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Improved solution treatment for an as-rolled Al–Zn–Mg–Cu alloy. Part II. Microstructure and mechanical properties

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ABSTRACT

In this work, improved solution treatments and their effects on the microstructure and mechanical properties of as-rolled Al–6.22Zn–2.11Mg–2.39Cu alloy have been investigated. By exploring the dissolution process of soluble constituent particles and monitoring changes in the grain structure, a solution treatment sequence with a good balance between a minimum volume fraction of remaining constituents and a partially recrystallised grain structure has been obtained. The results indicate that $MgZn_2$ (η) particles can be completely dissolved after holding at 475 °C for only 5 min, whereas the dissolution of Al_2CuMg (S) particles is relatively difficult and their complete dissolution requires a stepped solution treatment. By conducting a final solution treatment step at an increased temperature of 495 °C, all the S-phase particles can be dissolved, whereas the recrystallised fraction can still be controlled to be less than 50%. After retrogression and re-ageing (RRA), samples subjected to a final-stage solution treatment at 495 °C have a relatively higher volume fraction of η' and η precipitates and can therefore have better mechanical properties.

1. Introduction

Previous work has demonstrated that the main constituents in alloy AA7150 are $MgZn_2$ (η with a little Cu), Al_2CuMg (S), Al_7Cu_2Fe and Mg_2Si [1]. In addition, the main soluble particles were found to be $MgZn_2$ and Al_2CuMg, but Al_7Cu_2Fe and Mg_2Si particles were quite stable and insoluble during solution treatment [1]. According to differential scanning calorimetry (DSC) results, the melting temperatures of ($\alpha+\eta$) and ($\alpha+\eta+S$) were determined to be 478^{+1}_{-2} and 488^{+1}_{-2} °C, respectively.

Generally, when more than one soluble phase is formed during solidification, as found with Al–Zn–Cu–Mg and Al–Si–Cu–Mg alloys, it can be readily supposed that phases with lower melting points are melted locally during subsequent heating [2]. In practical aluminium alloys, a uniform solute-atom concentration is not realised during solidification owing to non-equilibrium solidification [2–6], resulting in the formation of eutectic compounds with a low melting point. Therefore, the applied homogenisation and solution heat treatment temperatures can sometimes exceed the final solidification temperature at which eutectic compounds are formed during solidification. To avoid the re-melting associated with the phase particles, industrially employed solution treatment temperatures are generally controlled below the melting temperature of phases with lower melting points. For example, the widely used solution treatment temperature range in the industry for alloy AA7150 is 471–482 °C [7], which is just around the melting point of ($\alpha+\eta+S$). However, this temperature can only dissolve η particles and has very slight effect on the dissolution of S particles. In previous work, the authors found that some coarse S-phase particles can still exist in alloy AA7150 even after 4 h holding at 485 °C [1]. Recently, it has been reported that by firstly dissolving the lower melting point phases in a multi-phase eutectic through stepped heating or lower temperature pre-treatment, it can increase the initial melting temperature of the remaining eutectics in alloy AA7055 [8]. Therefore, to effectively dissolve other soluble particles with higher melting points, a stepped solution treatment should be relatively applicable.

In the previous literature, it has been reported that the fracture resistance of Al–Zn–Mg–(Cu) alloys is very sensitive to the existence of any remaining coarse constituent particles that may act as void/crack initiation sites or provide preferential crack propagation paths and thus greatly degrade the fracture resistance [9]. In addition, these constituents can cause serious micro-galvanic corrosion when the alloys are exposed to aqueous environments [10], accounting for major maintenance issues in the aerospace sector with respect to corrosion fatigue [11]. Since these coarse particles consume much solute, the dissolution of these particles should be able to release extra solute for the formation of more strengthening precipitates during the ageing process. Therefore, the dissolution

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of existing soluble constituents is very helpful for improving the properties of 7xxx Al alloys.

Recently, Chen et al. [8,12,13] reported that the dissolution of constituent particles in as-extruded alloys AA7075 and AA7055 can be improved by using stepped solution treatments. However, the elevated solution temperature for the dissolution of constituent particles can accelerate the recrystallisation process. In alloy AA7050, a higher recrystallised fraction (≥50%) can significantly decrease the tensile properties [14]. Therefore, in order to efficiently make use of a solution treatment procedure to achieve an optimal combination of properties for 7xxx Al alloys, both the dissolution process of constituent particles and the recrystallisation process at different stages of a stepped solution treatment should be considered. However, so far no relevant literature could be found. In this work, the dissolution process of remaining constituent particles and changes in the recrystallised fraction at solution treatment stages from 475 to 495 °C have been investigated to obtain an improved solution treatment procedure in terms of mechanical properties of alloy AA7150.

2. Experimental procedures

The material used in this study was an 80 mm thick alloy plate AA7150 (6.22 Zn, 2.11 Mg, 2.39 Cu, 0.11 Zr, 0.09 Fe, 0.06 Si and the balance Al, in wt.%) in the hot-rolled condition, provided by Chalco. To obtain an optimal solution treatment procedure with a good balance between a minimum volume fraction of remaining constituents and a partially recrystallised grain structure, a series of stepped solution treatments from 475 to 495 °C were carried out on the as-rolled alloy AA7150. After solution treatment, all the samples were quenched into mineral oil at 20 °C and some were selected for subsequent retrogression and re-ageing (RRA), i.e. 120 °C/24 h + 185 °C/1.5 h + 120 °C/24 h.

The microstructures of as-polished samples cut from the quarter layer of the thick plate were checked using a JEOL 7001F Field Emission Gun Scanning Electron Microscope (FEG SEM). To quantitatively evaluate the dissolution of constituent particles, SEM images were taken at a magnification of 200× at least at three random sites for each sample. By using Image J software, the volume fraction of remaining constituents in the differently solution-treated samples was determined. After SEM observation, these as-polished samples were etched with Keller's etchant and grain structures were observed at least at three random sites using an optical microscope (OM) at a magnification of 200×. Based on a standard point counting technique [15], the recrystallised fractions occurring in differently solution-treated samples were calculated. In this study, those grains having an arbitrarily chosen aspect ratio of up to and including 1.75 are considered to be recrystallised, while the remainder are classed as unrecrystallised [16]. To confirm the validity of the above criterion, electron backscattered diffraction (EBSD) analysis for the calculation of the recrystallised fraction was also performed on the 475 °C/8 h condition.

The precipitates formed in the RRA-treated samples were analysed with a transmission electron microscope (JEOL 2011 TEM) operated at 200 kV. To quantitatively determine the relative volume fraction of precipitates in the RRA-treated samples, differential scanning calorimetry (DSC) was performed at least three times using a Perkin Elmer Diamond 6300 TG/DSC instrument at a heating rate of 10 °C/min. The detailed method for constructing baselines to measure peak areas from DSC curves can be referred to the previous work [1].

The final tensile sample geometry had a gauge length of 20 mm, a cross-sectional area of 4 mm × 3 mm and a radius between the gauge length and grip ends of 6 mm. Tensile testing in the short transverse direction (STD) was performed on a 100 kN screw-driven Instron materials testing machine (5500R/4505) at an extension rate of 1 mm/min at room temperature. Impact toughness testing was performed using Charpy specimens with dimension and orientation as shown in Fig. 1. Impact energies were measured with a pendulum-type Instron Dynatup instrumented impact tester at room temperature. To ensure the reliability of the measured data, three repeated tests were carried out for each condition. After testing, the fracture surfaces were observed using SEM with backscattered electron imaging.

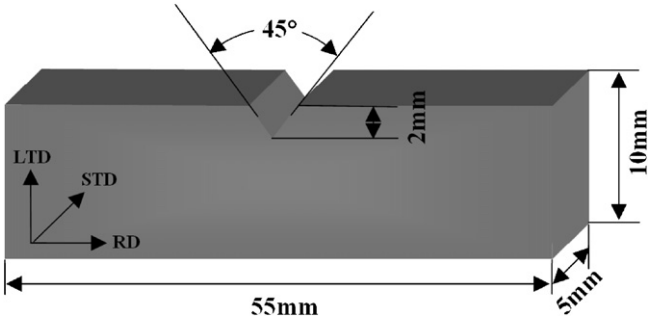

Fig. 1. Dimensions of the charpy samples used for impact fracture toughness testing, where RD is the rolling direction, STD is the short transverse direction, and LTD is the long transverse direction.

3. Results

3.1. Dissolution of constituent particles

Fig. 2 shows the volume fraction variation of remaining constituent particles over the different stages of a stepped solution treatment. After 5 min holding at 475 °C, the volume fraction of constituent particles in the alloy drops rapidly from 6.5 ± 0.3 to 3.5 ± 0.2%, as shown in Fig. 2(a). When the holding time increases to 8 h, the volume fraction decreases slowly from 3.5 ± 0.2 to 3.0 ± 0.1%. With a further increase of holding time, the volume fraction stabilises at about 3%. When holding at 485 °C after 475 °C/8 h, the volume fraction further decreases from 3.0 ± 0.1 to 1.3 ± 0.1% with increasing holding time to 4 h and decreases only slightly with further increases in holding time, as shown in Fig. 2(b). When holding at 495 °C after 475 °C/8 h + 485 °C/4 h, the volume fraction decreases from 1.3 ± 0.1 to about 0.8% with increasing holding time to 2 h and basically stabilises at this value with further increases in holding time, as shown in Fig. 2(c).

To understand the rapid drop of the volume fraction of constituent particles after 5 min holding at 475 °C, samples solution treated at 475 °C for 0, 1, 2.5 and 5 min were selected for SEM observation, as shown in Fig. 3. In previous work, the authors reported that in as-rolled AA7150, $MgZn_2$ particles (η with a little Cu) are small and appear bright and elongated, Al_2CuMg (S) particles are grey and rounded, Al_7Cu_2Fe particles are grey and irregularly shaped, and Mg_2Si particles appear dark and irregularly shaped in secondary electron images [1]. The results in Fig. 3 therefore demonstrate that only η particles can be dissolved during short time holding at 475 °C and that the dissolution of η particles can be completed after as little as 5 min at 475 °C, as shown in Fig. 3(d). Meanwhile, basically no changes can be observed for the other three types of constituent particles (Al_2CuMg, Al_7Cu_2Fe and Mg_2Si). This demonstrates that the rapid drop in volume fraction during short time holding at 475 °C is ascribed to the dissolution of η particles, and that the volume fraction of η particles in as-rolled AA7150 should be about 3%. Previous work indicated that Al_7Cu_2Fe and Mg_2Si particles were quite stable during solution treatment [1,17]. Therefore, further information about the dissolution behaviour of S particles can be obtained from Fig. 2.