Materials Science and Engineering A 552 (2012) 415–418

Contents lists available at SciVerse ScienceDirect

Materials Science and Engineering A

journal homepage: www.elsevier.com/locate/msea

Enhanced grain refinement of an Al–Mg–Si alloy by high-pressure torsion processing at 100 °C

K. Tugcu[a], G. Sha[a,c,*], X.Z. Liao[a], P. Trimby[b], J.H. Xia[a], M.Y. Murashkin[d], Y. Xie[a], R.Z. Valiev[d], S.P. Ringer[a,b,c]

[a] School of Aerospace, Mechanical & Mechatronic Engineering, The University of Sydney, NSW 2006, Australia
[b] Australian Centre for Microscopy & Microanalysis, The University of Sydney, NSW 2006, Australia
[c] ARC Centre of Excellence for Design in Light Metals, The University of Sydney, NSW 2006, Australia
[d] Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, 12K. Marx Street, Ufa 450000, Russia

ARTICLE INFO

Article history:
Received 25 April 2012
Received in revised form 10 May 2012
Accepted 22 May 2012
Available online 29 May 2012

Keywords:
Aluminium alloy
Grain refinement
HPT
Segregation
TEM

ABSTRACT

Ultrafine-grained structures were obtained in a solution-treated Al–Mg–Si (6060) alloy processed by high-pressure torsion at ambient temperature, 100 °C and 180 °C. Processing at 100 °C provided the highest hardness and the smallest average grain size because of the enhanced segregation of solutes at grain boundaries which hindered grain growth during the processing. This research demonstrated the importance of optimising processing temperature to enhance grain refinement in high-pressure torsion processing.

1. Introduction

High-pressure torsion (HPT), which is an effective severe plastic deformation (SPD) technique for grain refinement, has been widely used to produce bulk ultrafine-grained (<1 μm) and nanostructured (<100 nm) metals and alloys for superior mechanical properties [1–6]. SPD-induced grain refinement is mainly caused by dislocation activities and, in some systems, deformation twinning [7,8]. There is always a minimum average grain size that a specific SPD process can achieve in a specific material. This minimum size is a function of both intrinsic material properties including stacking fault energy [9,10] and extrinsic processing parameters [10]. One of the reasons that produce this minimum grain size is that SPD processes also result in dynamic grain growth. The minimum grain size is achieved by the dynamic balance between the grain refinement process and the grain growth process [11].

The effects of several extrinsic processing parameters, including specimen dimensions [12], applied load [13] and imposed strain [14], on the HPT-induced grain refinement have been extensively investigated. In contrast, because most previous HPT experiments were performed at ambient temperature, there is a lack of systematic experiments to investigate the effect of HPT processing temperature on grain refinement. Because temperature affects significantly the deformation behaviour and dynamic grain growth of SPD materials [15,16], it is expected that SPD temperature is also an important extrinsic parameter affecting the final microstructure and that increasing SPD temperature will in general result in larger final grain sizes [17,18]. However, the temperature effect for SPD alloys could be complicated because temperature affects significantly the solute behaviour in the alloys and this subsequently affects the SPD-induced dynamic grain growth. Furthermore, the temperature effect on the behaviour of some solute elements may be significantly strengthened by the SPD processing because of the excessive amount of vacancies induced by the SPD [19]. In this letter, we present interesting results that HPT of a solution-treated 6060 aluminium alloy at 100 °C yields the highest hardness and smallest average grain size compared to HPT processing at room temperature (RT) and 180 °C. Extensive transmission electron microscopy (TEM) characterisation reveals that this is closely related to the distribution of solutes in the alloy at different temperatures.

2. Experimental procedures

The as-received 6060 aluminium alloy was in the form of an extruded rod with a composition of Mg 0.6, Si 0.6, Cr 0.5, Fe 0.1–0.3,

* Corresponding author at: School of Aeospace, Mechanical & Mechantronic Engineering, The University of Sydney, NSW 2006, Australia. Fax: +61 293517682.
E-mail addresses: gang.sha@sydney.edu.au (G. Sha), xiaozhou.liao@sydney.edu.au (X.Z. Liao).

0921-5093/$ – see front matter
http://dx.doi.org/10.1016/j.msea.2012.05.063

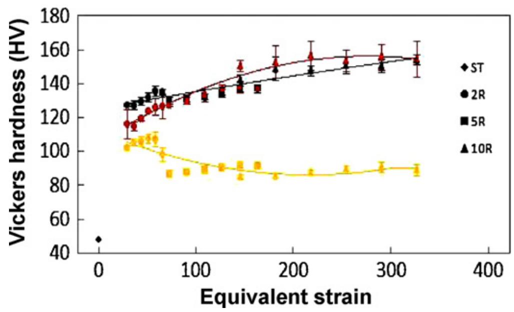

Fig. 1. (Colour online) Vickers hardness plotted against the von Mises equivalent strain for samples processed by HPT through 2, 5 and 10 revolutions at RT, 100 °C and 180 °C. The hardness of the solution-treated (ST) sample is also included. Data from the solution-treated sample, and 2-, 5- and 10-revolution disks are presented using a diamond, circles, squares, and triangles, respectively. The three processing temperatures – RT, 100 °C and 180 °C are represented using black, red and yellow, respectively.

Mn 0.1, Ti 0.1, Zn 0.15, and Al in balance (wt%). The alloy was cut into disks with a diameter of 20 mm and a thickness of 1 mm and then was solution heat treated in a salt bath at 560 °C for 30 min, followed by quenching in water. The disks were subsequently processed by constrained HPT for 2-, 5-, and 10-revolution at RT, 100 °C and 180 °C under a hydraulic press of 6 GPa and at a ram rotation speed of 1 rpm. The HPT disks were mechanically grinded using 1200 grade grinding papers to a thickness of ~700 μm to produce a smooth flat surface for hardness testing. Hardness tests were performed using a Leco LV700AT Vickers hardness tester with an applied load of 5 kg and a dwell time of 10 s, at radial positions of each disk r = 4, 5, 6, 7, 8 and 9 mm. Five datum points were tested for each radial value r and measured hardness for each r was averaged to obtain mean hardness values. The error bar for each mean hardness value was obtained from the tested highest hardness value to the lowest value. A newly developed transmission electron backscatter diffraction (t-EBSD) technique was employed to resolve the ultra-fine grains of HPT samples. In the t-EBSD technique, a Kikuchi diffraction pattern is projected from the lower surface of an electron transparent sample in the scanning electron microscope (SEM) [20]. The t-EBSD analysis was conducted using a high resolution field emission gun SEM (Zeiss Ultra Plus), using a spacing of 10 nm between individual analysis points. Samples for TEM were prepared using standard electro-polishing techniques from thin disks (3 mm in diameter) punched from HPT disks. TEM characterisation was conducted using a JEOL 1400 TEM operating at 120 kV and energy dispersive X-ray spectrometry (EDS) measurements were carried out using a JEOL 2200FS TEM operating at 200 kV, using a nominal probe size of 1 nm. Line-scan analyses were performed across grain boundaries properly titled to parallel the transmission electron beam.

3. Results

The hardness plots of the solution-treated and HPT 6060 Al alloy processed at different temperatures as a function of the von Mises equivalent strain [21] are shown in Fig. 1. The von Mises equivalent strain γ is calculated using the equation $\gamma = 2\pi Nr/\sqrt{3}h$, where N is the number of HPT revolutions, r is the radial distance from the disk centre and h is the thickness of the disk [6,21]. The average hardness of the solution treated (ST) sample is ~48 HV. The hardness of the alloy increased dramatically during the initial stages of the HPT processing at all temperatures. During RT HPT processing, the hardness increased almost linearly with the shear strain for the strain values larger than 29 and reached 154 HV at the edge

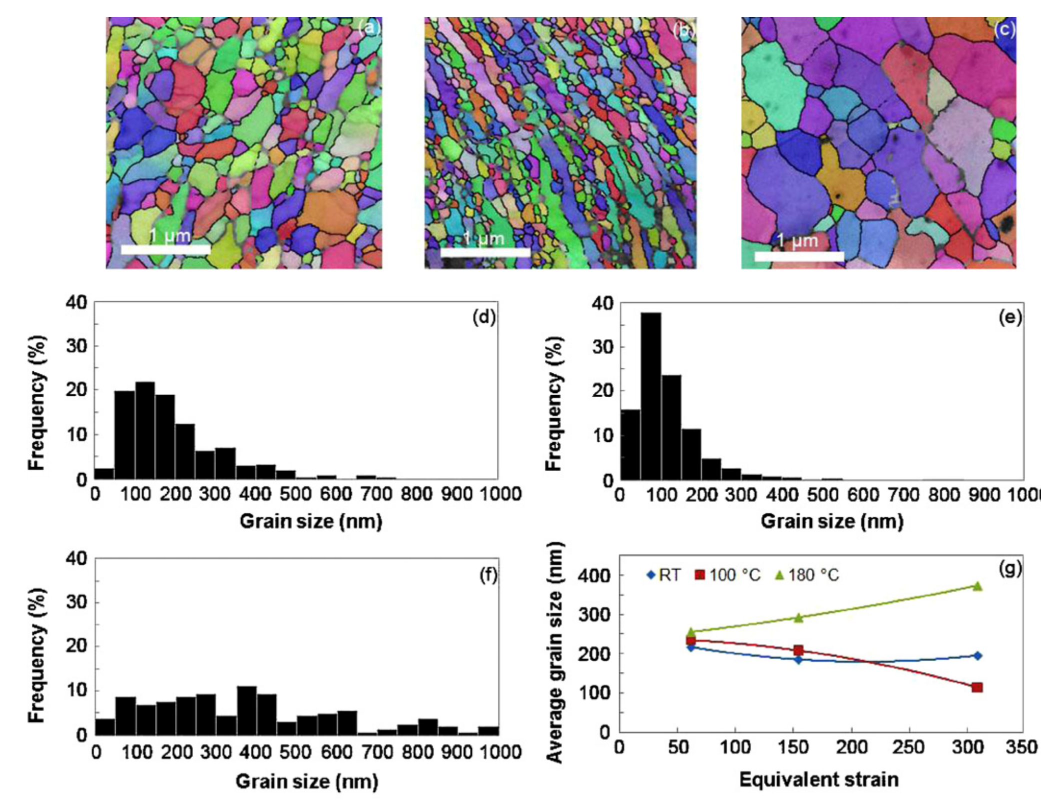

Fig. 2. T-EBSD orientation maps (a–c) and corresponding grain size distributions (d–f) at the edge of 10-revolution HPT disks processed at RT, 100 °C, and 180 °C, respectively. (g) Average grain sizes plotted against the von Mises equivalent strain for HPT disks processed at the three temperatures. The data were obtained from the edge of 2-, 5-, and 10-revolution HPT disks.