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## Recrystallization of a supersaturated Al–Mn alloy

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The response of thin Al–Mn strips to interannealing was investigated. The surface of the sheet samples cold rolled to  $\epsilon$  < 0.9 is rearranged during annealing at 500 °C merely by grain growth. Sheet samples cold rolled to  $\varepsilon > 0.9$  recrystallize fully at a lower annealing temperature of 450 °C. While recrystallization is accelerated with further increase in strain, recovery becomes the predominant mechanism through which the strain energy is released during annealing sheet samples starting at  $\varepsilon \sim 1.6$ . 2008 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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Al–Mn alloys with up to 1.5 wt.% Mn offer adequate strength, excellent formability, good corrosion resistance and weldability, and have become the favorite choice for a wide range of packaging and architectural applications [\[1,2\]](#page--1-0). They are generally used in the form of sheet, plate and foil and a significant portion is cold rolled from twin-roll cast (TRC) strips [\[3\]](#page--1-0). Mn is partially in solution in conventional Al–Mn strips (6– 10 mm) owing to the high solidification rates encountered in TRC [\[4,5\].](#page--1-0) The softening kinetics is generally slower in TRC material with respect to the conventional ingot-cast grades due to a finer dispersion of intermetallic particles and a higher level of matrix supersaturation [\[6–9\].](#page--1-0) The former provide Zener drag while high solute levels can cause a strong solute–dislocation interaction during an annealing heat treatment [\[7\].](#page--1-0)

The new generation twin-roll casters are able to cast thinner strips (<3 mm) with distinctly different features. A relatively smaller volume of heat source and a relatively larger heat sink lead to rapid solidification in the caster roll gap [\[10–13\].](#page--1-0) Hence, supersaturation of the aluminium matrix with Mn may be substantial in thin Al–Mn strips and ought to be accounted for during thermomechanical processing [\[14\].](#page--1-0) While a high-temperature anneal can counteract this problem by allowing the precipitation of excess Mn in solution before further processing, it would be very attractive to process thin strips without a homogenization treatment. The present work was undertaken to explore the potential of such

processing with a particular emphasis on the response of thin Al–Mn strips to interannealing. Pieces  $100 \times 100$  mm sectioned from the center of a commercial Al–1Mn alloy, with 1.19% Mn, 0.61% Fe and  $0.16\%$  Si (all in wt.%), strip cast at 3 mm, was deformed by cold rolling to a range of strains between 0.2 and 5.1. The cold rolled sheet samples were submitted to 2 h isothermal annealing treatments in air at temperatures up to 550  $\mathrm{^{\circ}C}$  in order to discover 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. A Sigma Test Unit was used to measure the electrical conductivity of the sheet samples to give the solute levels in the Al matrix. Microhardness measurements were carried out across the thickness of the sheet samples. DSC analyses were performed by placing the sample disc in the sample pan and super-pure Al of equal mass in the reference pan of the cell. The cell was heated to  $600\,^{\circ}\text{C}$  at  $10^{\circ}$ C min<sup>-1</sup> in a dynamic argon atmosphere  $(11 h^{-1})$ . The heat effects associated with precipitation/softening reactions were then obtained by subtracting a superpure 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 DSC unit and quenched from critical temperatures which mark the major enthalpic effects revealed in the DSC scans.

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These samples were then subjected to electrical conductivity and hardness measurements to identify the structural changes responsible for each of the exothermic signals in the DSC scans.

A relatively smaller volume of heat source (thinner strip) and a larger heat sink (bigger caster rolls) favor rapid solidification in thin-gauge strip casting. This, together with hot rolling/extrusion in the roll gap, imparts to thin Al strips several features which are distinctly different from those of conventional strips. The features of the present Al–Mn strip are typical of thin strips with a featureless surface and a deformed interior (Fig. 1). Rapid solidification of the present strip is reflected by the predominance of very fine  $\alpha_c$ -Al<sub>12</sub>  $(Fe, Mn)$ <sub>3</sub>Si particles and by the supersaturation of the matrix with Mn particularly near the surface [\[3\]](#page--1-0). The surface regions of thin strips are known to be in a recovered state [\[11\]](#page--1-0) as confirmed by hardness measurements which show the thin Al–Mn strip to be nearly half hardness through its thickness with some softening very near the surface.

The response to isothermal annealing treatments of sheet samples cold rolled to a range of strains is illustrated in Figure 2. A minimum annealing temperature of  $500 \degree C$  is required to replace the featureless as-cast surface in sheet samples cold rolled to strains smaller than 0.9. The surface grains after annealing at 500  $\degree$ C are coarse and grow even larger with increasing strain in this range. The DSC scan of the sheet sample cold rolled to a strain of 0.5 reveals a single exothermic signal which centers around 500  $\rm{^{\circ}C}$  (Fig. 3). This exothermic



Figure 1. (a) Through-thickness; and (b) surface grain structures of thin Al–Mn strip in the as-cast state.

effect is clearly linked with the formation of coarse surface grains which appear at 500  $\mathrm{^{\circ}C}$  and is thus consistent with the metallographic analysis. Investigation of the sections, however, show that these coarse grains are restricted to the surface and that the as-cast features prevail in the interior ([Fig. 4a](#page--1-0)). Lack of recrystallization in the interior of the strip is confirmed further by hardness measurements [\(Fig. 5\)](#page--1-0). Hardness of the as-cast state is largely retained after annealing at  $500\text{ °C}$  except for some softening very near the surface. It is reasonable to conclude from the foregoing that the surface of the sheet samples cold rolled to strains smaller than 0.9 is rearranged during annealing at  $500^{\circ}$ C merely by a growth process (i.e. the movement of low- and high-angle boundaries) and that no recrystallization occurs in samples with low strains ( $\varepsilon$  < 0.9).

While the deformation introduced into the strip in this low strain range fails to initiate recrystallization, it serves as a source of defects to promote the transformation of low-angle boundaries to high-angle boundaries during subsequent annealing treatment. It is reasonable to conclude from the macrographs in Figure 2 that only a fraction of the subgrain/grain boundaries can enjoy



Figure 3. DSC scans of Al–Mn sheet samples deformed by cold rolling to the indicated strain levels.



Figure 2. Grain structures of Al–Mn sheet samples cold rolled and subsequently submitted to 2 h isothermal annealing treatments.

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