STRUCTURE TRANSFORMATIONS DURING ANNEALING OF TWIN-ROLL CAST Al-Fe-Mn-Si (AA 8006) ALLOY SHEETS
I. EFFECT OF COLD ROLLING AND HEATING RATE

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The kinetics and temperature range of structure transformations occurring during annealing of twin-roll cast AlFeMnSi (AA 8006) alloy sheets were studied using thermoelectric power (TEP), electrical resistometry, light metallography, and transmission electron microscopy. The effects of cold rolling before heat treatment and of the heating rate were investigated. Both influence the onset and the kinetics of solid solution decomposition and the recrystallized grain size. The solid solution decomposition starts at 340 °C in the sample cold-rolled with 96 % reduction. It is more intensive above 430 °C in the material deformed to 33 % only. The non-equilibrium eutectic phase of the as-cast material fully transforms into individual particles of the cubic α-Al(Fe,Mn)Si, orthorhombic Al_6(Fe,Mn) and monoclinic Al_3Fe phases. The annealing above 510 °C leads to particle coarsening and partial re-dissolution of particles. This is an indirect evidence that dissolved Fe, Si and Mn atoms are still present in the matrix of the homogenized material.

K e y w o r d s: Al-Fe-Mn-Si alloys, twin-roll casting, phase transformations, electrical resistometry, thermo-electric power technique

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TRANSFORMACE MIKROSTRUKTURY PŘI ŽÍHÁNÍ PLYNULE LITÝCH PÁSŮ ZE SLITINY Al-Fe-Mn-Si (AA 8006) I. VLIV REDUKCE A RYCHLOSTI OHŘEVU

Kinetika a teplotní interval fázových transformací ve slitině AlFeMnSi (AA 8006) byly studovány pomocí termoelektrické síly, elektrické rezistometrie, světelné metalografie a transmisní elektronové mikroskopie. Bylo zjištěno, že velikost redukce tloušťky pásu i rychlost ohřevu ovlivňují počátek a kinetiku rozpadu tuhého roztoku a velikost rekrystalizovaných zrn. Rozpad tuhého roztoku ve slitině válcované za studena s redukcí 96 % začíná při 340 °C, zatímco u méně deformovaného materiálu je intenzivnější až nad 430 °C. Nerovnovážné eutektické fáze litého stavu se plně transformují na částice kubické fáze α-Al(Fe,Mn)Si, ortorombické fáze $A1_6$(Fe,Mn) a monoklinické fáze Al_3Fe. Žíhání při teplotách nad 510 °C vede k hrubnutí částic a jejich částečnému rozpouštění. Proto je možné tvrdit, že homogenizovaný materiál obsahuje stále určité množství atomů Fe, Si a Mn v tuhém roztoku.

1. Introduction

Thin sheets of an AlFeMnSi (AA 8006) alloy are frequently used as complex configuration fins in heat exchangers. In these applications, high formability and good strength is required. The continuous twin-roll casting (TRC) of strips only several millimetres thick followed by cold rolling offers important economical advantages – low capital investment, energy saving and low operational costs [1, 2]. The TRC route also offers some metallurgical advantages – the high solidification rate results in microstructure refining. Fine dendritic cells and small primary particles, increased alloying elements solid solubility and formation of metastable phases are accompanying phenomena typical for TRC materials [3]. Appropriately processed strip-cast AlFeMnSi alloys contain about 5 % volume fraction of finely dispersed particles, which, in turn, leads to a very fine grain size and a good combination of strength and ductility of the final gauge products.

The experience from the processing of other TRC aluminium alloys [4–6] indicates that the response of these materials to heat treatment is rather complicated. The information about the kinetics and temperature range of structure transformations in new alloys such as AA 8006 is only limited. Some recent results of our research indicated that long-term homogenization treatment prior to last cold rolling substantially improves final gauge strip properties [7]. However, a detailed study of all factors affecting final product microstructure and properties was necessary in order to find the optimum parameters for strip downstream processing.

This paper describes the effect of cold rolling and heating rate on the structure transformations occurring during annealing of twin-roll cast AlFeMnSi (AA 8006) alloy in the temperature range from 20 to 605 °C. The effect of homogenization is reported elsewhere [8].

2. Experimental details

The investigated material was cut from a twin-roll cast 8.5 mm thick strip of the commercial alloy AA 8006. The exact composition of the alloy in wt.% was: 1.5 Fe, 0.4 Mn, 0.16 Si, other elements < 0.015, balance Al. Two experimental methods were used for the investigation of microstructure evolution during annealing: 1) Thermo-electric power (TEP) measurements; 2) Electrical resistivity measurements (during heating *in-situ* and RRR measurement – see below).

Thermo-electric power (TEP) measurements were performed on samples cold rolled with 33 % reduction in thickness (mean plastic strain $\varepsilon = 0.46$). The samples were annealed and quenched from selected points of the annealing curve with the aim to simulate the temperature-time evolution during the industrial homogenization procedure. The rate of temperature increase during heating of industrial coils of 2 m in diameter is relatively high at low temperatures and much lower at high temperatures. The heating rate of the laboratory annealing was thus changed gradually from 0.5 to 0.05 ℃/min. The whole treatment consisted of heating from room temperature to 580 ℃, followed by 14 hours hold at this temperature. TEP was measured at 20 ℃ using special equipment designed and built in INSA in Lyon (France). The TEP coefficient of aluminium alloys depends mainly on the amount of Mn and Fe dissolved in Al matrix and it is only slightly affected by the strain in the matrix [12]. The relative TEP $\Delta S = S - S_0$ (where S and S_0 are the absolute TEP coefficients of the alloy and pure Al, respectively) is easily calculated. Recording of the ΔS evolution during annealing of AlFeMnSi alloys is thus a suitable tool for revealing the occurrence of transformations connected with changes in solid solution.

The phase transformations occurring during final gauge sheet annealing were studied by electrical resistivity, ρ, measurements. The resistivity evolution of 0.3 mm thick sample was measured *in-situ* during low rate linear heating of the specimen prepared by cold rolling of the as-cast material to 96 % reduction in thickness ($\varepsilon = 3.9$). The resistivity was measured during heating at the rate of 1 ℃/min from 20 to 605 ℃.

The ratio of $\rho(297\ \mathrm{K})/\rho(77\ \mathrm{K})$, known as the parameter RRR was also evaluated. RRR is independent of the specimen shape and size. RRR increases with increasing effective material matrix purity (it also depends mainly on Fe and Mn content) and for this reason it also characterizes the solid solution content.

Second phase examination was carried out by light microscope linked with an image analysis system. Grain structure in the long transverse strip plane was examined in polarized light on the samples anodised in Barker's solution. The degree of solid solution decomposition, intermetallic phases and matrix substructure were observed in transmission electron microscope (TEM) JEOL JEM 2000 FX equipped with X-ray energy dispersive spectrometer (XEDS) LINK AN 10 000. Thin foils were prepared parallel to the rolling plane of the sheet.

3. Results and discussion

3.1 Samples deformed with 33 % reduction before annealing

The as-cast strip microstructure consisted of elongated dendrites containing 7–8 % volume fraction of fine eutectic phase which was situated in inter-dendrite space [9, 10]. The maximum equilibrium solubility of Mn, Si and Fe in Al is 1.82 wt.%, 1.65 wt.% and 0.052 wt.%, respectively [11]. Local chemical analysis performed using TEM-XEDS indicated higher than equilibrium content of solute atoms in the matrix. The high solute content is caused by the high solidification rate during twin-roll casting. The content of solute manganese atoms was high and homogeneous, iron was concentrated in the eutectic phase and silicon was dissolved in the matrix. The eutectic phase was in the form of colonies of small needles, lamellas and ellipsoidal particles arranged in stringers. Besides iron, the eutectic particles contained also small amount of manganese, but no silicon.

Light microscopy examinations showed that cold rolling to 33 % thickness reduction induces only small changes in strip microstructure, reducing the spacing between eutectic colonies in the short transverse (S) direction – (Fig. 1a). TEM observations of thin foils showed that dislocations were arranged in cells.

Significant microstructure changes were observed during the slow rate annealing: the eutectic phase gradually transformed into coarser individual particles (Fig. 1b) and recrystallized grains replaced the deformed structure (Fig. 3b). Precipitation of intermetallic particles and their coarsening and re-dissolution were also observed. The microstructural changes may be represented by the evolution of the relative thermo-electric power coefficient, ΔS, as a function of temperature (and duration) – Fig. 2. The shape of the TEP curve indicates the occurrence of extensive changes in the solid solution. The slope of the curve changes several times and can be divided into 5 ranges with upper limits 270, 370, 430, 510, and 580 ℃, respectively.

The increase of ΔS up to 270 ℃ is very small (Fig. 2). TEM examinations indicated that no precipitation occurred up to this temperature. The dislocation density was significantly lower than in the as-rolled sample. ΔS is the most sensitive to matrix solute atoms distribution. Therefore, the observed increase of ΔS is supposed to be caused by the redistribution of solutes in connection with the deformed substructure recovery. In the as-rolled sample, the solute atoms are dispersed homogeneously in the matrix, whereas in the annealed sample cell wall dislocations probably trap them and thereby they are clustered.

A more important increase of ΔS is observed in the temperature range from 270 to 370 ℃. In the sample quenched from 370 ℃, the TEM revealed fine pre-

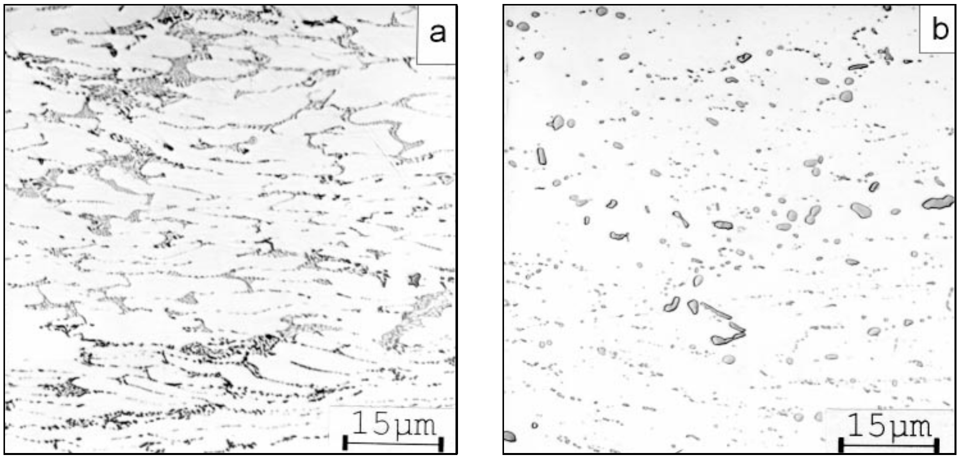


Fig. 1. Eutectic phase morphology after cold rolling of the twin-roll cast strip with 33 % reduction in thickness (a), and transformed particles after homogenization for 14 hours at 580 °C (b).

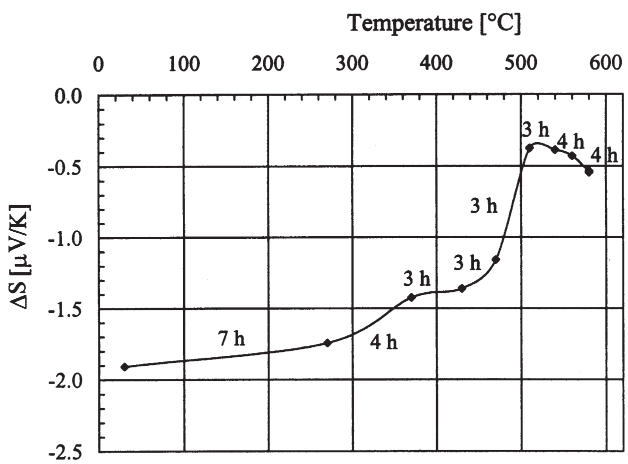


Fig. 2. Relative thermoelectric power ΔS as a function of temperature of as-cast strip cold rolled with 33 % reduction.

cipitates in the matrix. The effect of silicon on TEP is an order of magnitude lower than that of iron and manganese [12] and Si diffusivity is much higher than that of Fe and Mn. In addition, Si reduces the activation energy of Fe diffusion

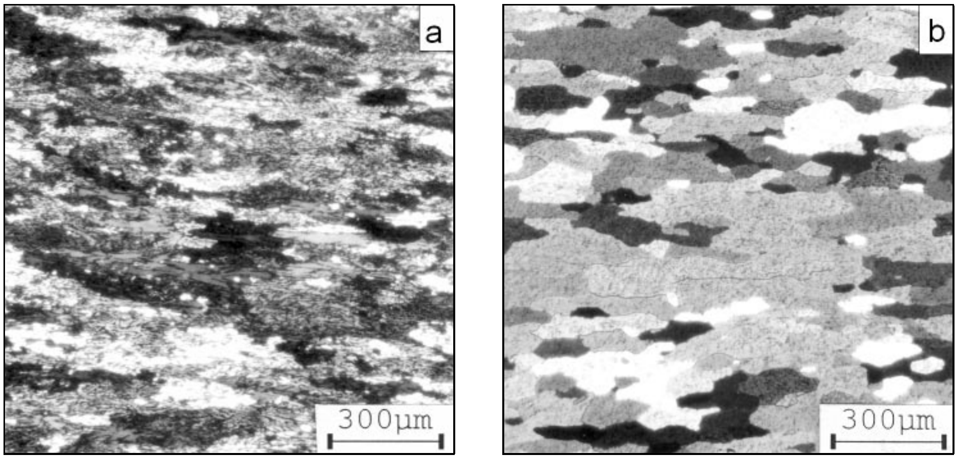


Fig. 3. Micrograph of grain structure evolution at high temperatures during annealing simulating industrial homogenization: a) sample quenched from 510 ℃, b) after heating for 14 h at 580 ℃.

in Al and thus increases its mobility [11]. Therefore, it is most probable that the increase of ΔS above 270 ℃ is due to the precipitation of particles rich in Si, Fe and Mn. The observed precipitate should be the cubic α-$Al_{12}(Fe,Mn)_3Si$ phase, because this phase was identified by selective TEM diffraction in samples annealed to higher temperatures containing coarser particles. TEM observations indicated that recovery of dislocation structure is more advanced, the dislocation cells have transformed into subgrains with well-depicted boundaries.

At temperatures ranging from 430 to 510 ℃, an abrupt increase of ΔS is observed. The TEM and light microscopy examinations revealed a dense population of fine particles inside former dendrite cells. In this temperature range, the eutectic clusters started to transform – some of the particles dissolved, while other coarsened. The relative TEP of Al alloys is affected mainly by the amount of Mn and Fe in solid solution. Therefore, the observed large increase of ΔS is most probably due to intensive precipitation of $Al_6(Fe,Mn)$ and Al_3Fe phases. The precipitation of α-$Al_{12}(Fe,Mn)_3Si$ particles continues and particles become coarser (selective TEM diffraction).

Above 510 ℃, a gradual decrease of ΔS is observed (Fig. 2). The TEM examination of samples quenched from 510, 540, 580 ℃ and after annealing for 14 h at 580 ℃, indicated that this decrease is caused by a partial particle dissolution resulting in the re-enrichment of the solid solution. The local X-ray analysis provided evidence for the enrichment of primary eutectic particles in Mn and Si and their transformation into α-AlFeMnSi phases. Coarse $Al_6(Fe,Mn)$ particles were observed both in former dendrite boundaries and on grain boundaries. Light microscopy examinations indicated that the density of small particles inside the former dendrite cells (originated from the solid solution decomposition) is the largest at 470 ℃ and decreases at higher temperatures.

The size and the density of second phase particles in samples quenched from temperatures above 510 ℃ were measured by image analysis using light microscopy. The mean values of particle characteristics such as the area A and the length D_{max}, the number of sections on unit of surface N_{A}, the volume fraction V_{v}, and the fraction of particles with $D_{\mathrm{max}} > 2\ \mu\mathrm{m} - F_{\mathrm{r}}$, are compared in Table 1. Even though the size distributions are truncated at the small sizes end, due to the resolution limit of the method, the results show an unambiguous tendency of particle coarsening.

Table 1. Second phase characteristics in the strip rolled with 33 % reduction

Annealing parameters		Particle characteristics			
Temperature [℃]	Time [hours]	A [μm^2]	D_{max} [μm]	$\overline{N}_{\mathrm{A}}$ [10^3 mm^{-2}]	F_{r} [%]
510	20	0.41	0.89	175.5	6.4
540	23	0.44	0.91	161.2	6.7
560	27	0.46	0.91	138.7	7.4
580	31	0.53	0.99	119.5	7.6
580	45	0.62	1.04	92.4	8.3

Both TEM and light microscopy examinations indicated that the phase transformations are accompanied by the recovery of the deformation substructure. At temperatures up to 470 ℃, recovery is the predominant softening process. The first recrystallization nuclei were found in the sample quenched from 470 ℃. More advanced recrystallization was observed at 510 ℃ (Fig. 3a). The alloy is fully recrystallized at 580 ℃. Recrystallized grains, markedly elongated in the rolling direction, are formed (Fig. 3b). The grain size in the sample held at 580 ℃ for 14 hours is $\sim 110\ \mu$m and $\sim 46\ \mu$m in the long and short transverse direction, respectively. The anisotropy of grain structure is caused by the specific spatial distribution of particles found in TRC alloys. The dendrites in TRC alloys are of pancake shape and are further flattened by cold rolling prior to homogenization. Many particles were found on former dendrite boundaries. They have strong pinning effect inhibiting grain growth in the short transverse direction. Recrystallized grains are flattened in the direction normal to strip surface as a result of grain boundaries and particles interaction.

3.2 Samples deformed with 96 % reduction before annealing

TEM examinations of the heavily cold rolled sample showed dislocation arrangements in cell walls and subgrain boundaries. The cell and subgrain size in the rolling plane was about 1 μm. The second phase was in the same form as in the less cold worked sample (section 3.1). However, cold rolling caused expressive flattening of eutectic colonies and their orienting parallel to the rolling plane. Individual particles rich in Fe and Mn pinning subgrain boundaries were also observed.

The evolution of resistivity $R = f(T)$ during the low rate linear heating and the corresponding normalised differential curve $(\mathrm{d}R/\mathrm{d}T)/R = f(T)$ are plotted in Fig. 4. The main anomaly of the differential curve is the presence of a deep minimum at 275 ℃. Metallographic and mainly TEM observations of samples quenched from 230 and 300 ℃ revealed the occurrence of pronounced recovery of the deformed dislocation substructure. The deformation recovery in Al alloys is usually not manifested by such abrupt change in resistivity. The contribution of solute atoms to alloy resistivity is much higher than that of intrinsic crystal defects [13, 14]. However, the situation may be complicated by the interference of solute (in this case of Si) and dislocation rearrangements. The redistribution of solutes, trapped by dislocations, is affected by dislocation recovery and this could result in the pronounced changes in resistivity. As solute rearrangements occur on the atomic scale, obvi-

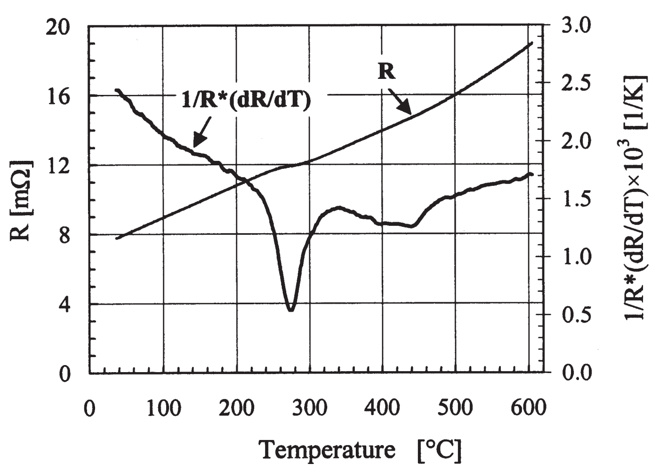


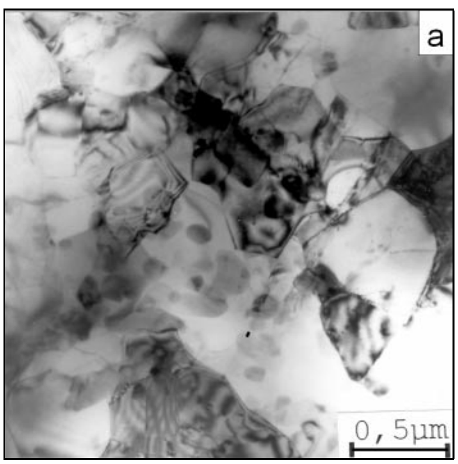
Fig. 4. Temperature dependence of resistivity R and the normalised differential resisitivity $(\mathrm{d}R/\mathrm{d}T)/R$ of strip cold rolled with 97 % reduction during *in-situ* linear heating at the rate of 1 ℃/min.

ously they cannot be observed by conventional TEM, but with a high probability they may be detected by resistivity measurements.

A gradual decrease of $(\mathrm{d}R/\mathrm{d}T)/R$ starting at 340 ℃ is observed (Fig. 4) indicating that solid solution decomposition occurs. The shape of the curve indicates that the process continues up to 445 ℃. TEM observations of samples quenched from 350 and 430 ℃ (Fig. 5a) showed the precipitation of α-Al(Fe,Mn,Si) particles, first in vicinity of eutectic colonies and later inside dendrite cells. The slope of the differential curve changes again at about 420 ℃. TEM observations of the sample quenched from 430 ℃ revealed the presence of new particles containing only Al, Mn and Fe. Similarly as in samples measured by TEP, compositions and crystallographic lattices of these particles correspond to these of the orthorhombic Al_6(Fe,Mn) and the monoclinic Al_3Fe phases.

Fig. 4 also shows that heating above 445 ℃ results in an increase of $(\mathrm{d}R/\mathrm{d}T)/R$ indicating the possible start of the partial dissolution of small precipitates and some of the eutectic particles. TEM observations showed that the primary eutectic phases are gradually dissolved and transformed into equilibrium ones. Particle coarsening was also observed (Figs. 5a,b). However, the particles in the sample subjected to linear heating up to 605 ℃, achieved in 10 hours, are significantly smaller and their number is higher than in the sample deformed 33 % and annealed at 580 ℃ during an overall longer time (Table 1).

Similarly as in the observations in section 3.1, the recovery of the deformation substructure proceeded in parallel with second phase transformations. The



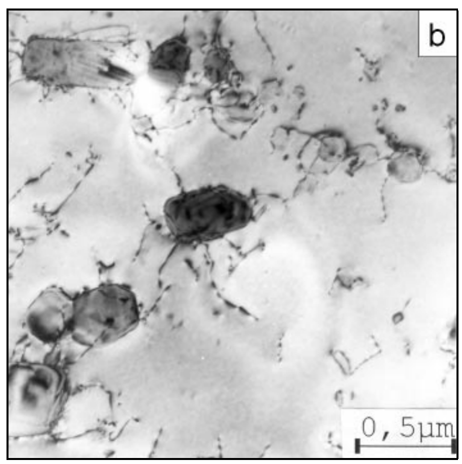


Fig. 5. TEM micrographs showing the second phase evolution in the homogenized thin strip during *in situ* linear heating at 1 ℃/min: a) sample quenched from 350 ℃, b) sample quenched from 605 ℃.

substructure did not change substantially up to 230 ℃. A marked decrease in dislocation density was observed by TEM of thin foils at 300 ℃. Light microscopy revealed the first recrystallization nuclei in the sample quenched from 430 ℃, i.e. at a lower temperature than in the less cold-rolled sample (470 ℃). Coarse recrystallized grains were observed in the sample heated up to 600 ℃. The grain size in the long and short transverse direction was ~ 2900 μm and ~ 240 μm, respectively.

The RRR value of the cold rolled sample was 2.3, whereas in the sample heated at 1 ℃/min up to 600 ℃ it was 4.0. This difference indicates a significant decrease of matrix solute concentration due to slow heating. On the other hand, TEP measurements on the material deformed 33 % showed that the minimum solute concentration is achieved at about 510 ℃. Above 510 ℃, a slow enrichment of the solid solution with solute atoms was confirmed. This result is of a great importance indicating that solid solution decomposition can occur in AA 8006 homogenized strips when the material is subjected to further annealing during the down-stream processing. The effect of homogenization on TRC final product properties is discussed elsewhere [8].

3.3 Discussion of the effect of cold rolling reduction and heating rate

TEP and resistometric measurements were carried out on samples subjected to cold rolling with distinctly different reductions (33 and 96 %). In addition, different heating rates during annealing of the strip were used. The highest heating rate of the slightly deformed material was 0.5 ℃/min, and it was gradually decreased to 0.05 ℃/min in parallel with temperature rising. The heavily deformed material was heated at constant rate of 1 ℃/min. The results of the experiments indicate that both the magnitude of cold rolling reduction and the heating rate influence strongly the kinetics of structure transformation during annealing.

The first recrystallized nuclei were observed at 430 ℃ in the heavily rolled sample. In the case of slightly deformed sample the temperature of recrystallization was 470 ℃. It is clear that the increase of cold rolling reduction combined with higher heating rate causes a shift of the onset of recrystallization to lower temperatures. The amount of stored deformation energy, which is the driving force for recrystallization, is higher in the heavily deformed sample and lower temperature start of recrystallization is expected. In addition, heating rate also affects the level of recovery. The amount of recovery is lower in the more quickly heated material. Due to this reason, recrystallization is easier, e.g. it begins at lower temperature, after shorter heating. Both effects, higher amount of deformation energy and higher heating rate, act in the same way.

The recrystallized grain size in the heavily rolled sample is significantly coarser than in the less deformed sample. This difference is due to the interaction between precipitation and recrystallization – the dense population of small particles formed earlier (at lower temperatures) in the more deformed sample inhibits grain nucleation and the growth of multiple nuclei.

In the less deformed sheet recrystallization begins at higher temperatures when the particles are sufficiently coarse. The start of recrystallization at higher temperatures facilitates simultaneous forming of nuclei and results in a finer grain size.

Furthermore, our results showed that in the material subjected to higher plastic deformation and thermally treated at higher heating rate the precipitation is shifted to lower temperatures. The size and density of precipitates observed at 350 ℃ (temperature reached in 5 hours) are significantly higher than in the less strained sample heated to 370 ℃ during 11 h. The origin of this difference is not yet well understood. One of the possible reasons is the accelerating effect of dislocations on precipitation nucleation observed by Koster and Hornbogen [15] in Al-Cu alloys or by Tsubakino et al. [16] in dilute Al-Fe and Al-Fe-Si alloys. In our case, the more heavily deformed material (96 % reduction), which was also heated at higher rate, has a higher density of dislocations. The dislocations have thus less time to recover and the density of nucleation sites for precipitates is much higher, consequently allowing precipitation to start at lower temperature. This explanation is supported also by the fact that the density of particles at the end of annealing is significantly higher in the more deformed material than in the less deformed one. Furthermore, the less deformed sample is subjected to longer annealing with a long-term stay at high temperature (580 ℃). It was shown that annealing at temperatures higher than 510 ℃ leads to dissolution of small precipitates. Therefore, both these differences contribute to the decrease of particle number density and to the increase of their mean size due to particle dissolution and coalescence.

4. Conclusions

The following conclusions can be drawn from the study of structure transformations during annealing of an AlFeMnSi (AA 8006) twin-roll cast and cold rolled alloy:

1. Long term high temperature (homogenization) annealing results in an extensive decrease of the concentration of solutes in Al matrix. The non-equilibrium eutectic phase formed during strip solidification fully transforms into individual particles of the cubic α-Al(Fe,Mn)Si, orthorhombic Al_6(Fe,Mn) and monoclinic Al_3Fe phases.

2. The heating rate and the level of deformation preceding heat treatment influence the onset and kinetics of solid solution decomposition. The precipitation starts at 340 ℃ in heavily cold-rolled material annealed at a higher heating rate (1 ℃/min). In the less deformed sample annealed at lower temperature and gradually decreasing heating the precipitation is the most intensive above 430 ℃. Moreover, the combination of higher reduction in thickness and higher heating rate results in a distinctly larger size and higher density of precipitates.

3. The grain size of recrystallized alloy is affected both by the degree of cold-rolling reduction and by the heating rate: coarse and uneven grains are formed in heavily cold-rolled sample and fine and uniform grain size is observed in the slightly deformed sample heated at the lower rate. This result is explained by the pinning effect of the dense population of particles in the more deformed material.

4. Both low and high rate annealing experiments proved that high temperature heat treatment (up to 580 and 600 ℃, respectively) results in a significant decrease of the amount of free solute atoms in the solid solution in comparison with the as-cast condition.

5. Nevertheless, annealing above 510 ℃ yields coarsening and partial redissolution of the particles. The matrix of the material annealed for 14 h at 580 ℃ contains dissolved Fe, Si and Mn atoms. In consequence, precipitates are formed during slow cooling or subsequent low temperature annealing of the homogenized alloy.

Acknowledgements

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