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Partial dissolution of strengthening particles induced by equal channel angular pressing in an Al–Li–Cu alloy

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ABSTRACT

An Al–Li–Cu (AA2091) alloy was subjected to equal channel angular pressing (ECAP) at 210 °C following route B_C to a maximum strain of ε=8.64. A considerable particle age hardening anticipation with respect to an isochronal aging treatment was documented. At ε=6.48, the alloy reached a peak hardness level, while at ε=8.64, the alloy overaged and partial dissolution of the two most abundant hardening particles, T_1 plates and S' laths, was induced by ECAP/B_C.

1. Introduction

Aluminum–lithium alloys are of great interest in aerospace construction because of their low density and high strength, good stiffness and reasonable ductility [1,2]. Complex Al–Li alloys, such as the one examined in the present study, are subject to the precipitation of a variety of phases according to alloy composition, deformation state, and heat treatment conditions [3]. Cu and Mg are generally added to the Al–Li alloys to promote precipitation of T_1 (Al_2CuLi — hexagonal structure), δ′ (Al_3Li — tetragonal structure), θ′ (Al_2Cu — cubic structure), and S′ (Al_2CuMg — cubic structure) phases, depending on alloy composition and processing treatments [1,4,5]. T_1, δ′ and θ′ phase precipitates are generally within grains or at grain boundaries [5–7]. T_1 phase is usually the most common Al–Li alloy second phase. θ′ phase does precipitate for high Cu/Li alloy contents, δ′ phase appears upon relatively low temperature treatments [4,8]. The presence of S′ strongly depends on the thermo-mechanical history of the alloy, since it directly competes with the T_1 phase formation during alloy processing and following heat treatments. S′ fine precipitates tend to form homogeneously throughout the matrix [4,5]. The presence of these precipitates offers various benefits to the mechanical behavior of Al–Li–X alloys [9–12].

Other than simple heat treatments, it is also well understood that relatively low levels of prior deformation can accelerate and refine precipitation, thus improving mechanical properties [5,7]. Other than some research works dealing with the role of small amounts of prior strain (4–8%) on refining precipitation [5,7], few studies have examined the role of severe plastic deformation, by equal channel angular pressing (ECAP), on precipitation behavior and evolution [3,14–17]. ECAP in Al–Li alloys is generally carried out at elevated temperatures, 150 [13,14], 200 [13,14], 230 [15], 300 [13,16], or even 400 °C [3,17], because of the alloy high strength and low room temperature ductility. Indeed, high temperature plastic deformation, introduced after solutioning of Al–Li–Cu–X alloys, has been found to enhance the strength, ductility and aging kinetics over non-deformed material through the introduction of dislocations. These act as preferential matrix nucleation sites for the primary alloy strengthening phases

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[18–20]. With this respect, the dislocation density increment induced by ECAP is strongly dependant on deformation temperature and strain level. According to Muñoz-Morris and Morris [13,14], the dislocation density decreases by a factor of 3 for each 100 °C temperature rise, and increases to a lesser extent passing from 3 to 7 ECAP passes. Data reported in [13,14] on the hardening precipitation sequence induced by ECAP, revealed a complete precipitation to 15 nm-tick precipitates. These are likely to be further sheared to a smaller size by the following ECAP deformation levels. A mean hardening particle diameter of some 4 nm was reported for aging temperatures of 200 °C [13]. Particle number fraction changed very little upon ECAP at 200 °C. On the other hand, they found that particle volume fraction was larger in the case of ECAP at high temperature than by same temperature isochronal aging treatments.

The present work shows that high temperature severe plastic deformation through ECAP, up to $\varepsilon=8.64$ (8 passes), and using the so-called route B_C (that is, a +90 deg. sample rotation per pass) greatly anticipate the peak aging hardness at 210 °C and eventually induce partial dissolution of the two major strengthening T_1 and S′ phase particles.

2. Experimental Details and Method

2.1. Experimental Details

The alloy used for this study is an AA2091 (2.0Li, 2.1Cu, 1.4Mg, 0.09Zr, traces of Fe + Si). The alloy was homogenized at 500 °C, and then hot-rolled to plate thickness of 30 mm in a reversible hot-rolling mill. The plate was annealed and then cold-rolled to 10 mm-thick sheets in a reversible cold-rolling mill. Sheets of 10 mm were solution treated 1 h at 540 °C and then water quenched. Samples of length 160 mm and diameter 10 mm were machined from the plates, with the longitudinal (ECAP pressing) direction parallel to the original rolling direction. To homogenize the Al–Li–Cu cylindrical bars, samples were heated 6 min to the die temperature (210 °C) immediately prior ECAP pressing. ECAP was carried out at a speed of 40 mm/min, thus involving a 4 min duration of severe plastic deformation at 210 °C per pass. Thereafter, the overall aging duration time ranged from 10 min (after a single ECAP pass, that is, 6 min of isochronal aging plus 4 min into the die) to 38 min (after 8 passes). The cylindrical bars were pressed with forces ranging between 40 and 80 kN. ECAP die consisted of two blocks of SK3 tool steel (Fe–1.1%C), which were bolted together to give an L-shaped channel with a circular cross-section of 10 mm in diameter, consisting of two linear parts intersecting at an angle $\Phi=90°$ with a curvature extending over an angle $\Psi=20°$. With this die configuration, a true strain of $\varepsilon=1.08$ was imposed to the specimens at each pass [21,22].

The aging temperature (210 °C) was selected on the basis of earlier studies [7] on the solution hardening of such alloy series. Microhardness was measured using a load of 200 gf, and they were averaged over at least 11 different measurements, carried out in all tested conditions.

Thin foils for transmission electron microscopy (TEM) were first sliced on the extrusion-normal directions plane, then mechanically grinded down to ~150 μm and finally polished by twin-jet polishing method using an electrolyte of 25% nitric acid and 75% methanol at −20 °C, V = 22 V, in a Struers™ Tenupol-5® electropolisher unit. TEM observations were performed at 200 kV using a Philips™ CM20®, equipped with a double-tilt specimen holder. To analyze regions surrounding T_1 particles at $\varepsilon=8.64$, a Zeiss™ Supra 40® equipped with EDS microanalysis was used. Sample for the EDS microanalysis was polished to surface flatness and then chemically etched few seconds using a solution consisting of 30 pct. HNO_3 in methanol.

2.2. Method for Secondary Phase Particle Inspections

All precipitate sizes and numbers were determined from TEM images. Number density, size and volume fraction of the primary strengthening phases T_1, θ′, δ′, and S′ for each condition were obtained using point counting and stereological methods [23,24]. T_1 phase has a plate morphology and a hexagonal crystal structure with an orientation relationship of $(0001)_{T1}//\{111\}_{Al}$ and $<1010>_{T1}//<100>_{Al}$. θ′ phase precipitation occur as disks lying on the $(100)_{Al}$ planes and are semi-coherent with the matrix. The spherical δ′ precipitate has a very small lattice parameter mismatch with the matrix. It has a cube-on-cube $(111)_{\delta'}//(111)_{Al}$ orientation relationship with the matrix. S′ phase nucleates on dislocation loops and helices formed during quenching [20] and grows as laths on $\{210\}_{Al}$ planes along $<100>_{Al}$ directions [25]. Particles were quantified from transmission electron micrographs taken near $\mathbf{B}=[110]$. Particle size and number density were statistically determined using stereology methods of correction generally applied for TEM thin foils [23,24]. These are based on the precipitate size to thin foil thickness ratios. Stereological corrections were in fact necessary to account for particle truncation, especially for T_1 thin rods, S′ laths, and δ′ spherical particles.

3. Results and Discussion

Fig. 1 reports TEM images of the alloy immediately prior to plastic deformation (at 540 °C/1 h + 210 °C/6 min), and of three ECAP conditions, 1, 4, and 8 passes. The microstructure of the initial 210 °C/6 min aging treatment (Fig. 1a) shows cell and grain boundary nanometer δ′ (Al3Li) particles. Increasing amount of ECAP strain, from a strain of 1.08 (a single ECAP pass) to a strain of 4.32 (4 passes) and up to 8.64 (8 passes), led to a corresponding increase in the dislocation density, especially after the first four passes, that is, following the first cycle of shearing deformation induced by the route B_C [21]. Dislocation cells were elongated along a direction close to the pressing direction. Their sizes were 280 nm width and 830 nm long, at $\varepsilon=1.08$, which reduced to 95 nm per 870 nm, at $\varepsilon=8.64$ (8 passes). Correspondingly, the cell aspect ratio (length-to-width ratio) drastically increased from 2.96 to 9.16. Therefore, the high-temperature ECAP led to only a slight recovery of the substructure, which remained as dislocation cells, according to Muñoz-Morris and Morris [17]. Changes in strain path from pass to pass served to activate new slip systems so that the chances of annihilation of dislocations are reduced and new sets of excess dislocations are stored,