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Influence of pre-recovery on the subsequent recrystallization and mechanical properties of a twin-roll cast Al-Mn alloy

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A B S T R A C T

A two-step annealing treatment consisting of a pre-recovery process (450 °C/5 h) and a following recrystallization process (530 °C/15 h) was developed for a cold-rolled twin-roll cast 3003 alloy (Al-1.13Mn-0.45Fe-0.1Si in wt%) sheet. The effects of pre-recovery on recrystallization behavior and mechanical properties were systematically studied. Our results show that fine quasi-equiaxed grains together with very weak ND-rotated cube (001) < 310 > texture was successfully obtained using the proposed two-step annealing treatment. It is found that pre-recovery stimulates the formation of precipitates-free zones around constituent particles. Within such precipitates-free zones, substantial random oriented sub-grains can readily grow to critical size for nucleation, which contribute to enhanced nucleation without preferential orientation. This is the reason for the formation of fine grains as well as a weak texture in the two-step annealed sheet. The sheets that were subject to this two-step annealing also exhibits an extremely weak mechanical anisotropy with a Δr value 0.03, much lower than that of sample (0.31) subjected to a single-step annealing at 530 °C for 15 h.

1. Introduction

Al-Mn alloys are widely used in packaging container, beverage can body and automobile industry because of a good combination of adequate strength, excellent corrosion resistance and formability of these types of alloys [1,2]. Generally, they are supplied in the form of semi-finished sheets before manufacturing into final products by deep drawing. Grain structure and texture control are of essential significance for deep drawability. A coarse grain structure might cause orange peel and a strong cube texture could give rise to 0 and 90° ears during cup drawing. Therefore, refined and equiaxed grain structure, weak texture and low anisotropy of mechanical properties are attempted to attain for the Al-Mn alloy sheet to ensure a satisfactory formability.

Conventionally, Al-Mn sheets are predominantly manufactured from direct chill (DC) cast ingots by rolling and annealing. Numerous works have devoted to understanding the recrystallization behavior of the cold-rolled DC Al-Mn sheets. The fine controlling of the microstructure and texture of DC Al-Mn sheets have been already achieved to date [2–7]. However, the process of producing sheets from DC ingots involves multiple procedures, such as scalping, homogenization and hot and cold rolling etc., which inevitably cause relatively high cost. For the sake of cost reduction, twin-roll cast (TRC) was taken as an alternative technique to traditional DC in manufacturing of Al-Mn sheets which has recently attracted extensive attention [8]. In the TRC process, Al-Mn strips are directly fabricated from molten metals, so that they undergo a rapid solidification and a large amount of Mn solutes retain in the solid solution. The supersaturated Mn will precipitate as fine Mn-bearing dispersoids in the subsequent thermo-mechanical procedures (TMP). These fine particles pin sub-grain boundaries and inhibit the sub-grains from growing into sufficient size for nucleation during annealing, resulting in the formation of coarse and elongated grains with strong specific texture components [2–5,9–12]. Therefore, the formability of TRC strips is generally inferior to that of their DC counterparts after the same downstream processing.

So far, the microstructure and texture evolution of cold-rolled TRC Al-Mn alloys are far less studied than that of DC alloys. It is documented in the limited existing literature that two methods have been tried to control the recrystallization behavior of heavily deformed TRC Al-Mn alloys. One approach is to use a cyclical homogenization for a long duration (more than 120 h) to increase the overall size of constituent particles and reduce the Mn supersaturation in the matrix [13]. The other way is to employ an isothermal annealing (or extremely fast heating rate) in a lead bath above the critical temperature at which the alloys recrystallize before precipitation [14]. The essence of both methods is to enhance the nucleation rate, either by eliminating the negative effect of concurrent precipitation or by increasing particles-stimulated nucleation (PSN) sites. Both techniques have been proved to be effective in developing fine grain structure. However, these ap-

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proaches are impractical for massive industrial production since they are time-consuming and exclusively expensive. Moreover, the texture and mechanical properties anisotropy were not addressed in these investigations. Therefore, it is desirable to develop a feasible TMP route to refine the grains and reduce the texture intensity of the TRC strips in a cost effective manner.

Apart from the aforementioned techniques, pre-recovery treatment is another potential way to enhance the nucleation rate of recrystallization in high stacking fault energy (SFE) materials and refine the final grains. It has been recently reported that, in pure molybdenum [15,16] and Al-Mg alloys [17], the large recovered sub-grains generated during an extended recovery can act as new nuclei, which promotes nucleation during subsequent recrystallization. In addition, the nucleation also strongly affect the annealing texture [18]. For instance, Gatti et al. [19] found that pre-recovery treatment affected the nucleation rate of cube-oriented grains and thereby significantly reduced the cube texture in warm-rolled Al-2.5 wt% alloys. In this regard, it can be expected that pre-recovery may serve as an effective method to simultaneously tailor the microstructure and texture to optimize mechanical properties of Al-Mn alloy. However, no publications have addressed this issue.

In this paper, a two-step annealing process consisting of a pre-recovery process (450 °C/5 h) and a following recrystallization process (530 °C/15 h) was developed to tailor microstructure, texture and mechanical anisotropy of a cold rolled TRC 3003 alloy sheet, with the aim to study the influence of pre-recovery on the microstructure and texture evolution. A fine grain structure together with very weak texture was obtained through the proposed two-step annealing process. Finally, the Al-Mn alloy sheet with an almost completely removed planar anisotropy was achieved. The corresponding mechanisms were studied and discussed.

2. Materials and experimental

Commercial TRC 3003 alloys (Al-1.13Mn-0.45Fe-0.1Si in wt%) were used in this work. The as-received sheets with 6.4 mm in thickness were cold-rolled to about 4.5 mm with a 28.6% reduction. It should be noted that the sheets have been already deformed during the casting process, the total deformation (hot and cold) reduction was supposed to be 50%. The cold-rolled materials were then subjected to a two-step annealing (hereafter referred as TSA) in a pre-heated muffle furnace. TSA included an initial low temperature pre-recovery annealing at 450 °C for 5 h and a subsequent high temperature recrystallization annealing at 530 °C for 15 h at a typical industrial heating rate of 60 °C/h. A single-step (referred to as SSA) at 530 °C for 15 h was also carried out for comparison. All the samples were immediately water-quenched after annealing.

The softening and precipitation behaviors were examined by Vickers hardness tests and electrical conductivity measurements at room temperature, respectively. Hardness measurements were conducted using a 500 g load with a dwelling time of 15 s, and the average hardness from eight independent measurements was given. Specimens with dimensions of 16×16×4.5 mm were machined for electrical conductivity measurements. Electrical conductivity was measured using a digital D60K conductivity instrument. Second-phase particles (constituent particles and precipitates) were examined by back-scattered electron (BSE) channeling contrast imaging in a Focused ion beam scanning electron microscope (FIB/SEM, ZEISS AURIGA).

The microstructure and crystallographic texture of the annealed sheets were investigated by means of Electron backscatter diffraction (EBSD) on a TESCAN MIRA3 scanning electron microscope (SEM). In the EBSD analysis, a step size of 0.05 μm was selected for the recovered samples, while a larger step size of 1–2 μm was chosen to investigate the grain structure and texture of the partially and fully recrystallized samples. EBSD orientation maps of annealed sample, covering more than one thousand grains were used to examine the orientation of the recrystallized grains and the texture as well. The crystallographic texture was represented by orientation distribution functions (ODFs), with orthotropic sample symmetry and a Gaussian half-width of 5° applied. Boundaries with misorientations between 2° and 15° were defined as low-angel boundaries (LABs) and those of misorientations above 15° as high-angle boundaries (HABs). The grain size d was automatically measured by the *HKL* Channel 5 EBSD system. All micrographs presented below were characterized at the quarter of thickness of the rolling direction (RD) and normal direction (ND) planes.

Mechanical properties and Lankford values (r-values) under tension along the RD, TD and 45° away from the RD were determined using tensile tests at room temperature. Among the above parameters, r value was determined by a tensile strain of 20%. The gauge length of the tensile specimens was 50 mm, and the width was 12.5 mm. Tensile tests were performed on a SHIMADZU (AG-X) universal testing machine with a constant crosshead speed of 1 mm/min, which corresponded to an initial stain rate of 3.33×10^{-4} s^{-1}.

3. Results

3.1. Softening and precipitation behavior

Softening behaviors during SSA and TSA are tracked by Vickers hardness as a function of annealing time as illustrated in Fig. 1. Hardness decreases slowly during recovery and drops abruptly as the primary recrystallization starts. In the SSA case (Fig. 1a), the recrystallization of the cold-rolled TRC 3003 sheets starts after 20 min of incubation period at 530 °C; while in the case of TSA (Fig. 1b), recrystallization initiates immediately after the temperature ramps to 530 °C. Before the onset of recrystallization, the hardness drops from 63.2 in the cold-rolled state to 54.2 and 49.1 in SSA and TSA samples respectively, indicating that pre-recovery releases much more stored energy. Eventually, it takes about 3.8 h and 6 h for the SSA and TSA samples to complete recrystallization, respectively, after which the hardness value reaches a plateau of about 33.5 for both cases.

The content of Mn in the Al matrix was frequently estimated according to the relationship between the conductivity and concentration of alloying elements in solid solution, the details of which can be found in Refs. [11,20]. The electrical conductivity measurements and the corresponding estimation of Mn in solid solution are shown in Table 1, where the subscripts CR, RV and RX stands for cold-rolling, recovery and primary recrystallization, respectively. From the changes of electrical conductivity, it can be found that the precipitation evolution during SSA and TSA behaves quite differently. For the SSA case, precipitation of Mn-bearing dispersoids occurs concurrently with the recovery and recrystallization process; approximately 0.56 wt% Mn

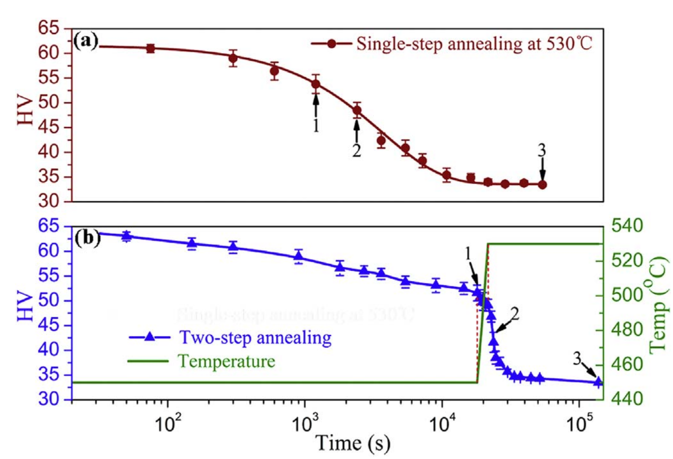

Fig. 1. The variation of hardness as a function of annealing time for (a) SSA and (b) TSA. Arrows 1, 2 and 3 represent the stage of recovery, partial recrystallization and complete recrystallization, respectively.