Hot Tensile Ductility and Dynamic Grain Growth in Submicron AI-Sc and AI-Mg-Sc Alloys

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Abstract

An Al-0.2Sc and Al-2.2-Mg-0.1Sc alloy (wt.%) were deformed by ECAP in the solution treated condition then aged for 3h at 350 °C to produce a submicron microstructure containing nanosized Al₃Sc particles. The alloys were then deformed in tension over a range of strain rates and temperatures. The addition of Mg to the binary alloy resulted in a higher ductility during testing at a given set of conditions. Despite the lower concentration of Sc in the ternary alloy, both the fine Al₃Sc particles and Mg in solution stabilises the initial fine grain size by impeding dynamic grain growth thereby resulting in higher ductility.

1. Introduction

Superplasticity is a phenomenon whereby very high ductility of up to several thousand percent elongation is possible in a material during tensile deformation [1]. Benefits of superplastic forming are numerous and include reduced weight, cost and energy consumption as well as improved component and system reliability. Superplasticity is known to occur in materials that have a very fine grain size, usually less than 10 μ m, that are deformed in a definite temperature-strain rate interval; as a rule, *T* = 0.5-0.6 *T*_m (*T*_m is the absolute melting temperature) and at strain rates in the range 10⁻³ – 10⁻⁴ s⁻¹ [1,2].

The past few years have seen a considerable interest in materials that have exceptional tensile ductility at low temperature and/or high strain rates. High strain rate superplasticity (HSRS) has been defined as a minimum tensile elongation of 200 % at strain rates greater than 10^{-2} s⁻¹ [2-5]. The essence of obtaining HSRS is to: (i) refine the grain size, and (ii) stabilise this grain size during deformation by the addition of fine, hard, dispersed particles that pin the grain boundaries and retard the tendency for strain-induced grain growth [5]. Nanocrystalline materials, which contain a very large density of high angle grain boundaries (i.e. the average misorientation between groups of adjacent grains is >15°), provide the potential to achieve superplasticity at much higher strain rates and lower temperatures than previous thought possible.

One useful method for producing submicron (nanocrystalline) grain sizes with a large fraction of HAGBs is by equal channel angular pressing (ECAP) [2,6]. The technique involves subjecting a material to severe plastic deformation by repeatedly shearing a billet under constrained conditions, in a die with two channels of identical cross section that meet at a particular angle, Φ .

For a range of submicron alloys produced by this processing route, Tables 1 and 2 summarise the notable studies of HSRS and low temperature superplasticity (LTS). It is clear that a number of alloys are capable of superplastic extensions at both high strain rates and low temperatures.

The aim of the present work was to use ECAP to produce a submicron starting microstructure in an Al-Sc and Al-Mg-Sc alloy and to study the influence of alloying additions on ductility and dynamic grain growth during hot tensile testing.

Ref.	Alloy	True Strain	Tensile Temperature (°C)	Strain Rate (s ⁻¹)	Tensile Elongation (%)
4	Al-6Cu-0.4Zr	12	300	1 x 10 ⁻²	970
7	AI-3Mg-0.2Sc	8	400	3.3 x 10 ⁻²	2280
7	Al-3Mg-0.2Sc	8	400	1 x 10 ⁻²	1500
8	5083AI + 0.2Sc	4	500	1 x 10 ⁻²	741
9	AI-5.5Mg-2.2Li-0.12Zr	8	400	1 x 10 ⁻²	1240
10	AI 2024	8	400	1 x 10 ⁻²	300
11	Al-3Mg-0.2Sc	8	400	1 x 10 ⁻¹	1080
12	Al-Mg-Li-Zr	12	350	1 x 10 ⁻²	1180
13	Cu-38.2Zn-3.1Sn	2	400	1 x 10 ⁻²	580
14	Cu-40Zn	1	400	1 x 10 ⁻²	640
15	Al-6Cu-0.4Zr	12	400	1 x 10 ⁻²	1070

Table 1: Comparison of some recent results for HSRS in submicron alloys produced by ECAP ($\dot{\varepsilon} \ge 10^{-2} \text{ s}^{-1}$).

Ref.	Alloy	True Strain	Tensile Temperature (°C)	Strain Rate (s⁻¹)	Tensile Elongation (%)
4	Al-6Cu-0.4Zr	12	300	1 x 10 ⁻²	970
9	AI-5.5Mg-2.2Li-0.12Zr	8	350	1 x 10 ⁻²	825
12	Al-Mg-Li-Zr	12	350	1 x 10 ⁻²	1180
16	Al-3Mg-0.2Sc	8	300	1 x 10 ⁻²	1280

2. Experimental Procedures

A binary Al-0.2Sc alloy and a ternary Al-2.2Mg-0.1Sc alloy (in wt.%) were produced by chill casting into 15 mm diameter ingot, swaged to 10 mm diameter, solution heat treated above their solvus (*Al-0.2Sc*: 24 h at 640 °C and *Al-2.2Mg-0.1Sc*: 24 h at 480 °C then 1h at 610 °C) then cold water quenched. For Al-Mg-Sc, selection of the solution treatment temperature was critical because incipient melting can occur at temperatures slightly above 610 °C [17].

To produce a submicron grain size in each alloy, solution treated billets of length 100 mm were deformed at room temperature by ECAP to an equivalent true strain of 9.2 for Al-Sc (8 passes) and 6.9 (6 passes) for Al-Mg-Sc. The ECAP rig had cylindrical bore and characteristic angles $\Phi = 90^{\circ}$ and $\psi = 0$ and pressing was carried out using graphite-based lubricant at a ram speed of 10 mm/min with 90° rotation between passes (termed route B_c [18]). Following deformation, both alloys were aged for 3h at 350 °C in order to precipitate very fine (< 10 nm) spherical particles of Al₃Sc onto the deformation substructure and, hence, produce a stable grain size through the well-known concept of Zener pinning [19].

For mechanical testing, cylindrical tensile test specimens of diameter 4 mm and gauge length 20 mm were machined from the deformed and pre-aged billets. Each specimen was heated to a given test temperature in 1800 s within an MTS Environmental Chamber, held for 600 s at this temperature in order to reach thermal equilibrium then strained to failure using an MTS hydraulic tensile machine. The testing conditions used in this work are given in Table 3.

Alloy	Deformation Temperature (℃)	Strain Rate (s ⁻¹)	Tensile Elongation (%)
AI-0.2Sc	350	1 x 10 ⁻³	163
	400	1 x 10 ⁻³	216
	400	1 x 10 ⁻²	143
	450	1 x 10 ⁻⁴	161
AI-2.2Mg-0.1Sc	300	1 x 10 ⁻³	429
	300	5 x 10 ⁻³	249
	300	1 x 10 ⁻²	34
	350	1 x 10 ⁻²	183

Table 3: Tensile testing conditions and elongation to failure.

After both pre-ageing and tensile deformation, grain size and particle/grain boundary interactions were investigated using scanning electron microscopy (SEM), focussed ion beam microscopy (FIB) and transmission electron microscopy (TEM). The information was used to assess the stability of the submicron structures produced by ageing and during tensile testing. A detailed description of the SEM, FIB and TEM procedures is given in ref. [20].

3. Results and Discussion

3.1 Pre-aged Microstructures

Figure 1a shows a typical microstructure of AI-0.2Sc following ECAP and pre-ageing for 3h at 350 °C. An equiaxed grain structure is generated with an average grain diameter of ~800 nm with grains exhibiting a large spread in orientations, as indicated in the selected area electron diffraction (SAED) pattern. For the same post-deformation heat treatment, Figure 1b shows a typical microstructure of AI-2.2Mg-0.1Sc which also shows an equiaxed grain structure but with an average grain diameter of ~520 nm. Again, the SAED pattern indicates that grains exhibit a large spread in orientations. The influence of Mg in solid solution in AI is known to reduce the rate of dynamic and static recovery [7] which, despite the lower strain by ECAP, results in additional grain refinement in the Mg-containing AI alloy.

Following the pre-aging treatment, a dispersion of nanosized AI_3Sc particles was observed by TEM with the interaction between particles and both dislocations and grain boundaries a common feature of the microstructures in both alloys. Figure 2 shows a large number of AI_3Sc particles both within and decorating the boundaries of recovered grains in the AI-2.2Mg-0.1Sc alloy. After 3h at 350 °C, the particles are below the critical size range (16-30 nm) where full coherency is expected to be lost [21,22]. Therefore, the majority of particles present in both alloys after pre-aging are expected to be, at least, semi-coherent.



Figure 1: Bright field (BF) TEM micrographs and SAED pattern of a representative region of microstructure of (a) AI-0.2Sc (courtesy of N. Hamilton) and (b) AI-2.2Mg-0.1Sc after pre-ageing for 3h at 350 °C (samples sectioned perpendicular to extrusion axis).



Figure 2: BF TEM micrograph of Al-2.2Mg-0.1Sc pre-aged at 350° C showing Al₃Sc particles both within grains and interacting with grain boundaries.



Figure 3: BF TEM micrograph of Al-0.2Sc pre-aged at 350 $^{\circ}$ C and strained to failure (216% elongation) at a strain rate of 10⁻³ s⁻¹ at 400 $^{\circ}$ C

3.2 Tensile Elongation in Al-Sc and Al-Mg-Sc Alloys

The present study has shown that the largest tensile elongations were achieved in the ternary alloy, although it contained only half the concentration of scandium. The highest ductility was achieved in this alloy at a strain rate of 10^{-3} s⁻¹ at 300 °C. By comparing this result with those given in Tables 1 and 2, it is clear that, while the present alloy is very dilute, the achievement of 429 % elongation at such a low temperature is a significant result.

The results also indicate that AI-0.2Sc has a lower ductility than AI-2.2Mg-0.1Sc, for a given set of processing conditions, which indicates that Mg has a strong influence on the ductility of these alloys. These results are comparable with recent work on similar alloys where it was found that AI-3Mg-0.2Sc alloy exhibited the most remarkable superplastic extension [7] (see *e.g.* Table 1). Similar to the present alloy, these alloys exhibited a submicron grain size containing a dispersion of fine Sc-rich particles [7]. It is known that HSRS or LTS is achievable in these alloys if a fine grain size can be produced that resists strain-enhanced grain growth via the generation of fine, stable particles at grain boundaries.

3.3 Grain Stability during Tensile Deformation

Single-phase submicron grain structures are highly unstable at high temperatures due to the large amount of energy associated with grain boundaries [23]. During processing or holding at elevated temperature, energy will be released by the reduction in grain boundary area and the fine structure will be replaced by a coarse structure by either continuous or discontinuous subgrain growth [23,24]. Thermal stability of the alloy can be realised if a dispersion of stable second phase precipitates is present in the microstructure. These particles impede grain coarsening at high temperature via grain boundary pinning.

Despite the presence of a large volume fraction of fine particles in the AI-Sc alloy, hot tensile deformation resulted in dynamic grain growth. Figure 3 is a TEM micrograph showing an increase in the average grain diameter from 0.8 μ m (Figure 1a) to 1.8 μ m after tensile straining to 216 % elongation at a strain rate of 10⁻³ s⁻¹ at 400 °C. This result may be compared with the unstrained AI-0.2Sc alloy isothermally annealed for 2h at 400 °C where the grain size increased to just over 1 μ m [25].

To further study the influence of deformation on dynamic grain growth, fractured samples were cold mounted, sectioned and electropolished then examined by ion channeling contrast (ICC) imaging using FIB microscopy. The micrographs in Figure 4 show the orientation distribution of grains in the as-deformed AI-0.2Sc alloy. It is clear that some coarsening has occurred along the length of the tensile specimen, increasing from 1.5 μ m at the undeformed (grip) region to ~3.7 μ m near the fracture zone (tip). The homogeneity of the grain size across each of the microstructures indicates that discontinuous (sub)grain growth has not occurred. This work has also shown that the ternary alloy undergoes less dynamic grain coarsening (se *e.g.* [7]); a necessary prerequisite for achieving large elongations during tensile straining [7,17].

4. Conclusions

An Al-0.2Sc and Al-2.2-0.1Sc alloy (wt.%) were deformed at room temperature by ECAP in the solution treated condition then pre-aged for 3h at 350 °C to produce a submicron microstructure containing nanosized Al₃Sc particles. It was found that the addition of Mg to Al-Sc results in higher ductility during high temperature tensile testing. Despite the lower concentration of Sc in the Al-Mg-Sc alloy, Mg in solid solution has an additional stabilising effect by impeding dynamic grain growth during tensile straining. This is a necessary requirement for achieving large superplastic elongations [7,17].

FRACTURED REGION



Figure 4: ICC FIB micrographs of the AI-0.2Sc alloy after tensile straining to failure at 10⁻³ s⁻¹ at 400 °C showing dynamic grain growth in the region exhibiting the greatest amount of deformation (note: figures were generated at different magnifications).

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