Proceedings of the 9th International Conference on Aluminium Alloys (2004)
Edited by J.F. Nie, A.J. Morton and B.C. Muddle

Particle Pinning in a Nanocrystalline Al-Sc Alloy

N.E. Hamilton, M. Ferry

School of Materials Science and Engineering, University of New South Wales
UNSW, Sydney, NSW, 2052, Australia

Keywords: aluminium, ECAP, microstructure, grain growth, Zener pinning

Abstract

A submicron grained microstructure in an Al-0.2 wt.% Sc alloy was produced by Equal Channel Angular Pressing (ECAP). The alloy was solution treated prior to deformation, deformed to ε = 9.2 by ECAP then aged at 350 ^{o}C to produce a fine grained microstructure exhibiting a large fraction of high angle grain boundaries and a dispersion of nanosized Al_3Sc particles. The work has demonstrated that the fine-grained microstructure was remarkably stable during annealing at temperatures up to 500 ^{o}C due to pinning from highly stable Al_3Sc particles.

1. Introduction

The term *nanocrystalline* is often used for alloys with microstructures that are extremely fine, with grain sizes in the order of tens to hundreds of nanometers [1]. The fine-grained microstructures present in nanocrystalline alloys are responsible for several beneficial property improvements; including improvements in strength, toughness and superplasticity [1-4]. Many techniques have been employed to produce nanocrystalline microstructures, with the most recent investigations achieving such structures through severe plastic deformation (SPD) [2-4]. One of the reasons that SPD has received considerable attention is that prior methods (such as rapid solidification or powder metallurgy) have limited scope for extension into large-scale production since they produce highly restrained quantities of the desired product. Equal Channel Angular Pressing (ECAP) is typically capable of producing uniform, refined microstructures at the submicron level [2], independent of sample size, making them of significant interest for extension into standard industry practice [5].

A notable property improvement resulting from a submicron grain size is enhanced superplasticity [2]. However, the ‘Achilles heel’ of this property is that it often requires processing at high homologous temperatures and, under normal circumstances, this provides enough thermal agitation for rapid discontinuous coarsening of the microstructure [6], thereby losing the very structure required for superplastic processing. Therefore, a fine-grained microstructure is not without its limitations, one being its vulnerability towards recrystallization and grain growth which are driven by the desire to reduce the grain boundary energy of the system [4]. In 1948 [7], Zener proposed that a microstructure containing particles can be prevented from undergoing such grain coarsening beyond a certain critical limiting size which was dependent on both particle radius and volume fraction [7,8]. The concept of Zener pinning is of particular importance to nanocrystalline alloys since the many property improvements (arrived at as a result of grain refinement) can be maintained at elevated temperatures via the introduction of a thermally-stable particle dispersion.

The aim of this work was to take an Al-0.2 wt.%Sc alloy processed by ECAP and, through the formation of a fine dispersion of Al_3Sc particles, study the influence of the particle dispersion on grain growth.

2. Experimental Procedure

A high purity Al-Sc alloy containing 0.2 wt.% Sc was chill cast to produce 15 mm diameter ingot, swaged to 10 mm diameter then solution heat treated above the solvus for 48 h at 620 ^{o}C and cold water quenched after solution treatment. Samples of length 100 mm were then deformed at room temperature by ECAP to an equivalent true strain of 9.2. The ECAP rig had characteristic angles $\Phi = 90^{o}$ and $\psi = 0$ and pressing was carried out using graphite-based lubricant at a ram speed of 10 mm/min to a total of 8 passes with 90^{o} rotation between passes (termed route B_c [9]).

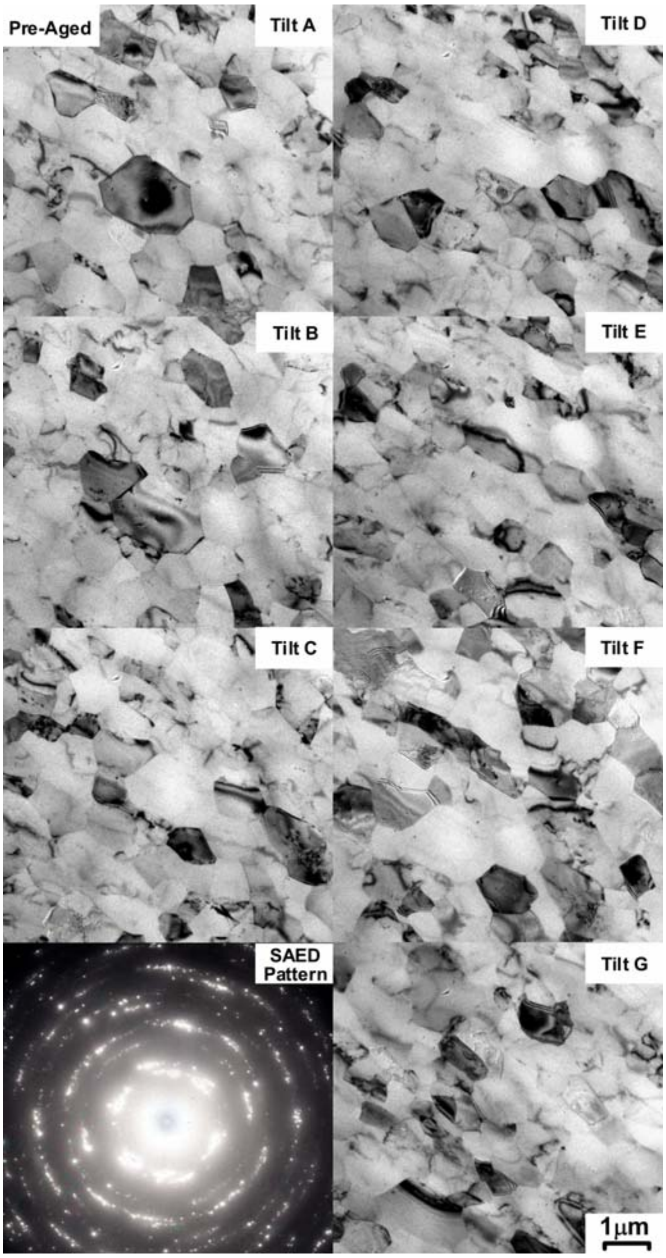
Following deformation, the alloy was pre-aged for 3h at 350 ^{o}C. The Al-rich end of the Al-Sc phase diagram is characterised by a eutectic point at 655 ^{o}C and 0.5 wt.% Sc and a maximum solubility of 0.35 wt.% Sc at this temperature [10]. The solid solubility is markedly reduced with decreasing temperature. Therefore, on ageing at low temperatures, this alloy system is expected to precipitate spherical particles of coherent Al_3Sc by homogeneous nucleation and growth [11]. The presence or absence of a dislocation substructure was shown previously to have no marked influence on precipitation behaviour [10]. Therefore, the aim of this pre-ageing treatment was to produce a stable grain size via the precipitation of fine (< 10 nm) particles onto the deformation substructure.

After pre-ageing, samples were annealed isothermally for up to 7200 s at temperatures of 400, 450 and 500 ^{o}C. The kinetics of grain growth and particle/grain boundary interactions were investigated using scanning electron microscopy (SEM), focused ion beam (FIB) microscopy and transmission electron microscopy (TEM). A detailed description of these procedures is given in ref. [12].

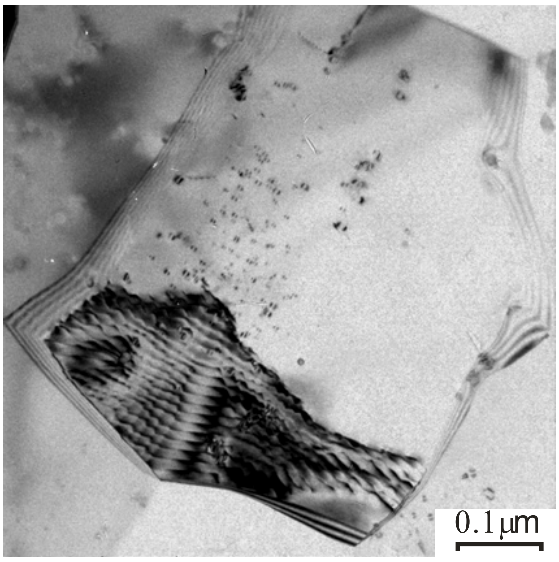
3. Results and Discussion

3.1 Grain Stability during Pre-Ageing and Subsequent Annealing

Figure 1a shows the microstructure following deformation and pre-ageing for 3h at 350 ^{o}C which shows an equiaxed grain structure of average radius 400 nm. The series of micrographs at various tilts illustrates the distribution of grain orientations as well as the very fine, equiaxed grain size. The large spread in orientation of the grains is confirmed by the SAED pattern in Figure 1a which also implies that a large fraction of high angle grain boundaries is generated by ECAP and pre-aging. While the Al_3Sc particles are not resolvable in the low magnification micrographs, Figure 1b shows nanosized Al_3Sc particles within a recovered grain and some slightly coarser particles at grain boundaries.



(a)



(b)

Figure 1: (a) Series of Bright field TEM micrographs at various tilt angles and the SAED pattern of a representative region of microstructure after pre-ageing for 3h at 350 ^{o}C (samples sectioned perpendicular to extrusion axis, ED). (b) Bright field TEM micrograph of microstructure after pre-ageing for 3h at 350 ^{o}C showing fine coherent Al_3Sc particles within a recovered grain and slightly coarser particles on grain boundaries.

The effect of annealing time on grain coarsening at elevated temperatures (400 to 500 ^{o}C) is given in Figure 2 with grain sizes determined by linear grain size measurements on six TEM micrographs per annealing condition. It is clear that grain growth is extremely limited, particularly at temperatures below 500 ^{o}C.

Figure 3 shows representative TEM micrographs during various stages of annealing at 450 ^{o}C which demonstrates the high stability of the initial fine-grained structure with only gradual and uniform coarsening of the microstructure. During annealing at temperatures greater than 500 ^{o}C [12] or for long annealing times at 500 ^{o}C (> 1h), there was evidence that discontinuous grain coarsening, i.e. recrystallization, eventually replaces such continuous coarsening.

Figure 2: The effect of annealing time on grain radius during annealing at 400 to 500 ^{o}C.

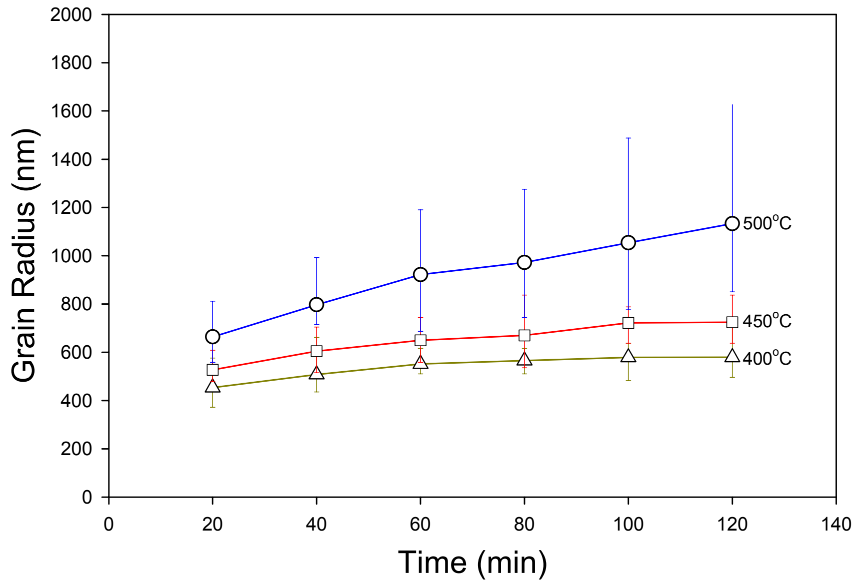
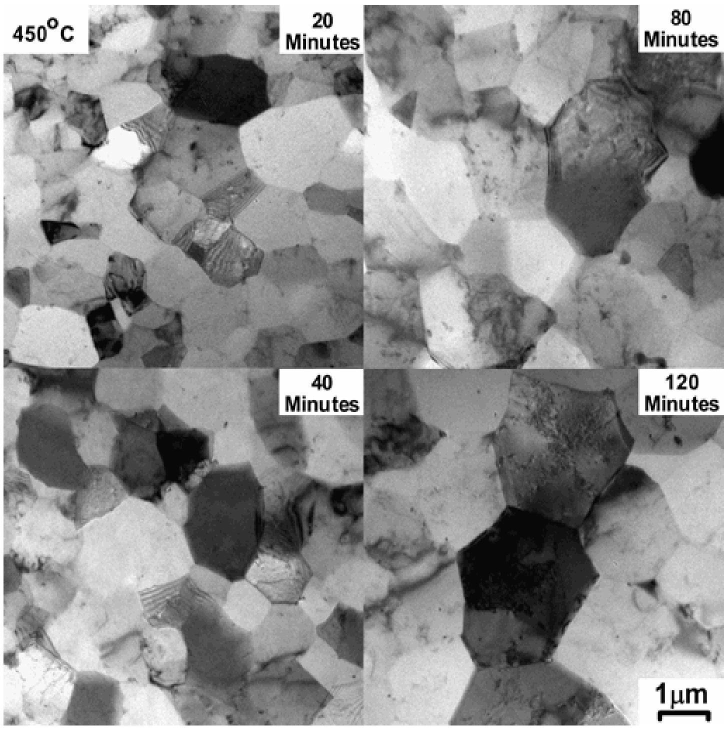


Figure 3: Bright field TEM micrographs during various stages of annealing at 450 ^{o}C (samples sectioned perpendicular to the extrusion axis).



3.2 Particle Interactions with Dislocations and Grain Boundaries

Following annealing at various temperatures, a dispersion of nanosized Al_3Sc particles was observed by TEM. Figure 4 shows a large number of Al_3Sc particles within a recovered grain, with coherency characterised by the presence of mushroom-shaped strain fields as a result of the anisotropy in their coherency [13], and some larger, particles present on grain boundaries. At the highest annealing temperature (i.e. 2h at 500 ^{o}C), the average particle radius is predicted to be ~15 nm which is below the critical range where full coherency is expected to be lost [10,13]. Therefore, the majority of particles present in Al-0.2Sc during annealing at temperatures up to 500 ^{o}C are expected to remain, at least, semi-coherent.

The interaction between particles and both dislocations and grain boundaries was commonly observed during annealing. Figure 5 is a bright field TEM micrograph showing the interaction between particles and the recovering substructure where particles are clearly located on individual dislocations, grain faces and grain edges.

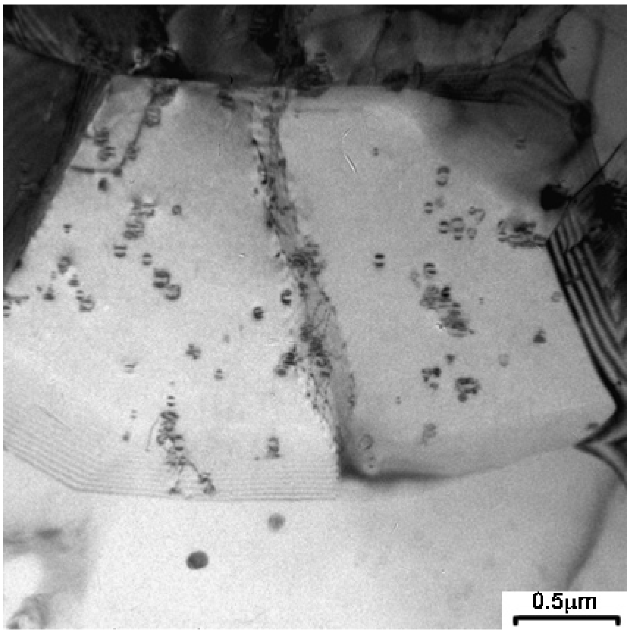


Figure 4: Al-0.2Sc pre-aged for 3h at 350°C and annealed for 2h at 500 °C showing coherent particles within a recovered grain and coarser particles interacting with grain boundaries.

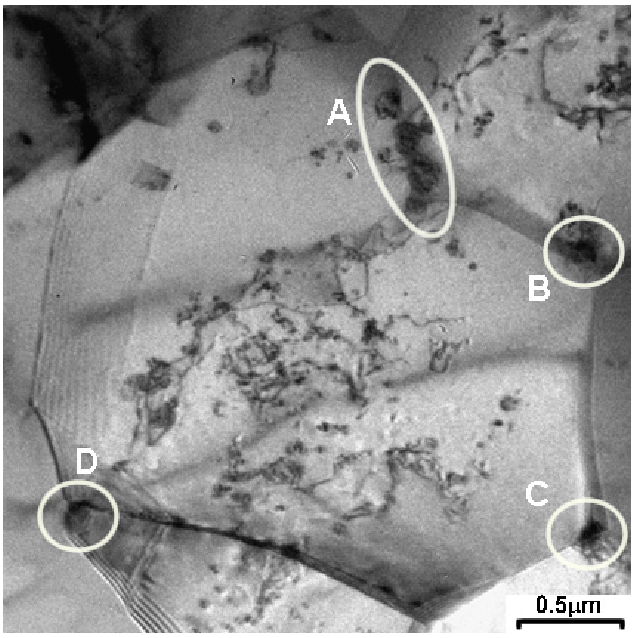


Figure 5:Al-0.2Sc pre-aged for 3h at 350°C and annealed for 1h at 500 °C. Particle pinning is evident at point A (4 particles) as well as B, C and D (1 particle each at triple points).

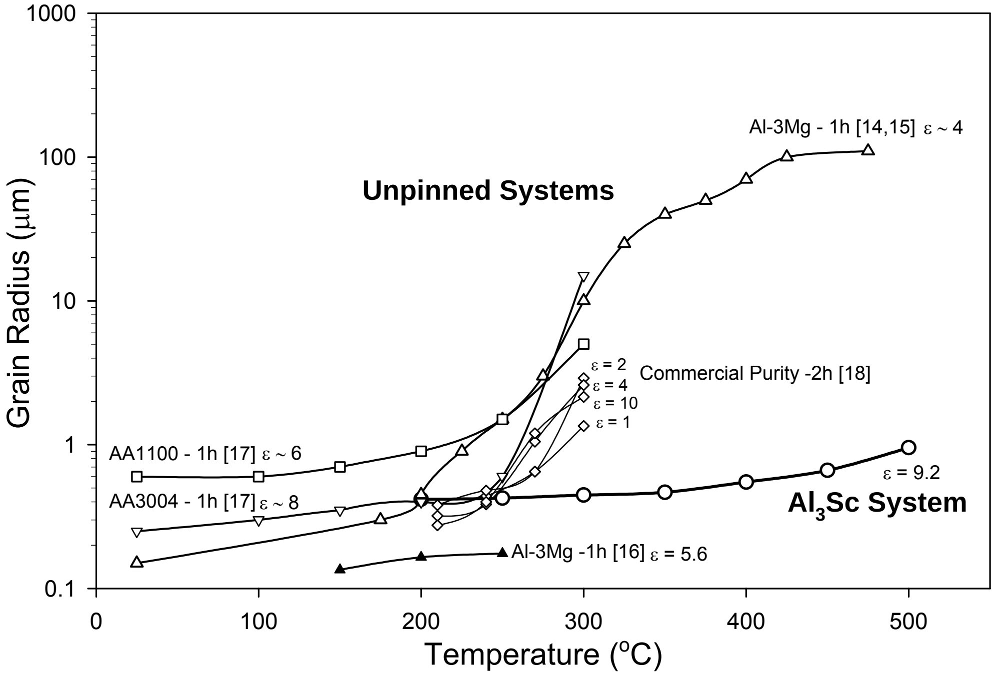


Figure 6: Grain radius as a function of annealing temperature (1h and 2h annealing at a given temperature) for Al-0.2Sc and various commercial Al alloys produced by ECAP.

3.3 Kinetics of Grain Growth in Severely Strained Al Alloys

It is useful to compare the thermal stability of the Al-0.2Sc system with other non-pinning alloy systems processed by ECAP. Figure 6 shows the effect of annealing temperature on the average grain radius for the present alloy together with published data on Al-3Mg [14-16], AA3004 and AA1100 Al alloys [17] and a commercial purity 99.5% Al alloy [18]. These alloys were also produced by ECAP to varying true equivalent strains ranging from 1-10. It is clear that, while the Al-Sc alloy is very dilute, it is remarkably stable compared with other Al alloys with the grain size remaining essentially constant up to high temperatures.

4. Conclusions

An Al-0.2 wt.% Sc alloy was deformed at room temperature in the solution treated condition to a true strain of 9.2 by equal channel angular pressing then pre-aged at 350 ^{o}C to produce a submicron grained microstructure containing nanosized Al_3Sc particles. It was found that the structure was highly stable during annealing at temperatures up to 500 ^{o}C with very limited continuous grain growth due to Zener pinning from the highly stable Al_3Sc particles.

Acknowledgements

This work is supported by the Australian Research Council (ARC) under the ARC Discovery grants scheme (Project ID: DP0342766).

References

[1] H. Gleiter: Acta Mater. 48 (2000) 1.
[2] R.Z. Valiev, R.K. Islamgaliev and I.V. Alexandrov: Prog. Mater. Sci. 45 (2000) 103.
[3] F.J. Humphreys, P.B. Prangnell, J.R. Bowen, A. Gholinia and C. Harris: Phil. Trans. Royal Society A. 357 (1999) 1663.
[4] F.J. Humphreys and M. Hatherly: *Recrystallization and Related Annealing Phenomena*, 2nd edition. Pergamon Press, Oxford, UK (2004).
[5] Z. Horita, T. Fujinami and T.G. Langdon: Mater. Sci. Eng A. 318 (2001) 34.
[6] M. Ferry and F.J. Humphreys: Acta Mater. 44 (1996) 1293.
[7] C.S. Smith (private communication with C. Zener): Trans. AIME, 175 (1948) 15.
[8] P.A. Manohar, M. Ferry and T. Chandra: ISIJ International. 38 (1998) 913.
[9] M. Furukawa, Z. Horita, M. Nemoto and T.G. Langdon: Mater. Sci. Eng A. 324 (2002) 82.
[10] M.J. Jones and F.J. Humphreys: Acta Mater. 51 (2003) 2149.
[11] Y. Miura, T. Shioyama and D. Hara: in *Proc. 5th Int. Conf. on Aluminium Alloys*, eds J.H. Driver, Genable, France. vol. II (1996) 505.
[12] N.E. Hamilton: *B.Eng Thesis*. University of New South Wales (2003).
[13] S. Iwamura and Y. Miura: Acta Mater. 52 (2004) 591.
[14] M Furukawa, Y. Iwahishi, Z. Horita, M. Nemoto and T.G. Langdon: Mater. Sci. Eng A. 257 (1997) 328.
[15] M. Furukawa, Z. Horita, M. Nemoto, R.Z. Valiev and T.G. Langdon: in *Proceedings of Recrystallization 96*, TMS, USA (1996) 149.
[16] D.G. Morris and M.A. Munoz-Morris: Acta Mater. 50 (2002) 4047.
[17] Z. Horita, T. Fujinami, M. Nemoto and T.G. Langdon: J. Mater. Proc. Tech. 117 (2001) 288.
[18] W.Q. Cao, A. Godfrey, W. Liu and Q. Liu: Mater. Lett. 57 (2003) 3767.