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Principles of grain refinement and superplastic flow in magnesium alloys processed by ECAP

Roberto B. Figueiredo [a], Terence G. Langdon [a,b,*]

[a] Departments of Aerospace & Mechanical Engineering and Materials Science, University of Southern California, Los Angeles, CA 90089-1453, USA
[b] Materials Research Group, School of Engineering Sciences, University of Southampton, Southampton SO17 1BJ, UK

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ABSTRACT

The superplastic properties of metallic materials are associated with the length scale and with the thermal stability of their grain structure. Whereas equal-channel angular pressing (ECAP) may be used to produce ultrafine-grained structures in f.c.c. metals through the homogeneous subdivision of the grains, research on two magnesium alloys reveals a different and heterogeneous process of grain refinement which is dependent upon the initial grain structure in the alloys. Experiments demonstrate that different structural features may be achieved using different processing routes and this leads to the development of a processing strategy for achieving an optimum microstructure. It is shown by mechanical testing that the optimum superplastic properties also depend on the processing route and, depending on the structural characteristics, the maximum elongations to failure may occur either in the early stages of processing by ECAP or after processing through large strains.

1. Introduction

It is now established that processing by Severe Plastic Deformation (SPD) can refine the grain structure of metallic materials to the ultrafine (<1 μm) or even the nanometer (<100 nm) scale [1]. Processing by equal-channel angular pressing (ECAP) appears to be the most effective SPD technique because it uses simple facilities and it is easily scaled-up to produce materials having reasonably large volumes [2]. A direct consequence of the grain refinement introduced by ECAP is the development of superplastic capabilities when testing in tension at elevated temperatures. Thus, it is well established in superplasticity that if the microstructure is refined to grain sizes of less than ∼10 μm, and provided this structure is reasonably stable at high temperatures, the material has a potential for exhibiting superplastic flow [3]. To date, superplasticity has been reported in several metals processed by ECAP and a detailed tabulation of these results was presented recently [4].

The majority of early reports on superplasticity in materials processed by ECAP focused on aluminum alloys or other f.c.c. materials but more recently there has been a considerable and growing interest in extending the same approach to magnesium alloys. There are now several reports of excellent superplastic ductilities in various magnesium alloys including elongations of ∼1780% in a Mg–8% Li alloy [5], 2040% in a ZK60 alloy (Mg–5.8% Zn–0.6% Zr) [6] and ∼3050% in a ZK60 alloy [7] where the latter elongation is the highest reported to date for any metal processed by ECAP and also the highest recorded for a magnesium alloy processed by any procedure.

Despite these excellent results, it is now apparent that there are two significant differences between magnesium alloys and the many f.c.c. metals processed by ECAP.

First, in f.c.c. metals such as pure aluminum the refinement of the grains follows in a simple and direct way from observations taken on aluminum single crystals processed by ECAP. Specifically, the large initial grains contain bands of elongated cells or subgrains after the first pass through the ECAP die and on subsequent passes these bands evolve into an ultrafine-grained structure in which the final stable grain size is dictated by the initial spacing between the subgrain walls [8]. The grain refinement occurring in aluminum is consistent both with the behavior predicted on a macroscopic scale [9] and with the models that were developed to describe the shearing patterns for different ECAP processing routes [10,11].

By contrast, grain refinement in magnesium alloys appears to be more complex and there are numerous reports delineating the development of a bimodal grain structure after processing by ECAP. For example, the report of an elongation of 2040% was recorded in

* Corresponding author at: Departments of Aerospace & Mechanical Engineering and Materials Science, University of Southern California, Los Angeles, CA 90089-1453, USA. Tel.: +1 213 740 0491; fax: +1 213 740 8071.
E-mail address: langdon@usc.edu (T.G. Langdon).

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a ZK60 alloy with a bimodal grain distribution [6] whereas, by contrast, the elongation of ~3050% was recorded in the same alloy with a homogeneous grain structure [7]. There are also reports for magnesium alloys of a continuous reduction in grain size with increasing numbers of passes in ECAP [12–14] and with the inhomogeneous formation of fine grains [15–17]. Furthermore, although there is a general consistency in the grain sizes reported by different investigators for f.c.c. metals processed by ECAP, significantly different final grain sizes have been reported in the same magnesium alloys after similar ECAP processing: for example, an AZ31 alloy with a grain size of ~2.5 μm processed by 8 passes of ECAP at 473 K produced a final grain size of ~0.7 μm [18] whereas the same alloy with an initial grain size of ~15.5 μm processed by ECAP at the same temperature produced a final grain size of ~1.9 μm [19]. All of these disparate results suggest that the initial pre-processing condition of the alloy probably plays a key role in determining the nature of the final microstructure.

Second, earlier experiments on an aluminum alloy showed that the measured superplastic elongations increase with the numbers of passes through the ECAP die, at least up to 8 passes [20]. Similar trends were reported also for a Mg–0.6% Zr alloy [21] and an AZ61 alloy [22] but more recently the record elongation of ~3050% was achieved using a ZK60 alloy processed through only 2 passes and thereafter the measured elongations decreased with additional numbers of passes [7]. There are no similar reports of decreasing superplastic elongations with increasing numbers of passes for any f.c.c. metals.

The reasons for the differences between f.c.c. metals and magnesium alloys are not understood at the present time and they formed the rationale for the present investigation. Accordingly, tests were conducted using magnesium ZK60 and AZ31 alloys where these alloys were selected because both ZK60 [6,7,23–26] and AZ31 [27,28] exhibit excellent superplastic properties after ECAP. There are conflicting reports for ZK60 of both bimodal [6,15,16,23] and homogeneous [7,24–26] grain distributions after processing by ECAP and there are similar reports of bimodal [27] and homogeneous [28] microstructures in the AZ31 alloy. The present tests were therefore undertaken with two specific objectives. First, to determine the principles of grain refinement during the ECAP processing of magnesium alloys with special emphasis on the factors governing the occurrence of either homogeneous or bimodal grain distributions. Second, to determine the optimum processing conditions in order to achieve the maximum superplastic ductilities during tensile testing at elevated temperatures.

2. Experimental materials and procedures

The experiments were conducted using two commercial magnesium alloys, AZ31 (Mg–3% Al–1% Zn) and ZK60 (Mg–5.5% Zn–0.5% Zr), where the compositions are given in wt.%. Both materials were supplied by Timminco Corporation (Aurora, CO) in the form of extruded rods with diameters of 10 mm. These rods were cut into billets with lengths of 60 mm. The processing by ECAP was conducted at 473 K for both alloys using a Dake hydraulic press of 150-tonnes capacity operating at a pressing speed of ~7 mm s^{-1}. An MoS_2 lubricant was used between the billets and the die walls. The solid dies had angles between the channels, Φ, of either 90° or 110° and angles of external curvature, Ψ, of 20°. These two die configurations impose strains of ~1.1 and ~0.8 on the billets during each pass for the dies having angles of 90° and 110°, respectively [29]. Billets of both materials were pressed through multiple numbers of passes using processing route B_C where the billets are rotated by 90° in the same sense between each pass [10]. Both alloys were processed in the as-received condition but some billets of the ZK60 alloy were also annealed prior to ECAP to eliminate the effect of the prior extrusion. This annealing was conducted at 673 K for 4 h followed by furnace cooling. The as-received ZK60 alloy was successfully pressed for up to 6 passes using the die with $\Phi = 90°$ and up to 8 passes using the die with $\Phi = 110°$. Annealing of the ZK60 alloy reduced its formability and the billets failed by segmentation when pressing using the 90° die. However, these billets were pressed successfully up to 6 passes using the die with $\Phi = 110°$. This observation is consistent with an earlier report which outlined the principle of increasing the die angle in order to process difficult-to-work alloys [30]. A similar effect occurred with the AZ31 alloy which failed in the 90° die but was successfully pressed up to 4 passes in the die with $\Phi = 110°$.

Tensile specimens having gauge lengths of 4 mm and cross-sectional areas of 2 mm × 3 mm were machined from the billets before and after ECAP. Each specimen gauge length was oriented parallel to the axial direction of the billet. Tensile testing was conducted using an Instron machine operating at a constant rate of cross-head displacement and, based on earlier reports, with the testing conditions chosen to optimize the superplastic behavior. Thus, optimum superplasticity occurs in the ZK60 alloy at ~473 K at strain rates in the range of ~10^{-4} s^{-1} [6,7,23–26] and in the AZ31 alloy at similar strain rates but at a higher temperatures of ~623 K [27,28]. Instantaneous values of the strain rate sensitivity, m, were determined by making cyclic changes in the rate of cross-head displacement during the tests. Values of the load and displacement were converted to true stress and true strain by assuming homogeneous deformation throughout the gauge lengths.

Small samples were cut from the billets of both alloys before ECAP and after ECAP to different numbers of passes. These samples were mounted, ground on abrasive paper, polished with 0.05 μm alumina powder and etched in a solution of picric acid, acetic acid, distilled water and ethanol to reveal the grain boundaries. Optical microscopy was selected in order to make microstructural observations over large areas. The average grain sizes were estimated for each condition using the mean linear intercept procedure.

3. Experimental results

3.1. Grain structures before and after ECAP

The evolution of the grain structure during multiple passes of ECAP is presented in Figs. 1–4.

Fig. 1 shows the evolution of the grain structure for the ZK60 alloy processed from the extruded condition. The initial average grain size for the as-received condition was ~2.9 μm as shown in Fig. 1(a). The grain structures shown in Fig. 1(b) and (c) were recorded after 2 and 6 passes, respectively, using the die with $\Phi = 90°$: in both conditions the average grain sizes were measured as ~0.8 μm. Similar results were recorded by processing the extruded ZK60 alloy in the die with $\Phi = 110°$ with the same average grain size of ~0.8 μm after pressing through from 2 to 8 passes. These results show that, as in f.c.c. metals, the grain size produced in the early stages of ECAP remains reasonably constant during subsequent passes. It should be noted that early experiments on high-purity aluminum showed that, although the grain size remains essentially constant with additional processing, increasing numbers of passes lead to an increase in the fraction of grain boundaries having high angles of misorientation [31].

The microstructural evolution occurring in the ZK60 alloy when processed from the annealed condition is shown in Fig. 2 where Fig. 2(a) gives the initial structure where there was a large average grain size of ~180 μm. Fig. 2(b)–(d) shows the annealed billets after pressing through totals of 1, 4 and 6 passes, respectively. After