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Interannealing twin-roll cast Al–Fe–Si strips without homogenization

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When annealed without homogenization, a twin-roll cast Al–1Fe–0.2Si strip undergoes precipitation of α_c particles below 200 °C, dissolution of primary Al_6Fe and further precipitation of α_c particles above 350 °C. Recrystallization and then recovery processes are superimposed on this reaction sequence when the cast strip is cold rolled before thermal exposure. Recrystallization is over before there is measurable α_c precipitation at high strain levels and thus enjoys favourable conditions, producing an acceptable grain structure. Recovery predominates at still higher strains.

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

Al–Fe–Si alloys can provide a suitable combination of strength and formability at foil gauges and are preferred for a wide range of packaging applications [1]. Thermomechanical processing of these alloys have thus received considerable attention in recent years [2]. Of the several commercial alloys from the Al–Fe–Si system, Al–1Fe–0.2Si (AA8079) is a popular foil stock alloy with a relatively higher Fe/Si ratio [3,4]. A significant portion is cold rolled from twin-roll cast (TRC) strips [4,5] which exhibit slower softening due to a finer dispersion of intermetallic particles and a higher level of matrix supersaturation with respect to the conventional ingot-cast grades [6–11]. Processing cycle almost always involves a recrystallization anneal at some intermediate gauge in order to restore the ductility to facilitate further rolling to foil gauges [4,5]. Supersaturation of the aluminium solid solution matrix with Fe may have to be accounted for during thermomechanical processing in order to ensure a fine recrystallized grain structure [12]. While a high temperature anneal offers to counteract this problem by allowing the precipitation of excess Fe in solution before further processing [4,13], it would be very attractive to process TRC AlFeSi strips without a homogenization treatment [14]. The present work was undertaken to explore the potential of such processing with a particular emphasis on the response of TRC Al–1Fe–0.2Si strips to interannealing.

The present investigation was carried out with a 6 mm-thick twin-roll cast Al–Fe–Si alloy strip with 0.245 wt.% Si and 0.932 wt.% Fe. 100 mm × 100 mm pieces sectioned from the centre of the cast strip were cold rolled in a fully instrumented laboratory rolling mill to a range of strains between 0.5 and 3.0. The cold rolled sheet samples were submitted to 1 h isothermal annealing treatments in air at temperatures up to 550 °C in order to find out about their response to thermal exposure. Metallographic techniques, hardness testing, electrical conductivity measurements, differential scanning calorimetry (DSC) and X-ray diffraction (XRD) were employed to investigate the structural transformations taking place during thermal exposure.

The cast strip and cold rolled sheet samples were ground with SiC paper, polished with 3 μm diamond paste and finished with colloidal silica. Their microstructures were examined after etching with a 0.5%HF solution using an Olympus BX51M model optical microscope. The grain structures were investigated after anodic oxidation with Barker's solution, using cross polarizers. A Sigma Test Unit measured the electrical conductivity of the samples to estimate the extent of precipitation activities. The XRD patterns were recorded with a Shimadzu XRD 6000 diffractometer equipped with CuK_α radiation, at very low scanning rates to improve the counting frequency.

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About 3 mm diameter disc samples were gently machined from the cast strip and cold rolled sheet samples for DSC measurements. Runs were carried out at a heating rate of 10 K min^{-1} and under flowing argon (1 l/h) by placing the sample disc in the sample pan and super purity aluminium of equal mass in the reference pan of the cell. The heat effects associated with various 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, were heated in an electric resistance furnace at the DSC scan rate and quenched from critical temperatures which mark the major enthalpic signals revealed in the DSC scans. These samples were then subjected to electrical conductivity and hardness measurements to identify the structural changes responsible for each of these signals.

In spite of high solidification rates encountered in strip casting and a Si content far less than that required to fully precipitate Fe, only a portion of Fe was shown by electrical conductivity measurements to be in solution in the cast strip. The electrical conductivity after soaking at 560 °C is merely 2 MS m^{-1} higher than that measured in the as-cast state, much less with respect to that encountered in Al–Mn alloys [14]. The cast strip was shown by XRD to be dominated by the metastable Al_6Fe phase. Fe is known to precipitate out of the melt to form Al–Fe binary intermetallic particles during solidification since its solubility in solid aluminium is practically nil while a much larger fraction of Si dissolves in the matrix phase [12,15,16]. Al_6Fe is a metastable phase and was replaced almost entirely by α_c-$Al_{12}Fe_3Si$ upon soaking at 560 °C. Al_3Fe particles were also noted when the cast strip was soaked at still higher temperatures and/or after a cold rolling pass. It is also worth mentioning that the cast strip was harder than its soft-temper counterpart by about 15 HV. The deformation introduced by TRC was apparently restored only partially by dynamic processes during hot rolling of the just solidified strip. Strips with similar features were shown by TEM investigations to be largely recovered across the section [17].

The response to thermal exposure of the TRC Al–1Fe–0.2Si strip and sheet samples cold rolled to a range of strains (ε) is illustrated in Figure 1. The DSC scan of the cast strip reveals an endothermic trough between 275 °C and 400 °C (signal 1) and two neighbouring exothermic peaks which follow right after (signals 2 and 3). Detailed analysis of cast strip samples submitted to high temperature soaking have shown relaxation of the matrix supersaturation, Al_6Fe to α_c-$Al_{12}Fe_3Si$ transformation and grain growth to take place between room temperature and 560 °C. The grain structure of the cast strip annealed without prior cold rolling is rearranged during annealing merely by a growth process above 500 °C with no evidence of recrystallization. Grain growth, driven by the reduction of the total grain boundary energy, is indeed exothermic and was shown by metallographic analysis to start above 500 °C for the present alloy. Hence, any deviation from the baseline at $T > 500$ °C, including signal 3, can be appropriately linked with grain growth activities. Both conductivity and hardness were found to increase until 200 °C, implying a precipitation reaction which relaxes the supersaturation of the aluminium solid solution matrix via precipitation of α_c-$Al_{12}Fe_3Si$ particles. It is inferred from the DSC scan, however, that the amount of α_c precipitation is too small to generate a measurable exothermic peak.

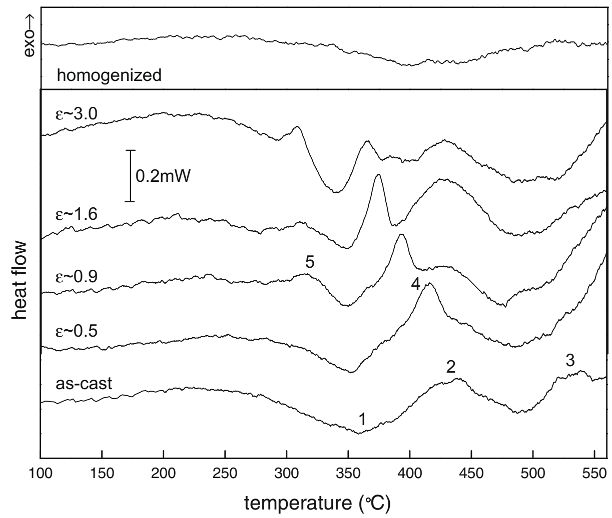

Figure 1. DSC scans of Al–Fe–Si sheet samples deformed by cold rolling to the indicated strain levels and of the strip submitted to a homogenization treatment at the cast gauge.

It is interesting to note that signals 1 and 2, while prominent in the as-cast state, are entirely missing when the strip is first soaked at 560 °C to transform the metastable phase to a stable variety (Fig. 1). Hence, of the three structural transformations that take place until 560 °C, $Al_6Fe \rightarrow \alpha_c$-$Al_{12}Fe_3Si$ transformation appears to be the only reaction that could be linked with these two signals. Dissolution of Mg–Si clusters and subsequent precipitation of the hardening β''-Mg_2Si phase during artificial ageing of heat-treatable 6XXX alloys, are known to produce similar peak configurations [18]. Al_6Fe to α_c-$Al_{12}Fe_3Si$ transformation is thus judged to occur via the solutionizing of the Al_6Fe particles followed by the precipitation of the α_c-$Al_{12}Fe_3Si$ variety. The former reaction is then claimed to be responsible for signal 1 while precipitation of the α_c-$Al_{12}Fe_3Si$ phase is linked with the exothermic signal 2. This account of the DSC scan is consistent with the metallographic analysis of samples annealed in this temperature range, showing first a decrease and then an increase in the population of intermetallic particles (Fig. 2a). Evolution of Bragg's reflections of the respective phases provides further evidence for this reaction sequence (Fig. 3).

Several changes are noted in the DSC scan when the cast strip is cold rolled before thermal exposure (Fig. 1). The sheet sample cold rolled to $\varepsilon \sim 0.5$ additionally reveals an exothermic signal (signal 4) which sits on top of signal 2. Signal 4 is displaced to lower temperatures with increasing strain and grows bigger until $\varepsilon \sim 1.6$ before it is reduced in size at still higher strains. Cold rolled sheet samples were shown by metallographic analysis and hardness measurements to fully recrystallize when isothermally annealed just above, but not before, signal 4, regardless of the strain level (Fig. 2b). Signal 4 is thus