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Precipitation of $Al_3(Sc,Zr)$ particles in an Al–Zn–Mg–Cu–Sc–Zr alloy during conventional solution heat treatment and its effect on tensile properties

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Abstract

The effect of heat treatment on precipitation and growth of coherent nanometer-sized $Al_3(Sc,Zr)$ particles and the effect of these particles on tensile properties of a direct chill (DC) cast Al–Zn–Mg–Cu–Sc–Zr alloy were studied. The size distribution, average size, number density and volume fraction of the $Al_3(Sc,Zr)$ particles were determined as a function of the solution treatment temperature and time. An increase in the solution treatment temperature and time resulted in $Al_3(Sc,Zr)$ particles with a larger mean diameter, higher volume fraction and lower number density. The particle size distributions were described well by normal (Gaussian) distributions. The kinetics of the phase transformation followed the Kolmogorov–Johnson–Mehl–Avrami law, with the Avrami exponent $m = 0.404$. Room temperature tensile properties were evaluated in the as-solution treated and artificially aged conditions. The coherent nanometer-sized $Al_3(Sc,Zr)$ particles provided additional Orowan strengthening, which increased with increasing particle volume fraction and decreasing particle size, and varied from 75 to 118 MPa after different heat treatments.

Keywords: Aluminum alloys; Aging; Isothermal heat treatments; Phase transformation kinetics; Precipitation

1. Introduction

The aerospace industry has a substantial interest in new low-cost materials with improved specific strengths and resistance to fracture. 7000 series Al–Zn–Mg–Cu alloys possess the highest strength of all commercial Al alloys [1]. However, these alloys generally have poor ductility and low fracture strength in the as-cast condition, and extensive processing, which includes a combination of heat treatment and hot working, is required to improve the mechanical properties [2]. The fracture-related properties of these alloys can be considerably improved by small additions of Sc and Zr, which enable a superior combination of higher strength levels and acceptable ductility, in both cast and wrought conditions [3–10]. Starting from refining the grain size of cast aluminum alloys, additions of Sc and Zr also increase the resistance to recrystallization during hot working and introduce additional strengthening through the formation of fine coherent $Al_3(Sc,Zr)$ particles from a supersaturated solid solution [8–10]. Homogeneous distribution of these particles promotes formation of a stable refined subgrain structure during deformation processing, which gives an additional increase in strength [6]. Unfortunately, the equilibrium solubilities of Sc and Zr in the 7000 series alloys are very low at temperatures below the solidus temperature, which is typically in the range of 510–540 °C for these alloys. Therefore, the supersaturated solid solution of Sc and Zr, which is required to precipitate a high number density of nanometer-sized $Al_3(Sc,Zr)$

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particles in a cast-and-wrought aluminum alloy, can only be produced during casting with a relatively high cooling rate, such as direct chill (DC) casting. Accordingly, the first heat treatment conducted after the casting (in conventional alloys it is generally homogenization) becomes very important for precipitation of the $Al_3(Sc,Zr)$ particles with controlled size and number density, in order to achieve the best balance of mechanical properties, both in as-cast and wrought conditions [11].

There are several publications where kinetics of precipitation of the $Al_3(Zr,Sc)$ particles have been experimentally studied and also modeled for binary (Al–Sc and Al–Zr) [12–16] and ternary (Al–Sc–Zr) [17–19] alloy systems. No work has yet been reported on the kinetics of precipitation and growth of $Al_3(Sc,Zr)$ particles and the effect of the size and volume fraction of these particles on tensile properties in commercial heat-treatable aluminum alloys. In the present work, the kinetics of precipitation and growth of secondary $Al_3(Sc,Zr)$ particles were studied during homogenization/solution treatment of a DC-cast 7000 series alloy.

2. Experimental

A developmental 7000 series aluminum alloy, designated as SSA018, with the chemical composition shown in Table 1, was produced by semi-continuous DC casting in the form of a 76 mm diameter billet. Specimens for microstructural analysis and tensile testing were extracted via wire electro-discharge machining from near the mid-radius of the billet. Flat tensile specimens were produced with gauge dimensions of $20 \times 3.6 \times 2.5\ mm^3$ and the specimen tensile axis was parallel to the billet's long axis. The microstructure and tensile properties were studied in as-solution treated and aged conditions. Solution treatment was performed in air using a resistance heated box furnace with forced air convection. Different solution treatment (ST) temperatures (460 and 480 °C), heating rates (20 and 60 $°C\ h^{-1}$) and soaking times (1, 4, 20, and 48 h) were selected. After ST and water quenching, several specimens were subjected to artificial aging at 120 °C for 19 h.

Local chemical compositions were determined with 0.5% accuracy using electron probe microanalysis (EPMA) with CAMECA SX100. Microstructural analysis was performed using a Phillips CM200 transmission electron microscope (TEM) operating at an accelerating voltage of 200 kV. Thin TEM foils were twin-jet electropolished at −30 °C in a solution consisting of 20% HNO_3 and 80% CH_3OH. The sizes (diameters) and the number density of the $Al_3(Sc,Zr)$ particles were determined from several dark field (DF) TEM images taken at magnifications of ×38 000–150 000. These images were processed using Adobe Photoshop 7.0 and then analyzed using Clemex Vision Pro 3.5 image processing software. From 1000 to 16 000 particles were analyzed for every heat treatment condition (the number of counts increased with increasing particle number density) and the experimental error in the particle size, volume fraction, and number density measurements was about 5%, 10% and 10%, respectively.

Tensile tests were conducted at room temperature (RT, ~25 °C) using a servo-hydraulic MTS testing machine and a constant ram speed of 0.02 $mm\ s^{-1}$ (initial strain rate was $10^{-3}\ s^{-1}$). Two to five specimens per condition were tested. An extensometer attached to the specimen gauge was used to determine strain and total elongation. The experimental error in measurements of the stress and strain values was less than 1%.

3. Results

3.1. Microstructural analysis

In the as-cast condition, the SSA018 alloy billet had an equiaxed dendritic structure with the average grain size of 120 μm. This microstructure was described in detail elsewhere [8]. TEM analysis of the as-cast alloy revealed large eutectic-forming particles, which were enriched with Zn, Mg and Cu, and a dislocation network formed probably due to internal stress relaxation during casting (Fig. 1). $Al_3(Sc,Zr)$ particles were not detected in this condition, which indicates that both Sc and Zr were in a supersaturated solid solution.

During solution treatment, the Zn, Mg and Cu enriched particles rapidly dissolved. For example, after 1 h holding at 480 °C, in accord to the EPMA analysis, these elements were homogeneously distributed inside the Al matrix in the form of solid solution. Although some of Mg and Cu were still present in the form of Mg_2Si and Al_7Cu_2Fe particles after 1 h ST and these particles were not detected after 48 h ST, the concentration variations of Zn, Mg and/or Cu in the solid solution during the isothermal ST were no higher than 0.05–0.1 wt.% for each element. At the same time, fine, nanometer-sized, spherical $Al_3(Sc,Zr)$ particles with an Ll_2 cubic crystal structure were formed and grew during solution treatment (Figs. 2–4). Many regions where these particles were detected were free of dislocations, which might indicate homogeneous nucleation. The presence of Ashby–Brown contrast for bright-field images of these particles [21], as well as the common locations of the $(h,k,l)_{Al}$ and $(2h,2k,2l)_{L12}$ electron diffraction spots,

Table 1
Chemical composition (in wt.%) of SSA018 alloy

Element	Zn	Mg	Cu	Mn	Fe	Si	Zr	Sc	Al
Concentration (wt.%)	7.17	2.20	1.58	0.3	0.13	0.09	0.18	0.18	Balance
Concentration (at.%)	3.13	2.58	0.71	0.156	0.066	0.091	0.056	0.114	Balance