Materials Science & Engineering A 596 (2014) 52–58

Contents lists available at ScienceDirect

Materials Science & Engineering A

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

A critical evaluation of the processing of an aluminum 7075 alloy using a combination of ECAP and HPT

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ARTICLE INFO

Article history:
Received 30 October 2013
Received in revised form
9 December 2013
Accepted 10 December 2013
Available online 18 December 2013

Keywords:
Equal-channel angular pressing
High-pressure torsion
Microhardness
Superplasticity
Ultrafine grains

ABSTRACT

Experiments were conducted on a commercial Al-7075 alloy by processing by ECAP for 4 passes and then processing by HPT for up to a maximum of 20 turns. Measurements show that the grains were refined to ~680 nm after ECAP and to ~310 nm in the center of the disk after ECAP+HPT. Tensile testing at a temperature of 623 K revealed lower flow stresses and higher elongations to failure after processing by ECAP+HPT. Thus, the alloy was not superplastic after processing by ECAP but superplasticity was achieved with elongations up to ~800% after processing by ECAP+HPT. By plotting the Vickers microhardness against equivalent strain, it is shown that the hardness saturates at Hv ≈ 250 after ECAP+HPT. This saturation hardness is higher than the value of Hv ≈ 230 recorded after processing by HPT without a preceding step of ECAP. The results demonstrate that processing by ECAP+HPT produces higher hardness and greater grain refinement than processing only by HPT.

1. Introduction

Ultrafine-grained (UFG) materials are defined as bulk solids having average grain sizes within the submicrometer or nanometer ranges [1]. These types of materials can be produced by applying severe plastic deformation (SPD) to metals with coarse grain sizes using special techniques in which the processing imposes a high strain without any significant reduction in the overall dimensions of the samples [2]. The advantages of UFG materials include both high strength and the potential for achieving a superplastic forming capability at elevated temperatures. Several SPD processing techniques are now available but most attention has been concentrated to date on the two processes of equal-channel angular pressing (ECAP) [3] and high-pressure torsion (HPT) [4]. In ECAP the material, in the form of a rod or bar, is pressed repetitively through a channel bent through an abrupt angle and in HPT the sample, in the form of a thin disk, is subjected to a high pressure and concurrent torsional straining. Several experimental results are now available to demonstrate that, when ECAP and HPT are conducted using the same initial material, processing by HPT is more effective than ECAP in producing materials with exceptionally small grain sizes [5–7].

When materials are processed using SPD procedures, there is now good evidence that the grain size decreases with increasing strain until it ultimately reaches a stable minimum or saturation condition. The overall limitation in grain refinement has attracted much attention [8–10] but the physical process controlling this saturation grain size is not yet understood. Nevertheless, the minimum grain size appears to be an inherent feature of each material and it is reasonably reproducible between different laboratories. Recently, a dislocation model was used to predict the minimum grain size obtainable by ball milling [11] and this model was subsequently further developed to give additional models, based on available physical parameters, to predict the minimum grain sizes that may be attained in HPT [12] and ECAP [13].

Since HPT processing produces a significantly smaller grain size than ECAP, it is instructive to examine the microstructures that may be produced by combining an initial processing by ECAP with a subsequent processing of the same material by HPT. This type of experiment was first conducted by Stolyarov et al. [14] using commercial purity Ti and they reported that processing by HPT after ECAP produces additional refinement in the grain size and a consequent increase in the hardness. Other experiments on Ni [6,15] and a Cu–0.1% Zr alloy [16] also confirmed the occurrence of a decrease in grain size after a combination of ECAP and HPT. Nevertheless, a recent report by Popov et al. [17] provided additional insight into these experiments. Using commercial purity Nb, it was reported that the grain size after a combination

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http://dx.doi.org/10.1016/j.msea.2013.12.034

of ECAP and HPT was the same as when processing only by HPT and specifically it was claimed that there was a saturation condition which provided neither additional refining of the grains nor additional hardening beyond that which was achieved simply by the HPT processing of a coarse-grained material. Furthermore, it was noted that very similar microstructures were also achieved when processing single crystals of Nb by HPT [18,19]. These results are important because they suggest that the saturation condition may be an inherent feature of each material and it is unaffected by the initial state of the material.

The present research was initiated to examine this conclusion in more detail. Generally, it is not easy to obtain definitive results in HPT processing using microstructural observations of the grain sizes because it is well established that the grain sizes tend to vary with the precise positions on the HPT disks. Accordingly, emphasis was placed instead on taking measurements of the microhardness since this has the capability of providing a unique confirmation of the occurrence of a true saturation condition and thus it gives a direct comparison of different microstructural conditions. This approach is based on the early demonstration in HPT processing that the hardness measurements taken across HPT disks after different numbers of turns may be readily correlated by plotting the measured values of the Vickers microhardness, Hv, against the calculated equivalent strain at each point of measurement [20]. This same approach has been used to correlate hardness data recorded on a large number of different metals [21,22] and it is an effective tool for confirming the occurrence of a saturated condition.

The experiments were conducted using a commercial Al-7075 alloy where this material was selected for three reasons. First, results are now available documenting the processing of this alloy by HPT [23]. Second, earlier results on similar aluminum-based alloys demonstrated the potential for achieving saturated conditions in HPT processing through measurements of the Vickers microhardness [24,25]. Third, the presence of precipitated η' and η phases in the Al-7075 alloy suggests there will be little or no grain growth when conducting mechanical testing at elevated temperatures [26].

2. Experimental material and procedures

The experiments were conducted using a commercial aluminum-based Al-7075 alloy containing (in wt%) 5.6% Zn, 2.5% Mg and 1.6% Cu. The alloy was supplied in the form of extruded rods having a diameter of 10.0 mm and these rods were initially annealed in air at 753 K for 1 h and then cooled to room temperature. In the annealed condition, the grains were highly elongated with lengths up to ~450 μm and widths of ~8 μm. The annealed rods were processed by ECAP at 473 K using a hydraulic press with a capacity of 150 t and a solid die having an internal channel angle of $\Phi = 110^\circ$ and an outer angle at the intersection of the two parts of the channel of $\Psi = 20^\circ$. Using this configuration the shear strain, ε_N, in ECAP is given by a relationship of the form [27]

$$\varepsilon_N = \frac{N'}{\sqrt{3}}\left[2\cot\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right)+\Psi\,\mathrm{cosec}\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right)\right] \quad (1)$$

where N' is the number of passes through the die.

Samples were pressed through four passes using a pressing speed of ~0.5 mm s^{-1} and processing route B_C in which the billet is rotated by 90° around the longitudinal axis in the same direction between each pass [28]. This processing route was selected because earlier results on pure aluminum showed that it leads most expeditiously to an array of equiaxed ultrafine grains separated by grain boundaries having high angles of misorientation [29].

After processing, the ECAP billets were cut perpendicular to their longitudinal axes to provide a series of disks with thicknesses of ~1.5 mm and these disks were polished with abrasive papers to final thicknesses of ~0.83 mm. The polished disks were processed by HPT at room temperature under an applied pressure of 6.0 GPa through total numbers of 1/8, 1/4, 1/2, 3/4, 1, 2, 5, 10 and 20 turns. The HPT facility comprised two heavy anvils machined from high strength tool steel with spherical depressions in the centers on the inner surfaces of the two anvils. These depressions were 0.25 mm in depth with diameters of 10 mm. For HPT processing, a disk was placed in the depression on the lower anvil and this anvil was then raised so that the disk was also contained within the depression on the upper anvil and it was possible to exert a pressure on the disk. The present investigation was conducted under quasi-constrained conditions in which there is some minor outflow of material around the periphery of the disk during processing [30,31] and the torsional straining was achieved by rotation of the lower anvil at a constant speed of 1 rpm. Full details of the procedure for HPT processing were given earlier except that in this investigation a lubricant was not placed around the depressions in the lower and upper anvils [32]. The equivalent von Mises strain, ε_{eq}, exerted in HPT is given by a relationship of the form [33–35]:

$$\varepsilon_{eq} = \frac{2\pi N r}{h\sqrt{3}} \quad (2)$$

where N is the number of turns of torsional straining, r is the radial distance measured from the center of the disk and h is the initial height (or thickness) of the sample. It is readily apparent from Eq. (2) that the strain varies across the disk with a maximum strain at the outer edge and zero strain at the center of the disk. Eq. (2) suggests that the microstructure produced in HPT processing will be very inhomogeneous but in practice it has been shown, both experimentally [36–39] and theoretically [40], that there is a gradual evolution with increasing numbers of turns into a reasonably homogeneous microstructure.

Disks of the Al-7075 alloy were processed by HPT for up to 20 turns and then mounted and polished with abrasive papers until a mirror-like surface was achieved. Microhardness measurements were taken on the polished surfaces using an FM-1e microhardness instrument equipped with a Vickers indenter using a load of 100 gf with a dwell time of 10 s for each individual indentation. The values of the Vickers microhardness were recorded from indentations taken along randomly selected diameters on the surfaces of each disk. The microhardness value appropriate to each indentation was obtained from the average of four separate hardness measurements recorded at uniformly separated points displaced around the selected position. The distance between each indentation was 0.3 mm and this was effectively reduced to 0.15 mm due to the averaging procedure.

The microstructural characteristics of the alloy were investigated in the annealed condition after processing by ECAP for 4 passes and after a combination of ECAP for 4 passes together with HPT through up to 20 turns. These observations were recorded using electron back-scatter diffraction (EBSD) and orientation imaging microscopy (OIM). Samples were ground with 400 grit SiC paper, continued through 2500 grit SiC paper, polished with 9, 6, 3 and 1 μm diamond suspensions and then with a 0.04 μm colloidal silica suspension using a vibratory polishing machine. The microstructural data were obtained using an analytical field emission scanning electron microscope JEOL JSM-7001 F at an operating voltage of 15 kV. The EBSD patterns were collected at a working distance of 15 mm with a sample tilt of 70°. The scanning electron microscope was equipped with imaging detectors and used a TSL orientation imaging system and OIM™