous subgrain growth (referred as "continuous recrystallisation" or extended recovery). In homogenised materials PSN mechanism can also contribute partially to the restoration process but its contribution is
supposed to be the same for all strain levels and depends only on the dispersion level. The increase of resistance to recrystallisation with increasing strain observed in all materials, excepting material B, is therefore due to the increase of the fraction of HAGB, which stabilises the as-deformed structure, i.e. to inhibited discontinuous subgrain growth.

The extremely fine microstructure produced by heavy cold rolling could be therefore stable against discontinuous recrystallisation but, of course, will be very susceptible to rapid continuous grain growth. Nevertheless, the continuous grain growth may be suppressed by second phase particles or other alloying additions. Using the approach developed by Humphreys [3], the effect of particle dispersion level on recovery and recrystallisation can be also assessed. The character of microstructure produced by annealing can be predicted by calculating the difference between the driving pressure $P_D$ for restoration and the pinning pressure $P_Z$ exerted by particles. According to [3] there is a regime of moderate pinning effect $\psi$ and small mean grain misorientation $\bar{\theta}$, where discontinuous subgrain growth may occur under conditions where recrystallisation can not take place (which is the case of our materials). The physical reasons for this are that the subgrain assembly provides the driving pressure $P_D$ for both processes and this is opposed by the particle pinning pressure $P_Z$. A HAGB will have larger $P_Z$ than a LAGB because of its higher energy (Read-Shockley relationship), and therefore there will be condition under which $P_D > P_Z$ for the low angle boundaries involved in discontinuous subgrain growth, but not the HAGB involved in recrystallisation.

The response to annealing treatment with high heating rate was strain independent in the samples prepared from material B (Fig. 1 and 3). This material, which has not been homogenised, probably has higher Si solute content than all other materials. Also, the initial grain size $D_0$ in this material is not known and could be very different and can shift the critical strain for microstructure stability to higher values. The understanding of the strain independent response to annealing of material B needs further investigation involving the effect of solute mobility on the drag pressure exerted on mobile dislocations and boundaries.

All above described phenomena are time and temperature dependent. As can be seen from Fig. 5b, in AA8011 samples the slow heating rate annealing for 8 hours at 280°C results in more important deformation restoration than the short term annealing at 300°C. On the other hand, when annealing is performed at high temperature the effect of heating rate and annealing duration are less important.

5. CONCLUSIONS

1. The response of heavily deformed AA8006 and AA8011 sheets to recovery and recrystallisation annealing is affected by several factors. The most important among them are solid solution concentration, second-phase particle dispersion and cold rolling reduction.

2. High Fe, Si and Mn solute concentrations effectively inhibit recovery at low annealing temperatures and therefore make the microstructure stable against discontinuous recovery.

3. Second phase particles act as sites of discontinuous PSN recrystallisation and the extent to which this mechanism contributes to restoration depends on material pre-treatment (homogenisation parameters determining the fraction of large particles).

4. Experimental evidence for the stabilising effect of high plastic strain induced by cold rolling on deformed sheet substructure was found. Higher cold rolling reductions result in the creation of higher fraction of high angle grain boundaries, therefore in the stabilisation of the microstructure against discontinuous grain growth (recrystallisation).
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REFERENCES: