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ScienceDirect
Scripta Materialia 60 (2009) 5–8
Scripta MATERIALIA
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Impact of homogenization on recrystallization of a supersaturated Al–Mn alloy

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Received 2 June 2008; revised 1 July 2008; accepted 23 July 2008
Available online 9 September 2008

Impact of homogenization on the response of thin Al–Mn strips to interannealing was investigated. A homogenization anneal at the start of processing relaxes the supersaturation of the thin Al–Mn strip and avoids the precipitation–recrystallization interaction. The homogenized samples enjoy, in addition to an already decomposed matrix, a dispersion of coarse intermetallic particles resulting in a faster recrystallization. Reversion of the fine precipitates during cold-rolling and their reprecipitation during a subsequent annealing treatment displaces recrystallization to higher temperatures.

Keywords: Thermomechanical processing; Differential scanning calorimetry; Aluminium alloys; Recovery; Recrystallization

With as much as 1.5 wt.% Mn, Al–Mn1 alloys offer adequate strength, good corrosion resistance and weldability and are widely used in the form of sheet, plate and foil where formability is essential [1,2]. A significant portion is cold-rolled from twin-roll cast (TRC) strips [3] and is thus characterized by a finer dispersion of intermetallic particles and a higher level of matrix supersaturation with respect to the conventional ingot-cast grades [4–9]. These features are even more prominent in strips produced by the new generation of thin-gauge high-speed twin-roll casters where a relatively smaller volume of heat source and a larger heat sink (bigger caster rolls) provide very high solidification rates [10–13].

While processing thin Al–Mn strips without a homogenization treatment would be very attractive, the unhomogenized thin Al–Mn strips soften at unusually high temperatures and suffer from a coarse grain structure [14]. Hence, supersaturation of the Al matrix with Mn in the cast strip ought to be accounted for before further thermomechanical processing [15]. A homogenization anneal is known to make a critical contribution by allowing the precipitation of excess Mn in solution [4]. The present work was undertaken to identify the impact of a homogenization anneal on the subsequent recrystallization process during interannealing of a thin Al–Mn strip. The processing was performed in the same fashion and with the same Al–Mn alloy to those described in Ref. [14] but involved first a homogenization anneal at the cast gauge. Pieces 100 × 100 mm, were sectioned from the center of a thin Al–Mn strip, with 1.19% Mn, 0.61% Fe and 0.16% Si (all in wt.%), twin-roll cast at 3 mm. They were soaked at 560 °C for 8 h before they were finally furnace-cooled to room temperature and then deformed by cold-rolling to a range of strains between 0.2 and 3.5. The homogenized and cold-rolled sheet samples were submitted to 2 h isothermal annealing treatments in air at temperatures up to 400 °C in order to investigate their response to thermal exposure. The progress and interaction of precipitation and softening reactions were analyzed by the use of metallographic techniques, hardness testing, electrical conductivity measurements and differential scanning calorimetry (DSC).

The grain structures of the heat-treated sheet samples were investigated after anodic oxidation with Barker's solution, using cross polarizers. DSC analyses were performed by placing the sample disk in the sample pan and super-purity Al of equal mass in the reference pan of the cell. The cell was heated to 600 °C at 10 °C min^{-1} in a dynamic argon atmosphere (1 L h^{-1}). The heat effects associated with precipitation/softening reactions were then obtained by subtracting a super-purity Al baseline run from a given heat flow curve. A second set of sheet samples, much larger in size than those used in the DSC tests, was heated in an electric resistance furnace at the heating rate employed in the DSC unit and quenched from various temperatures between 200 and 550 °C. These samples were then subjected to electrical conduc-

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doi:10.1016/j.scriptamat.2008.07.047

tivity and hardness measurements to identify the structural changes responsible for each of the exothermic signals in the DSC scans. A Sigma Test Unit was used for the former, while microhardness measurements were carried out halfway between the center and the surface of these samples. Measures were taken to obtain reliable electrical conductivity data from the very thin foil samples produced by cold-rolling to high true strain levels. The foil samples were repeatedly folded, over an area of 20×50 mm, until a minimum thickness of 2 mm was obtained. The hardness values of the thin foil samples were measured with an ultra-microhardness tester capable of applying loads as small as 1 g.

The response to interannealing of the cold-rolled Al–Mn sheet processed without a homogenization anneal, and thus in a supersaturated state, was recently described in detail [14]. Interannealing these sheet samples below 450 °C produces only partial recrystallization regardless of the cold-rolling strain. While samples with a cold-rolling strain (ε) of at least 0.9 fully recrystallize above 450 °C, recovery becomes the predominant mechanism through which the strain energy is released during annealing at $\varepsilon > 3$. Recrystallization is once again retarded owing to extended recovery activities at still higher strains.

A homogenization anneal imparts to thin Al–Mn strips features that are distinctly different from those of the cast strip [3]. Fine α_c-$Al_{12}(Fe,Mn)_3Si$ particles, which predominate in the cast strip, are outnumbered by the relatively coarser $Al_6(Fe,Mn)$ particles, while supersaturation of the matrix with Mn is largely relaxed (Fig. 1a). Pinning of low- and/or high-angle grain boundaries by the very fine dispersoids forming during homogenization produces a coarse grain structure through the thickness but particularly near the surface (Fig. 1b). Nevertheless, the homogenization treatment improves the response of the thin Al–Mn strip to a subsequent annealing treatment in a profound way [3].

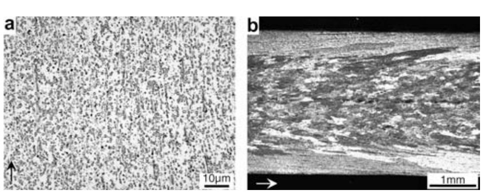

Figure 1. (a) Microstructure and (b) through-thickness grain structure of thin Al–Mn strip after homogenization treatment. Rolling direction is marked with an arrow.

The response to isothermal annealing treatments of sheet samples cold-rolled to a range of strains after a homogenization treatment is illustrated in Figure 2. No recrystallization occurs after cold-rolling to $\varepsilon \sim 0.2$. The DSC scan of this sample reveals no enthalpic signals between 100 and 500 °C, to confirm the grain structure macrographs (Fig. 3). An exothermic signal is noted first in the DSC scan of the sheet sample cold-rolled to $\varepsilon \sim 0.5$. Centering around 350 °C, this exothermic peak is readily linked with the formation of recrystallized grains which appear first after annealing at 350 °C. A cold-rolling strain of 0.5, however, produces only a partially recrystallized sample at this temperature. Further increase in cold-rolling strain helps to accelerate the recrystallization process. Sheet samples cold-rolled to $\varepsilon > 0.9$ recrystallize fully at 350 °C. The acceleration of the recrystallization reaction in sheet samples processed with a homogenization anneal is evidenced also by the gradual displacement of the recrystallization peaks in the DSC scans to lower temperatures. The cold-rolling deformation in this strain range, however, does not suffice to shift the recrystallization start temperature to below 300 °C. Hence, coarse grains inherited from the homogenization anneal survive the annealing treatment at 300 °C in sheet samples cold-rolled to $\varepsilon \sim 0.9$–1.6.

A further increase in the strain level to 2.3 displaces the onset of the recrystallization peak to nearly 280 °C and thus facilitates recrystallization during an isothermal anneal at 300 °C for the first time. The recrystallization finish temperature, however, is still above 300 °C and the recrystallization reaction is once again incomplete. Further increase in strain reverses this trend and the recrystallization peak starts to move to higher temperatures, starting at $\varepsilon \sim 3.5$. This implies an increasing resistance to recrystallization starting at $\varepsilon > 3.5$ and is evidenced by the failure to replace the coarse grains from the homogenization anneal in the sheet sample cold-rolled to $\varepsilon \sim 3.5$ and subsequently annealed at 300 °C.

While the configurations of their fully and partially recrystallized states with respect to ε and annealing temperature are similar, there are marked differences in the way sheet samples processed with and without a homogenization treatment respond to a subsequent annealing treatment. This is most evident in the respective DSC

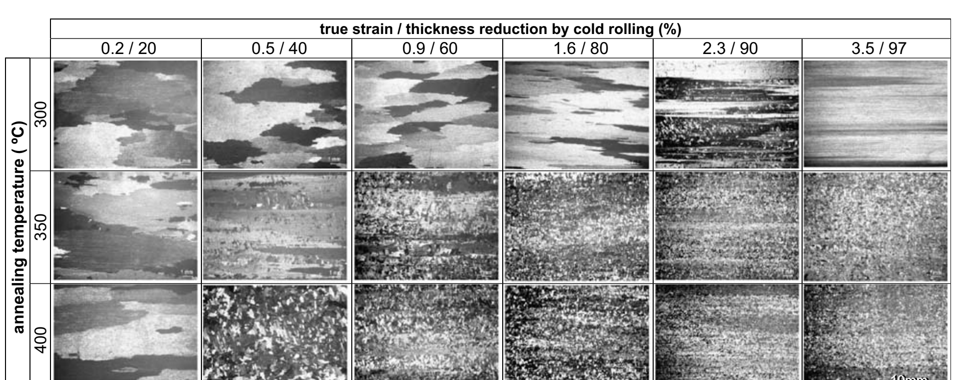

Figure 2. Grain structures of Al–Mn sheet samples homogenized, cold-rolled and subsequently submitted to 2 h isothermal annealing treatments. Macrographs were taken from the surface of the sheet samples.