Journal of Alloys and Compounds 473 (2009) 163–166

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

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

An evidence of high strain rate superplasticity at intermediate homologous temperatures in an Al–Zn–Mg–Cu alloy processed by high-pressure torsion

J.M. García-Infanta [a,*], A.P. Zhilyaev [a,b], A. Sharafutdinov [c], O.A. Ruano [a], F. Carreño [a]

[a] Department of Physical Metallurgy, Centro Nacional de Investigaciones Metalúrgicas (CENIM), CSIC, 28040 Madrid, Spain
[b] Institute for Metals Superplasticity Problems, RAS, 450001 Ufa, Russia
[c] Scientific Bureau "Iskra", 450000 Ufa, Russia

ARTICLE INFO

Article history:
Received 8 May 2008
Received in revised form 3 June 2008
Accepted 4 June 2008
Available online 17 July 2008

Keywords:
Al–Zn–Mg–Cu alloy
High-pressure torsion (HPT)
Small punch test
Superplasticity

ABSTRACT

A commercial Al–Zn–Mg-Cu alloy, 7075 Al, was processed by high-pressure torsion in unconstrained conditions at room temperature to five complete turns under load of 6 GPa. Microstructural characterization was performed by transmission electron microscopy in the peripheral region of the processed samples, showing equiaxed and submicron grain sizes of 100–150 nm. Vickers microhardness measurements revealed large strengthening at ambient temperature as a consequence of the grain refinement. Small punch testing, having the same axial symmetry as high-pressure torsion processing, was performed to evaluate the mechanical properties at intermediate temperatures. The processed disks showed high strain rate superplasticity at 250 and 300 °C.

1. Introduction

Using classical thermomechanical processing (TMP) for microstructural refinement of 7075 Al alloy [1], the requested high temperatures and low strain rates for subsequent superplastic forming give too high costs to be effective for final industrial processing. Severe plastic deformation (SPD) processes like equal-channel angular pressing (ECAP) [2,3], friction stir processing (FSP) [4], plane strain machining [5] and high-pressure torsion (HPT) [6,7] have been proposed as good alternatives for development of fine and highly disoriented bulk microstructures. Such refinement produces an improvement of the superplastic behavior of the processed materials [8–10]. The main problem of the ultrafine grained (UFG) microstructures when deformed at high temperatures is its thermal instability. Grain growth takes place readily causing the loss of superplastic properties, due to the strong dependence on grain size of superplastic deformation.

A careful inspection of the literature shows that the strategy followed in the majority of SPD processed Al alloys is to stabilize the microstructure by adding small amounts of elements such as Sc and Zr [9,11–13]. A fine dispersion of Al_3Sc and/or Al_3Zr formed during casting acts inhibiting grain growth at high temperatures. As a consequence, high strain rate superplasticity (HSRS) has been observed at high temperatures. Since Sc and Zr are expensive, such additions to the composition of widely employed alloys such as the Al 7075, would raise the production costs. Another strategy, explored in few publications [14,15], is to optimize SPD processes and routes to develop homogeneous, UFG and highly disoriented microstructures which showed superplasticity at low homologous temperatures at which grain growth is still slow enough. The later is a promising option since, not only avoid expensive composition changes, but also would considerably reduce costs in terms of temperature in superplastic forming processes.

The aim of this paper is to develop an UFG microstructure in a commercial Al 7075 alloy showing superplasticity at lower temperatures than those necessary for the conventional alloy.

2. Experimental procedure

A commercial rolled plate of a 7075 Al alloy with the following composition (wt.%): 5.65 Zn, 2.51 Mg, 1.59 Cu, 0.19 Fe, 0.19 Cr and balanced Al, in T6 peak aged condition was subjected to overaging at 280 °C during 5 h. Rods of 10 mm diameter were machined out from the plate, being the rod axis parallel to the rolling direction of the plate. Cylinders of about 1 mm in initial height were sliced from the overaged rods and processed by HPT in unconstrained conditions [16]. A pressure of 6 GPa was applied to the samples and then were subjected to five complete revolutions to a final height of about 0.7 and 10 mm in diameter.

Conventional optical microscopy techniques were employed to characterize the overaged microstructure. TEM of the processed microstructures was performed in a JEOL2010 microscope operating at 200 kV. Thin foils for TEM observation of the processed samples were cut from the periphery of the HPT disk, mechanically thinned on both faces up to 100 μm and electropolished using a solution (vol.%) of 20% HNO_3 and 80% CH_3OH at −28 °C and 20 V.

* Corresponding author. Tel.: +34 91 553 89 00; fax: +34 91 534 74 25.
E-mail address: jmgi@cenim.csic.es (J.M. García-Infanta).

0925-8388/$ – see front matter © 2008 Elsevier B.V. All rights reserved.
doi:10.1016/j.jallcom.2008.06.006

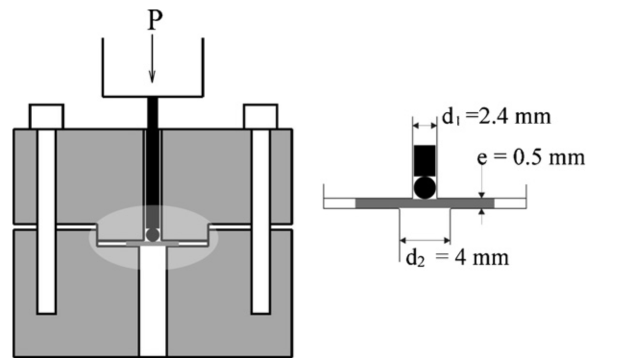

Fig. 1. Schematic of the SPT device with corresponding dimensions of the punch and sample.

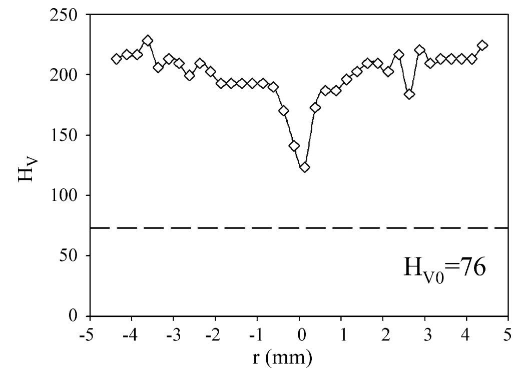

Fig. 2. Microhardness profile across the diameter of the HPT disks in comparison with the hardness of the initial overaged samples.

For hardness measurements of the as processed microstructure, one HPT disk was carefully grinded to its middle plane, mechanically polished to a mirror like finish concluding with 0.05 μm colloidal silica suspension. Microhardness was measured throughout the diameter of the HPT disk using a Vickers microhardness tester with load of 100 g.

The superplastic behavior of the HPT disks was evaluated using small punch testing (SPT) at intermediate homologous temperatures. SPT is a mechanical testing technique used in the power generation and nuclear industry [17], usually applied in fracture mechanics studies of pieces in service [18,19] and also in creep testing [20–22]. In this work, SPT was selected for the evaluation of mechanical behavior at high temperatures because it has the same axial symmetry as HPT processing and the small amount of material required for samples. Fig. 1 shows a schematic with the dimensions of the SPT design. HPT disks were carefully grinded in both faces to a thickness of h_0 = 0.5 mm and polished to 1 μm with diamond paste. The disks were centered with the load axis and clamped between two dies. The load is applied by a cylindrical punch on top of a hard steel ball. The SPT system was attached to a conventional screw driven testing machine and the tests were performed at constant crosshead speeds of $v_1 = 0.04\,\mathrm{mm\,s^{-1}}$ and $v_2 = 0.004\,\mathrm{mm\,s^{-1}}$. Prior to testing, the samples were heated to 250 and 300 °C, and kept at these temperatures during 30 min. Details on the SPT have been also given elsewhere [23].

3. Results and discussion

Fig. 2 shows the grain structure of the overaged (Fig. 2a) and the HPT (Fig. 2b) microstructures. Large elongated grains of about 30 μm in thickness are observed in the overaged alloy in the LT plane of the rolled plate (Fig. 2a). Fig. 2b shows an UFG microstructure about 100–150 nm in grain size developed in the periphery of the disk during HPT as a consequence of the severe deformation. This is the finest grain structure achieved in a commercial Al 7075 by HPT or other SPD methods at room temperature. These grain sizes are much lower than ~0.8 μm reported for pure Al processed by HPT [24], because of the presence of Cr rich, and Mg_2Zn precipitates in the overaged microstructure. Large strengthening is achieved as can be seen in Fig. 3 by comparison between hardness measurements of the overaged sample (HV = 76) and those carried out at the periphery of the HPT disk (HV = 220). Such strengthening is consequence of an increase of dislocation density and the Hall-Petch effect. These average grain sizes and hardness values are similar to the results reported for an Al–Zn–Mg–Cu Zr containing alloy in quenched condition also processed by HPT [25]. The presence of a softer central region in the HPT disks has been already reported for other Al alloys even after seven complete turns at 6 GPa [26]. Since the strain accumulated by the microstructure varies along with the radius of the disk, in such a way that the central region accumulates less strain than the periphery, a minimum number of HPT revolutions appear to be necessary to achieve hardness saturation across the whole diameter of the HPT disk. For instance, saturation has been shown in a commercially pure Al after four complete turns [24], but for alloys, like the 7075 Al alloy studied in this work, larger amount of accumulated strain during HPT appear to be necessary to reach saturation, what may be related to slower rates of dynamic recovery than in pure Al because of the presence of particles [27].

The results obtained by SPT are presented in form of punch load (P) vs. punch displacement (d) curves at 250 °C (Fig. 4a) and 300 °C (Fig. 4b) for either the overaged or HPT samples. It is noteworthy to point out that since the punch diameter (2.4 mm) is larger than the softer central region of the HPT samples (1.4 mm), the microstructure shown in Fig. 2b is representative of the initial microstructure of SPT deformed samples. In general, the processed disks show higher displacements and lower punch loads than the overaged samples under same testing conditions, suggesting a change of deformation mechanism in the HPT processed disks.

In diffusion controlled creep mechanisms of polycrystalline materials, the strain rate ($\dot{\varepsilon}$) is related to the stress (σ) and the absolute temperature (T) by the general creep expression $\dot{\varepsilon} = A(\sigma/E)^n \exp(-Q/RT)$, where A is a material constant, E the Young modulus and R the gas constant. n and Q are the stress exponent and

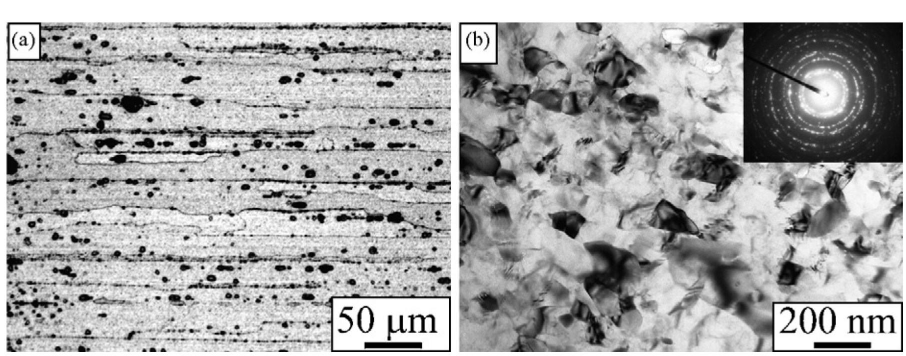

Fig. 3. Microstructure of the 7075 samples (a) elongated grains about 30 microns in thickness in the initial overaged condition; (b) ultrafine grains about 100 nm in size after HPT processing observed in the periphery of the disks.