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The mechanism of grain refinement and plasticity enhancement by an improved thermomechanical treatment of 7055 Al alloy

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ABSTRACT

An improved double step hot rolling (DTR) processing was proposed to manufacture fine grained Al–Zn–Mg–Cu alloys based on pre-deformation, short-period annealing and final hot rolling. The DTR processing can produce high-quality rolling sheets with much finer recrystallized structures and significantly improved mechanical properties when compared with the conventional hot rolling (CTR). The main differences between the two TMTs (thermomechanical treatments) were investigated and the specific grain refining procedure of DTR was carefully characterized. The results show that the grain refinement is mainly proceeded *via* dislocation rearrangement and low angle grain boundaries transition, which can be attributed to the pinning effect of deformation induced precipitates (DIPs). Pre-deformation can accelerate the formation and spheroidization of fine DIPs which prohibit the migration of grain boundaries and the movement of dislocations. As a result, high density dislocation cells are formed and turn into polygon sub-grains after short-period intermediate annealing. During the final hot rolling, low angle grain boundaries gradually transferred into high angle grain boundaries which contributes to the final fine grained structures. The initiation and propagation of cracks were also delayed by grain refinement.

1. Introduction

High strength/toughness and lightweight structural materials (such as Al-Zn-Mg-Cu (7xxx series) alloys with excellent stiffness/strength to weight ratio, fair corrosion resistance and plasticity/toughness, excellent weldability/processing properties [1]) are becoming critically important for the aerospace related fields [2].

Different strategies had been designed to improve structures and properties of 7xxx series Al alloys, such as (i) optimizing alloy composition including micro-alloying elements. Zn and Mg are the main elements of strengthening phase $MgZn_2$ in A1-Zn-Mg-Cu alloys [3], so their contents and ratio can greatly affect the mechanical properties of 7xxx series Al alloys. (ii) Microstructural refinement by advanced preparation techniques, *e.g.*, powder metallurgy [4], spray forming [5], low frequency electromagnetic casting [6], severe plastic deformation (SPD *e.g.*, Equal channel angular pressing, ECAP [7]; Multi direction forging, MDF [8]; Cryogenic rolling, CR [9]); or thermomechanical treatment, TMT [10]. Among these methods, the control of alloy composition is feasible. However, increasing contents of the key alloying elements (Mg, Zn, Cu) can produce higher volume fraction of precipitates that can enhance the mechanical properties. However, this may deteriorate the workability (plasticity)/corrosion resistance [11,12]. The SPD processes can greatly refine grains (*e.g.*, to sub-micrometer or nanometer level) that favors the strength, ductility and toughness [13,14]. But the required severe plastic strains and the difficulties in producing large-scaled metals or alloys limit some application of them [15]. As an effective method to produce fine-grained Al alloy sheets, the traditional TMT cannot obtain ultra fine-grained structures (grain size $d < 30$ μm) for the high stacking fault energy of Al-Zn-Mg-Cu alloys [16]. Interestingly, it has been revealed that some particles in Al-Zn-Mg-Cu alloys are verified to play important roles for microstructural refinement, *e.g.*, inhibiting grain growth (pinning effect) or accelerating recrystallization (particles stimulate nucleation, PSN) [17–19].

Russo [17] had designed a TMT process: homogenizing the ingots at low temperature → warm deformation → recrystallization, by which the grain growth would be retarded by the Cr dispersoids so as to refine grains as well as improve the ductility and toughness of 7075 Al alloy. However, it is difficult to acquire initial ingots with Cr in solution and deformation of these plates is hard to be realized [18]. Then, a multi-step TMT route (homogenization and step furnace cooling → warm rolling → solutionizing and recrystallizing) for manufacturing fine-grained 7075 Al alloy was developed by Waldman J to overcome this problem (using pre-precipitation *via* slow step furnace cooling to obtain

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large-sized particles (act as PSN) without considering whether the Cr dispersoids were in solution or not) [19], and fine grains, improved elongation/area reduction with equivalent strength were obtained. Later, a simple TMT process was developed by Wert using 674 k/8 h overaging instead of long-period step furnace cooling [20] and plenty of precipitates larger than 0.75 μm were formed to help the grain refinement (~ 10 μm in longitudinal and long transverse directions and ~ 6 μm in short transverse direction). The refined grains in 7075 alloy can contribute to super-plasticity/exfoliation corrosion resistance [20].

But a long-time heat preservation was still required to obtain a certain number of large particles for PSN action. Furthermore, it is difficult to accumulate sufficient storage energy by more than 80% deformation at low temperature for 7xxx series Al alloy plates [18], especially in the plate center and the large particles are hard to be redissolved completely, leading to the loss of tensile strength. Thus, a new cost-effective and practical TMT process, DTR, (including first stage warm rolling (pre-deformation DTR-20%), briefly intermediate annealing (BIA) and second stage hot rolling (DTR-80%)), was proposed to produce fine-grained 7055 Al alloy based on deformation enhanced precipitation. Our previous studies [21] indicated that the pre-deformation can spheroidize and refine the precipitates for increasing the drag forces to the boundaries and dislocations, and the optimized parameters for double step hot rolling were obtained. However, the microstructure evolution of each stage of the optimized DTR process and the final mechanical properties have not been studied as well as the specific mechanism for grain refinement. Hence, this study deals with the microstructure evolution during DTR processing 7055 Al alloy plates as well as the characterization of grain refinement, mechanical properties and the corresponding mechanism.

2. Experimental procedure

Commercial 7055 Al alloy plates (chemical composition (*wt*%): 8.38Zn-2.07Mg-2.31Cu-0.13Zr-0.16Ti-0.092Fe-0.056Si) with 15 mm thickness were solution treated (744 K × 16 h + 749 K × 8 h) in air furnace, and rapidly quenched into room temperature water (marked as SQ-7055). The SQ-7055 plates were then rolled by two routes (CTR: 674 K for 80% reduction 15 → 3 mm and DTR: 524 K for 20% reduction 15 → 12 mm, 704 K for 5 min (when the temperature is accurate, uniform and stable, put samples into the furnace) and 704 K for 75% reduction 12 → 3 mm) with two 270 mm-diameter rolls rotating at 15 rpm (Fig. 1). The rolled sheets were then recrystallized at 749 K for 0.5 h according to Refs. [20,22,23], using salt bath furnace with the sampling/transfer quenching time less than 5 s. The plates were heated with 2 K/min and held for 10 min before rolling and reheated for 5 min between passes to maintain constant rolling conditions.

Microstructures were observed with scanning electron microscopy (SEM, Zeiss Ultra 55) and transition electron microscopy (TEM, H800 and Tecnai G^2 F30). SEM samples were cut from ND-RD (normal direction and rolling direction) (cross-section) and mechanically polished. The resulting (sub)grain sizes/shapes and orientations were analyzed *via* electron back scattered diffraction (EBSD, Zeiss Ultra 55) and the software channel 5. Low angle grain boundaries (LAGBs) with misorientations θ among 2–15° and high angle grain boundaries (HAGBs) with misorientations $\theta > 15°$ were shown as gray and black lines in the EBSD maps, respectively. The EBSD samples sectioned in the ND-RD plane were electro-polished with the solution of 70% methanol and 30% nitric acid at 244 K (voltage: 30 V) after mechanically polishing. These samples were mounted on a pre-titled sample holder with a tilt angle of 70° (accelerating voltage: 20 kV, step size: 1 μm, scanning area: ~ 0.12 mm^2) and three random areas from the mid-thickness layer of plates were examined for the average value. The thin foils for TEM observation were prepared from 3 mm diameter discs cut in the ND-RD cross-section at the mid-thickness layer and ground to about 100 μm followed by twin-jet electropolishing with the above electropolishing solution (temperature: 244–254 K, potential difference: 30 V). The software 'imageJ' was used to analyze the sizes and volume fractions of precipitates.

Tensile tests were examined on a MTS-810 testing machine at room temperature with a nominal strain rate of $10^{-3}\,s^{-1}$. Tensile samples were cut into a dog-bone shape with a gauge length 25 mm and a gauge width 6 mm and were machined with their tensile axis paralleling to RD (rolling direction) according to ASTM E8-04. Three parallel samples were used to acquire consistent stress-strain curves and the average tensile/yield strengths and elongations.

3. Results and discussions

3.1. Microstructures and tensile properties of the rolled sheets after final recrystallization and T6 aging

Fig. 2 shows the typical recrystallization microstructure of the CTR-T6 alloy is very uneven with both elongated (11 μm in transverse and > 100 μm in length) and coarse equiaxed (~ 35 μm) grains, but the DTR-T6 alloy exhibits remarkable grain refinement (15 μm in transverse and 20 μm in length). A large number of HAGBs are formed in both CTR-T6 and DTR-T6 alloys but some LAGBs still appear in the grain interior (Fig. 2(a1, b1)), especially in the CTR-T6 alloy.

The grain orientation maps in Fig. 2 (a2, b2) indicate that (101) gains are dominant in the CTR-T6 alloy while they are relatively random in the DTR-T6 alloy. Moreover, the misorientation angle of the CTR-T6 alloy is mainly distributed among 2–15° and 45–60° while it is mainly distributed among 40–55° in the DTR-T6 alloy, as shown in Fig. 2(a3, b3), which indicates obvious recrystallization occurs in the DTR alloy leading uniform and refined grains. For the low storage energy in the CTR alloy, the recrystallization cannot be completely

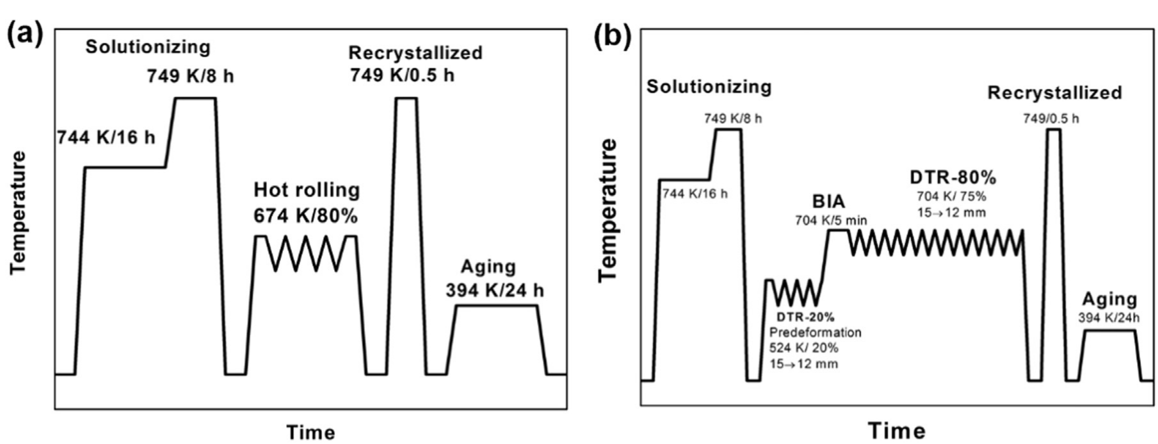

Fig. 1. Schematic presentations of TMTs: (a) CTR, (b) DTR.