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Improved solution treatment for an as-rolled Al–Zn–Mg–Cu alloy. Part I. Characterisation of constituent particles and overheating

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ABSTRACT

The main coarse constituents in as-rolled aluminium alloy AA7150 were determined by electron backscattered diffraction (EBSD) phase identification to be $MgZn_2$ (η), Al_2CuMg (S), Mg_2Si and Al_7Cu_2Fe. Based on differential scanning calorimetry (DSC) analysis and microstructural observations of single-step solution treated samples, the melting onset temperatures of (α + η) and (α + η + S) are 478^{+1}_{-2} °C and 478^{+1}_{-2} °C, respectively. However, Mg_2Si and Al_7Cu_2Fe particles are very stable and insoluble during typical solution treatments. When the re-melting of (α + η) or (α + η + S) occurs, holes will be left at the original sites of these particles. Since the size of the S-phase particles is larger than that of the η particles, the holes caused by the re-melting of (α + η + S) are relatively larger. When samples were rapidly heated up to 515 °C and solution treated at this temperature, network eutectics composed of $MgZn_2$, Al_2CuMg, Mg_2Si and α-Al were formed at the grain boundaries.

1. Introduction

Research about 7xxx series Al alloys has attracted much attention since these alloys can have a good combination of specific strength, hot workability, toughness and fatigue durability, depending on how they are processed [1,2]. Although the properties of 7xxx alloys are very attractive, the processing of these alloys is quite difficult due to their high alloying element content. Four major soluble constituent phases, i.e., η ($MgZn_2$), T ($Al_2Mg_3Zn_3$), S (Al_2CuMg) and θ (Al_2Cu) can typically exist in Al–Zn–Mg–Cu 7xxx series Al alloys of commercial interest [3,4]. It has been reported that the melting temperatures associated with η, T and S are about 475–478 °C [5,6], 482 °C [7] and 490–501 °C [7,8], respectively. Generally, it is widely accepted that the critical temperature for overheating is about 480 °C for alloys AA7055 and AA7150 [9–12]. Therefore, the conventionally used solution treatment temperature is normally between 470 and 480 °C [13–17]. However, only η particles can be typically dissolved at these temperatures, and a considerable quantity of other soluble constituents (especially S-phase particles) can still remain in these alloys [18–20]. Usually, these remnant constituents can degrade the age-hardening ability and aid crack initiation and propagation [21–24]. In addition, these constituents can cause micro-galvanic corrosion when the alloys are exposed to aqueous corrosive environments [25,26]. Therefore, the dissolution of existing soluble constituents is very important to obtain a good combination of properties.

To design a suitable solution treatment procedure for maximum dissolution of constituent particles in 7xxx alloys, one must know the critical meting temperatures of the existing constituent particles. So far, the melting temperatures of constituents are mainly determined by DSC analysis. However, the difference of measured temperatures from DSC results in the literature can vary by up to 10 °C [6,9–12]. Therefore, to accurately determine the melting temperatures of these constituents, only DSC results are not enough, and relevant microstructural checks should also be included. However, no comprehensive research work of this nature exists in the literature. In this work, the thermal stabilities of the existing coarse constituents in a hot-rolled Al alloy AA7150 have been investigated to determine their critical melting temperatures by using DSC analysis and subsequent microstructural confirmation. Additionally, overheating symptoms associated with different constituent particles have also been characterised on the basis of microstructural observations of single-step solution treated samples.

2. Experimental procedures

The material used in this study was high strength aluminium alloy AA7150 thick plate (6.22 Zn, 2.11 Mg, 2.39 Cu, 0.11 Zr, 0.09 Fe, 0.06 Si and balance Al, in wt.%), hot-rolled to 78 mm thickness by Chalco. Samples for microstructural observations were ground progressively up to 2500 grit emery papers followed by 3 and 1 μm DP-suspension polishing. To avoid the possible dissolution of

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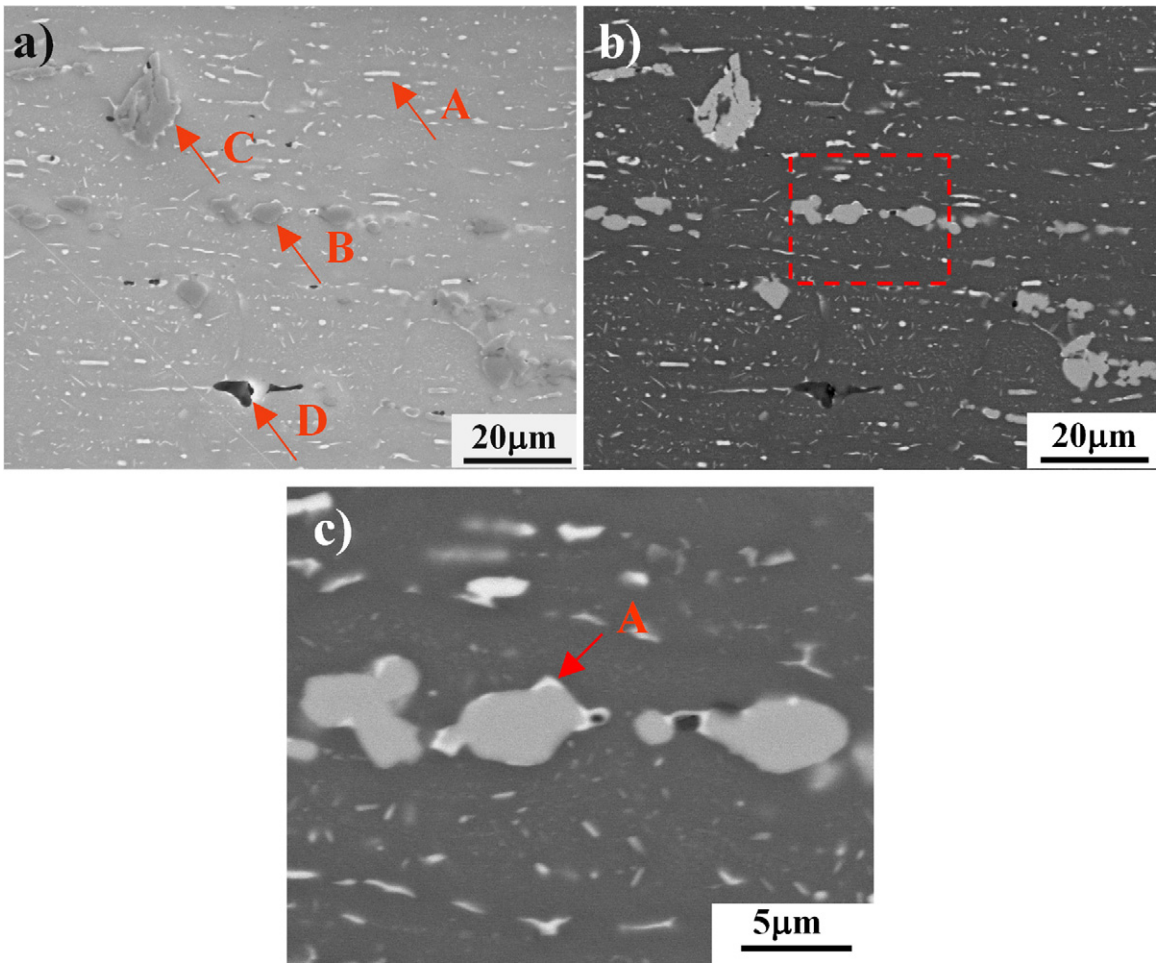

Fig. 1. Typical SEM images of as-rolled AA7150: (a) secondary and (b) backscattered electron images. Image (c) is a higher magnification image of the highlighted area in image (b).

some anodic constituents, ethanol was used as a lubricant during sample preparation. After that, the main constituent particles existing in the samples were identified using a JEOL 7001F Field Emission Gun Scanning Electron Microscope (SEM), equipped with a Bruker Quantax EDS System, Nordlys EBSD Detector and HKL Channel 5 Software. The procedure for the identification of constituent particles was as follows: (1) obtain an SEM image of relevant particles and corresponding quantitative EDS analyses, and (2) tilt the sample to the 70° orientation for EBSD pattern acquisition. During tilting, the same particles were kept in view.

To determine the melting temperatures associated with the constituent particles in the as-rolled sample, differential scanning calorimetry (DSC) was performed on a Perkin Elmer TG/DTA 6300 instrument at heating rates of 10, 20 and 50 °C/min. To ensure the reliability of the measured data, DSC analyses were also performed on pure Al (99.999%) discs for temperature calibration purposes. Samples for DSC analysis were punched into small discs 0.5 mm in thickness and 3 mm in diameter.

In order to confirm the melting temperatures measured from DSC curves, samples with a cross-section of $15 \times 15\,mm^2$ and a thickness of 5 mm were rapidly heated up and single-step solution treated at 475 °C for 8 h (475 °C/8 h), 480 °C for 4 h (480 °C/4 h), 485 °C for 4 h (485 °C/4 h), 490 °C for 2 h (490 °C/2 h), 505 °C for 1 h (505 °C/1 h) or 515 °C for 1 h (515 °C/1 h). Solution treatments were carried out in a salt bath and samples reached the set temperature within 30 s of immersion. During each solution treatment, extra thermocouples were used to ensure the reliability of the solution temperatures. After the solution treatments, all the samples were quenched into room temperature oil. Following that, the overheated microstructures were characterised by SEM on polished and impact-fractured surfaces of as-quenched samples.

3. Results

3.1. Phase identification of remaining constituents in the as-rolled condition

Fig. 1 shows the typical microstructure of the as-rolled AA7150. It reveals that many coarse constituent particles exist in the matrix. In addition, a large number of fine intermetallics of <1 μm in size are observed. These fine intermetallics are probably formed by precipitation during cooling after hot rolling. In this work, the focus is on the thermal stability of the coarse (>1 μm) particles. Finer intermetallics are noted to dissolve quickly during solution treatment [27], and discussion of this will not be included in any detail. Secondary and backscattered electron microscopy reveals that there are mainly four types of constituent particles in the matrix, as shown in Fig. 1(a and b). On the basis of their colour and shape (Fig. 1(a)), these particles are respectively labeled as constituents "A" (bright and elongated), "B" (grey and round), "C" (grey and irregular) and "D" (dark and irregular). Compositions of these constituent particles were analysed by EDS and are listed in Table 1. The higher magnification image in Fig. 1(c) indicates that some bright "A" particles (about 1 μm) can also be observed on the edges of the coarse grey "B" particles, as shown in Fig. 1(c).