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Effect of homogenization on recrystallization in a twin-roll cast Al–Fe–Si alloy

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Abstract The effect of homogenization treatment on the recrystallization process in a twin-roll cast AlFeSi alloy was investigated by means of calorimetry, microstructural analysis, electrical conductivity, and hardness measurements and cupping tests. The response to annealing of cold-rolled AlFeSi sheet processed with a homogenization treatment at the cast gauge is a typical two-stage, recovery and recrystallization process, while that processed without homogenization softens without recovery. The rather limited precipitation capacity in the former allows recrystallization to occur largely discontinously, favoring the annealing texture. The nucleation rate and the volume fraction of the discontinously recrystallized grains are largely reduced in the sheet processed without homogenization, owing to extensive dynamic precipitation. This reduces the strength of the annealing texture components and gives a more or less random crystallographic texture after annealing. With a relatively finer-grain structure and a nearly random crystallographic texture, AlFeSi sheet processed to soft temper at 1 mm without a homogenization treatment is an attractive foil stock material.

Introduction

AlFeSi alloys can provide a suitable combination of strength and formability at foil gauges and have thus been traditionally used as foil stock [1]. The manufacturing cycle often starts with continuous strip casting and involves a recrystallization anneal at some intermediate gauge in order to restore ductility to facilitate further rolling to foil gauges [2, 3]. A high-temperature annealing treatment at the cast gauge, often referred to as homogenization, is a common process step, since the formability of the strip-cast materials without it, is relatively poor [4]. Due to the high solidification rates encountered in strip casting, the strip microstructure is far from equilibrium and benefits a great deal from homogenization at the very start of down stream processing. Homogenization of the cast strip brings about changes in size, shape and distribution of primary and secondary precipitates as well as in the extent of the supersaturation of the matrix which in turn, impact the response of the strip to a subsequent annealing treatment [5–7]. The present work focuses on the effect of homogenization treatment on the recrystallization process in a twin-roll cast AlFeSi alloy by means of calorimetry, electrical conductivity measurements and microstructural analysis in an effort to identify optimum down stream processing cycles.

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Experimental

The present investigation was carried out on a 6 mm-thick twin-roll cast AlFeSi alloy strip with 0.62 wt% Fe and 0.48 wt% Si. Of the two sets of samples sectioned from the cast strip, the first set was homogenized at the cast gauge while the second set was processed without homogenization. The homogenization treatment involved slow heating and a subsequent isothermal step where the samples were soaked at 560 °C for 8 h before they were finally furnace-cooled to room temperature to facilitate the precipitation of solute Fe and Si. The samples with and without homogenization were then cold rolled in a fully instrumented laboratory rolling mill to a final thickness of 1 mm before they were submitted to isochronal annealing treatments between 150 and 450 °C for 1 h.

The sheet samples processed to the final gauge with different down-stream processing cycles were prepared with standard metallographic techniques: ground with SiC paper, polished with 3-micron diamond paste and finished with colloidal silica. Their microstructures were examined after etching with a 0.5%HF solution using a Olympus BX51M model optical microscope. Samples for grain structure evaluations were anodized with Barker's solution and were then observed with an optical microscope using cross polarizers. The X-ray diffraction (XRD) patterns were recorded with a Shimadzu XRD 6000 Diffractometer equipped with CuKα radiation. The diffractometer was operated at very low scanning rates to improve the counting frequency. A Sigma Test Unit measured the electrical conductivity of the samples before and after the final softening anneal to judge the extent of precipitation activities. 3 mm Diameter disc samples were sparked out of the cast strips and rolled sheets for DSC measurements which were performed using a SETARAM Labysys DSC apparatus. Runs were carried out at a heating rate of 10 °Cmin^{-1} and under dynamic argon atmosphere. High purity aluminum was used as reference. Earing results were obtained using a Tinius–Olsen ductometer and the rim profiles of the drawn cups were used to roughly estimate the plastic anisotropy in O-temper sheet samples.

Results and discussion

The AlFeSi sheet cold rolled and annealed without prior homogenization exhibits modest hardening at low annealing temperatures until 200 °C (Fig. 1). This mild hardening is believed to be due to low temperature precipitation activities, as evidenced by a marked increase in the population of intermetallic particles upon annealing in this temperature range (Fig. 2). Softening by recrystallization starts at 200 °C and lasts until 350 °C with no evidence of prior recovery. Increasing the annealing temperature further does not bring about a reduction in hardness suggesting that softening is fully over at 350 °C. The response of the homogenized sheet to an annealing treatment at low temperatures is markedly different and is dominated by recovery until 250 °C (Fig. 1). Recrystallization follows recovery and hardness drops suddenly until the sample is fully soft at 300 °C. The recrystallization

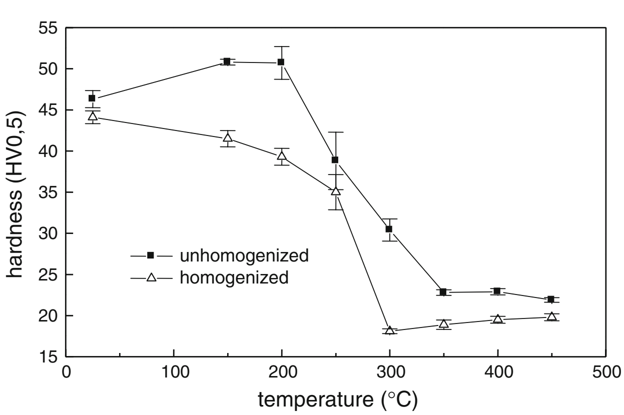


Fig. 1 Change in hardness of sheet samples processed with and without a homogenization treatment at the cast gauge with annealing temperature

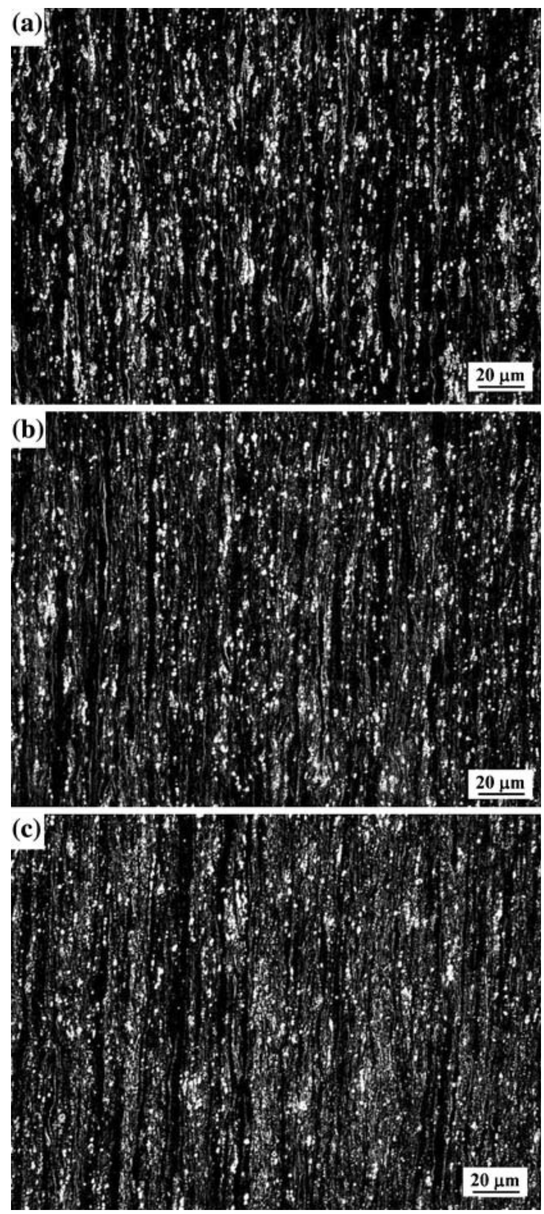


Fig. 2 Dark field optical micrographs of sheet samples (**a**) cold rolled to a final thickness of 1 mm and submitted to annealing treatments at (**b**) 150 °C and (**c**) 200 °C. (**b**, **c**) Show higher density of fine particles

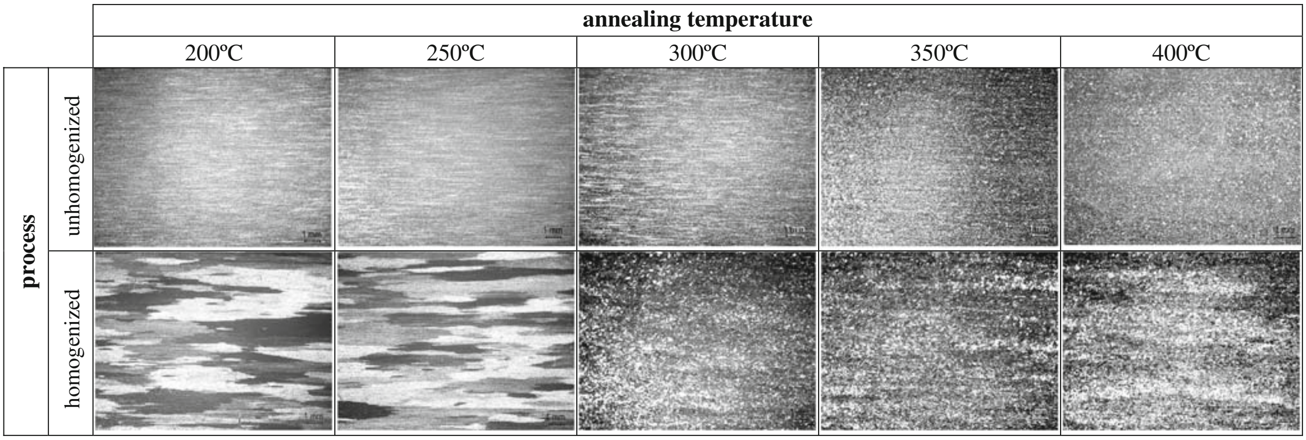


Fig. 3 Grain structures of sheet samples processed with and without homogenization at different annealing temperatures

finish temperatures estimated from softening curves are consistent with the evolution of grain structures in isochronally annealed sheet samples (Fig. 3). It is evident from both metallographic analysis and softening curves that recrystallization in the unhomogenized sheet is relatively sluggish.

While part of it may be linked with the annihilation of defects introduced during cold rolling, much of the conductivity increase in the unhomogenized sheet between 150 and 300 °C (Fig. 4) is accounted for by precipitation activities. The homogenization treatment at the start of processing is responsible for the higher starting conductivity in the homogenized sheet. Slow cooling following soaking at 560 °C has promoted the depletion of Fe and Si off the matrix and has reduced the precipitation capacity in this sample. The sheet sample processed without a homogenization treatment, on the other hand, has higher solute content at the start of annealing and thus offers a higher precipitation capacity as inferred from a relatively lower starting conductivity and a larger conductivity change upon annealing at all temperatures. Extensive

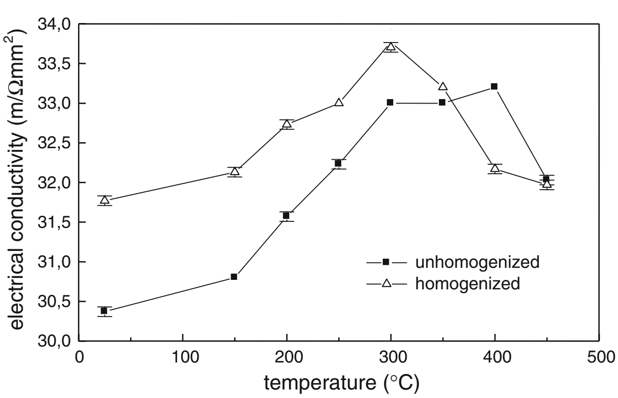


Fig. 4 Change in electrical conductivity of sheet samples processed with and without a homogenization treatment at the cast gauge with annealing temperature

dynamic precipitation and its interaction with the recrystallization reaction must be responsible for the relatively slower softening of the unhomogenized sheet [8–12]. Conductivity finally makes a peak before it starts decreasing, implying that some of the precipitates are dissolving back in the solid solution matrix at higher annealing temperatures. Metallographic work shows that the population of particles before and after the conductivity peak are indeed different (Fig. 5). XRD analysis of the isochronally annealed sheet samples suggests that there are Si particles dissolving in the aluminum matrix in this temperature range. The shift in the conductivity peak to lower temperatures in the case of processing with a homogenization cycle at the cast gauge (Fig. 4) implies an acceleration of both precipitation and subsequent dissolution activities in the homogenized sheet.

Further evidence for the account given above comes from the DSC experiments (Fig. 6). Sheet sample processed without a homogenization treatment reveals, between room temperature and 500 °C, three peaks two of which are exothermic. The first exothermic peak (peak A) occurs between 100 and 350 °C, and is followed by the much smaller second exothermic peak (peak B). The former is linked with the precipitation activities shown by electrical conductivity measurements (Fig. 4) and metallographic analysis (Fig. 2) to be in progress in this temperature range. The exothermic peak B, on the other hand, is produced by the recrystallization reaction which occurs in a discontinuous fashion, as inferred from a comparison of DSC curves of cast strip and cold-rolled sheet samples which shows that peak B is totally missing in the former. A rather large endothermic trough (trough C) follows right after peak B and is linked with the dissolutionizing of the Si phase, as evidenced also by the conductivity drop at these temperatures. While the peak arrangement in the DSC curve of the homogenized sheet is quite similar, the intensity and the temperature range of the

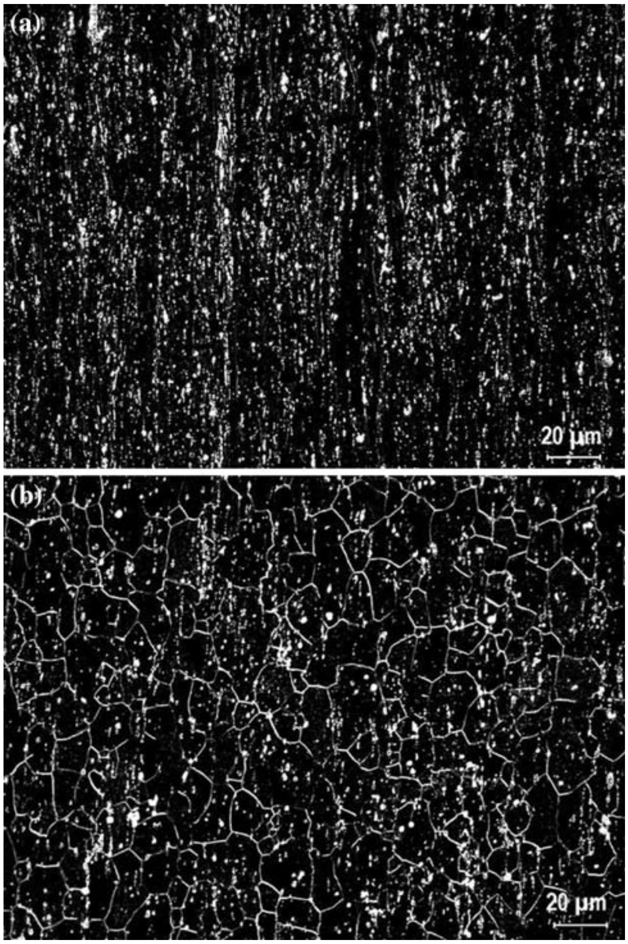


Fig. 5 Dark field optical micrographs of homogenized sheet samples cold rolled to a final thickness of 1 mm and submitted to annealing treatments at (**a**) 250 °C and (**b**) 450 °C, before and after the peak of electrical conductivity (Fig. 4)

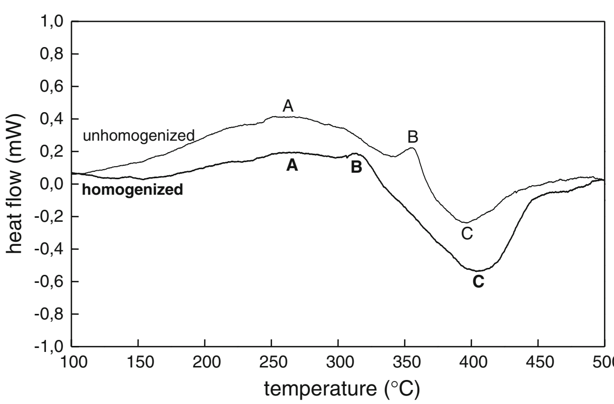


Fig. 6 DSC curves of sheet samples cold rolled to a final thickness of 1 mm with and without a homogenization treatment at the cast gauge

enthalpic signals are different. The exothermic peak A starts at higher temperatures and is much smaller, confirming that the precipitation capacity is reduced by the homogenization treatment as suggested by the conductivity

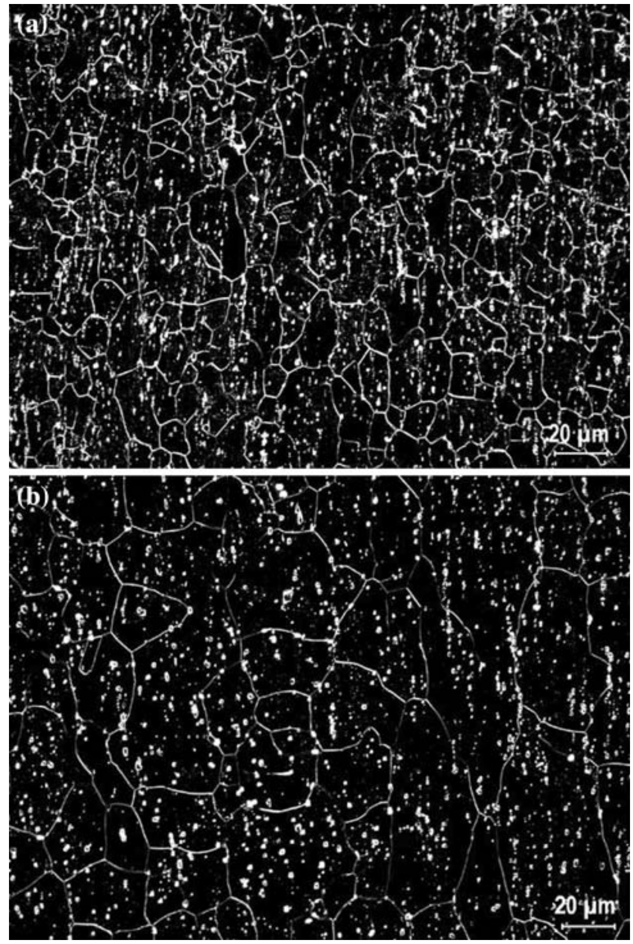


Fig. 7 Dark field optical micrographs of annealed sheet samples processed without (**a**) and with (**b**) homogenization

measurements. The second exothermic peak B, assigned to the discontinuous recrystallization reaction, has shifted to lower temperatures further confirming the acceleration in recrystallization in the homogenized sheet.

Of the two groups of sheet samples, the recrystallized grains in those processed without a homogenization treatment are relatively smaller with an average grain size of 13 microns (Fig. 7). The average grain size in the homogenized sheet is twice as large, 26 microns. With a relatively coarser dispersion of intermetallic particles formed after the homogenization heat treatment, homogenized sheet would be expected to take advantage of particle stimulated nucleation and thus to enjoy smaller grains. While the coarse particles might have contributed to the nucleation process in the homogenized sample, they are evidently involved also in the pinning of some of the grain boundaries (Fig. 8). This could be why the annealed grain structure is a heterogeneous and a relatively coarser one in the homogenized sheet. Moreover, the homogenized sheet suffers an abnormally coarse grain structure at the start of annealing (Fig. 3) inherited from the growth-dominated

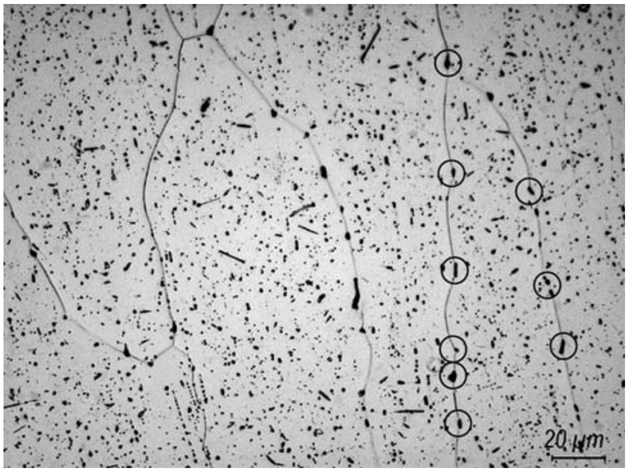


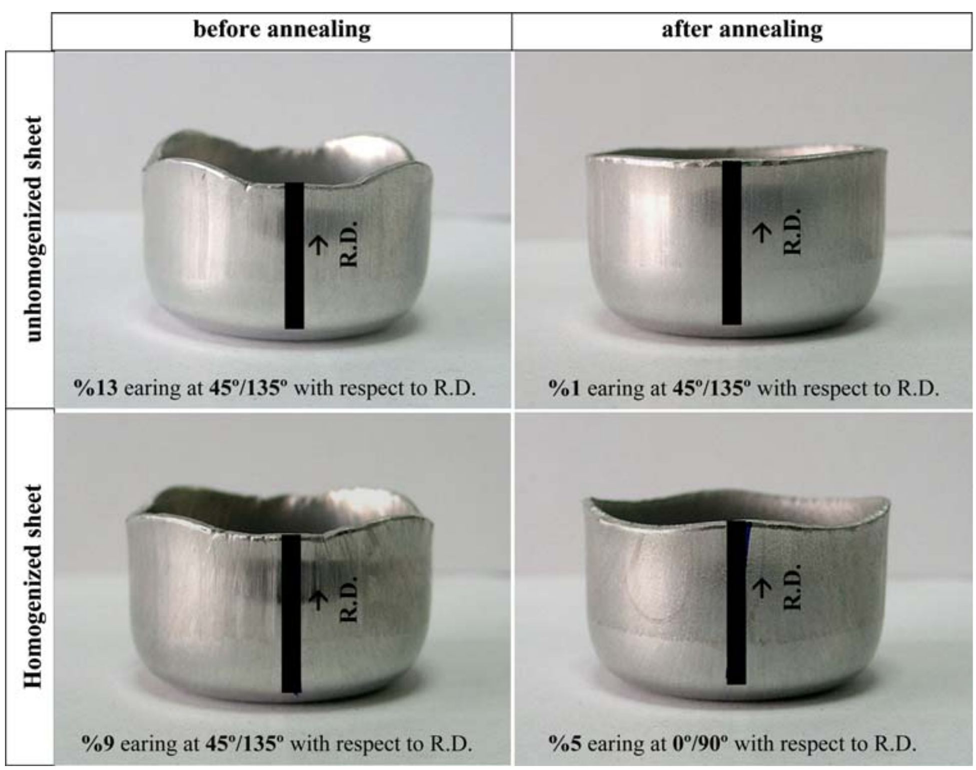
Fig. 8 Pinning of grain boundaries by AlFeSi intermetallic particles in the sample processed with a homogenization treatment at the cast gauge

homogenization treatment without prior cold rolling deformation and is thus at a disadvantage. Recovery which uses a significant portion of the deformation energy may also be, at least in part, responsible for the coarser grains in the homogenized sheet. While these could account for the coarse grains in the homogenized sheet, the higher cumulative deformation before annealing in the unhomogenized sheet may have contributed to the smaller grain size in this sample after annealing.

The rim profiles of the cups drawn from sheet samples processed with and without homogenization suggest that the homogenization has an impact on the texture of the annealed sheet (Fig. 9). Both samples are dominated by strong 45/135° ears in the as-rolled state. The latter exhibit a nearly flat rim profile with slight earing at 45/135° with respect to the rolling direction after annealing while the sheet samples processed with homogenization reveal 0/90° ears. The rolling texture in aluminum alloys is primarily composed of the S, Brass, and Copper orientations which promote earing essentially at approximately 45/135° with respect to the rolling direction [13, 14]. Annealing textures contain the Cube and the Goss orientations which give pronounced earing at the 0/90° directions [13, 14]. A nearly flat rim profile can be obtained only when a balance between the rolling and annealing texture components is achieved. It is fair to conclude then that the S, Brass, and Copper texture components are almost balanced by the Cube and Goss orientations in the sheet samples processed without homogenization, leading to a more or less random crystallographic texture. In contrast, the Cube and Goss components are dominant in annealed sheet samples processed with a homogenization treatment.

Sheet sample processed with a homogenization anneal not only has a higher density of coarse particles than those processed without homogenization, but also a matrix largely depleted of solute elements. Discontinuous recrystallization under such conditions changes the rolling

Fig. 9 Drawn cup rim profiles of sheet samples processed with and without homogenization, before and after recrystallization anneal



texture to annealing texture [14]. Therefore, stronger recrystallization textures are encountered in the homogenized samples after final annealing. Processing without homogenization, on the other hand, provides conditions suitable for dynamic precipitation during the softening anneal producing a very fine dispersion of intermetallic particles, as evidenced by the substantial change in electrical conductivity upon softening anneal at the final gauge and by the metallographic investigations. Dynamic precipitation in this case is believed to have reduced the nucleation rate and the volume fraction of discontinuously recrystallized grains and thus the strength of the annealing texture components [14].

Summary

The AlFeSi sheet cold rolled to 1 mm without a homogenization treatment has more solute than the corresponding material homogenized at the cast gauge. The response to annealing of the former is therefore dominated by precipitation activities at low temperatures. Recrystallization follows soon after and lasts nearly 150 °C until 350 °C. On the other hand the homogenized sheet softens fully at 300 °C by a typical two-stage recovery and recrystallization process. The rather limited precipitation capacity in the latter allows recrystallization to occur largely discontinuously, favoring the annealing texture and producing prominent 0/90° ears. In the sheet processed without homogenization the nucleation rate and the volume fraction of the discontinuously recrystallized grains are largely reduced, owing to extensive dynamic precipitation. This reduces the strength of the annealing texture components and gives a more or less random crystallographic texture after annealing. With a relatively finer grain structure and a nearly random crystallographic texture, AlFeSi sheet processed to soft temper at 1 mm without a homogenization treatment is an attractive foil stock material.

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