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Using Equal-Channel Angular Pressing for the Production of Superplastic Aluminum and Magnesium Alloys

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Equal-channel angular pressing (ECAP) is a useful tool for achieving exceptional grain refinement in bulk metallic alloys. Typically, the grain sizes produced through ECAP are in the submicrometer range, and thus they are smaller by up to an order of magnitude than the grain sizes attained through typical thermomechanical treatments. As a consequence of these ultrafine grains, the as-pressed alloys may exhibit superplastic ductilities at faster strain rates than in conventional superplastic alloys. This work initially describes the application of ECAP to two different alloys. First, results are presented for a commercial Al-2024 alloy where this alloy was selected because it contains no minor additions of either zirconium or scandium to assist in restricting grain growth. The results show that superplasticity is achieved through the use of ECAP. Second, results are described for a Mg-0.6%Zr alloy where this alloy was selected because it is the optimum composition for achieving a high damping capacity. Again, processing by ECAP produces superplastic ductilities not attained in the cast alloy. The second part of this work demonstrates that processing by ECAP may be extended from conventional rod or bar samples to samples in the form of plates. This is a very attractive feature for industrial superplastic forming applications.

Keywords aluminum alloys, equal-channel angular pressing (ECAP), magnesium alloys, severe plastic deformation (SPD), ultrafine grain sizes

1. Introduction

The superplastic forming (SPF) industry is now well established for the production of complex and/or highly curved parts from sheet metals for use in aerospace applications (Ref 1-7). However, the SPF procedures have attracted only limited attention to date for possible use in the fabrication of automotive or other high-volume components.

This apparent dichotomy was first addressed by Barnes (Ref 3) when he noted that, as a direct consequence of cost considerations, the SPF technology occupies a relatively small techno-economic niche because it cannot compete effectively in the fast production of large volumes of superplastically formed components. This difficulty may be readily understood when it is noted that the regime of superplastic deformation occurs in metals over a relatively small range of strain rates (generally termed region II), and the superplastic ductilities are lost both at rapid strain rates when intragranular dislocation processes become rate-controlling (termed region III) and at slow strain rates when the behavior is controlled by impurity effects (termed region I) (Ref 8-10). For conventional superplastic alloys, such as the 2004 and 5083 aluminum alloys, the production forming rates are within the range of ~10^{-3} to 10^{-2} s^{-1} (Ref 3) so that each separate component requires a forming time of the order of 20 to 30 min. These very long forming times are acceptable for the production of low-volume high-value components in the aerospace industry or in the fabrication of curved panels for exotic high-performance sports cars, but they are not commercially viable for the mass-production of high-volume low-cost components in the regular consumer-product industries.

To expand the techno-economic niche for SPF, an important step may be achieved if fast-forming operations can be developed utilizing the occurrence of high strain rate superplasticity. Barnes (Ref 3) recognized this potential solution in his earlier analysis where he refers to high strain rate superplasticity as "a new paradigm." In practice, high strain rate superplasticity is well documented in the scientific literature, and it refers to superplastic ductilities that are achieved at strain rates at and above 10^{-2} s^{-1} (Ref 11). However, a detailed review shows that the materials exhibiting these very rapid rates are generally limited to a rather narrow range of metal-matrix composites, intermetallics, and ceramics (Ref 11). Thus, to evaluate the potential for increasing the rate of SPF in conventional alloys, it is first necessary to examine the factors influencing the strain rate in the superplastic regime.

Superplastic ductilities are achieved in metals when the grains are able to move over each other in the process of grain boundary sliding (Ref 12). Two conditions are needed to

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achieve superplastic flow: a high operating temperature, at or above ~0.5 T_m, where T_m is the absolute melting temperature, and a small and stable microstructure having a grain size <10 μm (Ref 8). It has been shown, both experimentally and theoretically, that the steady-state strain rate ($\dot{\varepsilon}$) in the superplastic region II varies inversely with the grain size of the material (d) raised to a power of two (Ref 13, 14). Generally, superplastic materials are obtained using appropriate thermomechanical processing and the grain sizes produced in this way, and the grain sizes used in industrial SPF lie typically within the range of ~2 to 10 μm. Since $\dot{\varepsilon}$ varies with $1/d^2$ in region II, it follows that the production of bulk solids in which the grain sizes are reduced by one order of magnitude, to the range of ~200 nm to 1 μm, leads to an increase in the effective strain rate by two orders of magnitude. This means that the individual forming times may be reduced to 20 to 30 s, and it then becomes viable to expand the use of SPF technology to a much wider range of applications. It is necessary, therefore, to examine the possible procedures for developing bulk solids with ultrafine submicrometer grain sizes.

2. Achieving Polycrystalline Metals with Ultrafine Grain Sizes

Two separate procedures have been developed to produce materials having extremely small grain sizes. The first type, often known as the "bottom-up approach" (Ref 15), refers to the assembly of bulk solids from individual atoms as in inert gas condensation (Ref 16) or from nanoparticles as in cryomilling, where mechanical milling is performed at a low temperature such as 77 K followed by powder compaction (Ref 17). These types of procedures are especially effective in producing bulk materials with nanocrystalline grain sizes, but they have two critical deficiencies that severely impede their utility outside of specialized industries such as microelectronics. First, the fabricated materials are not fully dense so that some limited porosity is always present. Second, these procedures are suitable only for the production of very small samples that cannot be used in the SPF industry.

The second type, known as the "top-down" approach (Ref 15), involves the processing of bulk solids through the application of severe plastic deformation (SPD) (Ref 18). In processing by SPD, conventional materials with large grain sizes are severely deformed through the introduction of large numbers of dislocations, and these dislocations subsequently rearrange to form arrays of ultrafine grains. Typically, the grain sizes produced using these procedures lie in the submicrometer range (~100 nm to 1 μm), but some techniques are capable of producing bulk solids with grain sizes within the nanometer range (<100 nm). Furthermore, these procedures have the advantage that they lead to fully dense samples and they are capable of producing relatively large bulk solids.

Several different SPD processes are now available, but the most versatile, and the most appropriate for use in industrial applications, appears to be equal-channel angular pressing (ECAP), in which a material is pressed through a die and a high strain is imposed without incurring any change in the cross-sectional dimensions of the workpiece. The first development of ECAP may be traced to the early work of Segal et al. (Ref 19) in the former Soviet Union, and subsequently it was demonstrated that processing by ECAP is effective in producing exceptional grain refinement in bulk metals (Ref 20, 21). The principles of ECAP were described in a recent review (Ref 22), and the process is illustrated schematically in Fig. 1.

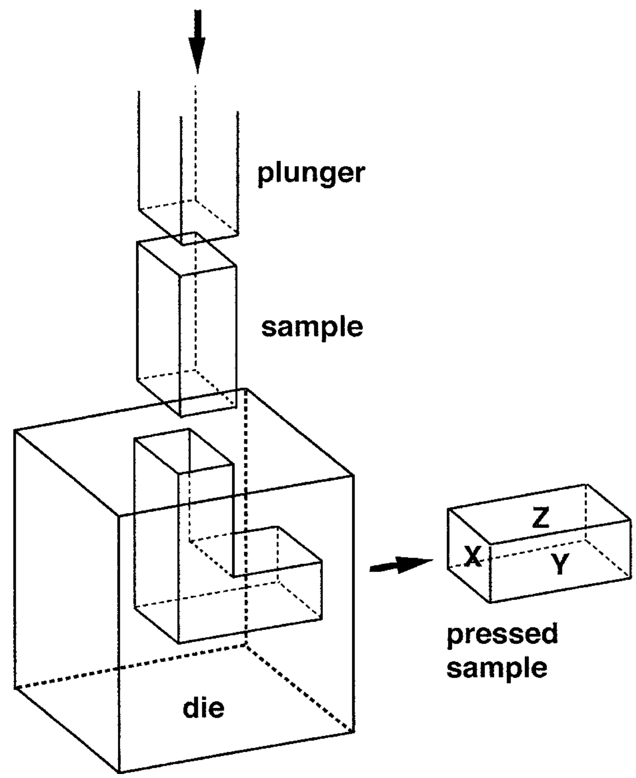

Fig. 1 Schematic illustration of the principle of ECAP. Source: Ref 22

In ECAP, a die is constructed having an internal channel that is bent through an abrupt angle usually at or very close to 90°. The sample is machined to fit within the channel, and it is pressed through the die using a plunger. Since the cross-sectional dimensions remain unchanged in a single passage through the die, it is apparent that multiple passes may be conducted on the same sample to impose a very high strain. In practice, it can be shown from first principles that the imposed strain depends on both the angle (Φ) between the two parts of the channel ($\Phi = 90°$ in Fig. 1) and the angle (Ψ) subtended by any curvature at the outer arc where the two channels intersect ($\Psi = 0°$ in Fig. 1) (Ref 23). For regular dies where $\Phi = 90°$, the imposed strain is close to ~1 with only a minor dependence on the value of Ψ. When repetitive pressings are conducted on the same sample, different slip systems may be introduced by rotating the sample about the longitudinal axis between each consecutive pass. Four possible processing routes have been analyzed in detail: route A, where there is no rotation; route B_A, where the sample is rotated by 90° in alternate directions between passes; route B_C, where the sample is rotated by 90° in the same sense between each pass; and route C, where the sample is rotated by 180° between passes (Ref 24, 25). Experiments have shown that route B_C is the optimum processing route for achieving a distribution of reasonably equiaxed grains separated by boundaries having high angles of misorientation (Ref 26), and it is also the optimum processing route for achieving the highest superplastic ductilities (Ref 27). Three orthogonal planes, X, Y, and Z, are also defined in Fig. 1 with reference to the pressed sample.

A series of detailed experiments on a cast Al-3%Mg-0.2%Sc alloy demonstrated the potential for using ECAP processing to achieve both superplastic ductilities in tensile specimens within the strain rates associated with high strain rate superplasticity (Ref 28, 29) and a superplastic forming capability when small disks are cut from the as-pressed billets and inserted into a biaxial gas-pressure forming facility (Ref 30). As is shown in the subsequent sections, processing by ECAP can be used effectively for achieving superplastic ductilities in both aluminum and magnesium alloys.

3. Developing Superplastic Properties in Bulk Alloys

3.1 Aluminum 2024

Tests were conducted on a commercial aluminum 2024 alloy containing, in weight percent: 4.4% Cu, 1.5% Mg, and 0.6% Mn. This alloy was selected because it contains no minor additions of either scandium or zirconium to restrict grain growth. However, inspection revealed the presence of a dispersion of fine (~50-100 nm) $CuMgAl_2$ particles that were distributed reasonably homogeneously throughout the material. In the as-received condition, the alloy contained platelike grains having dimensions of ~500 by 500 by 10 μm^3.

The experiments on the 2024 alloy are described in detail elsewhere (Ref 31). Briefly, the alloy was pressed at room temperature (298 K) and at 373 K using processing route B_C to eight passes with a die having an internal angle of $\Phi = 90°$. Following ECAP, the grains were essentially equiaxed with sizes of ~0.3 and ~0.5 μm after pressing at 298 and 373 K, respectively. Since grain stability is required to achieve superplastic elongations at elevated temperatures, samples of the as-pressed alloy were annealed for 1 h at various elevated temperatures. Figure 2 shows the variation of the measured grain size with the annealing temperature; also shown in Fig. 2 are representative results for pure aluminum, an Al-3%Mg alloy, and an Al-3%Mg-0.2%Sc alloy. Inspection of Fig. 2 shows that the small grain sizes introduced by ECAP into pure aluminum or the aluminum-magnesium alloy are not stable at temperatures above ~500 K due to the absence of any precipitates, whereas in the 2024 alloy and the Al-Mg-Sc alloy, the presence of precipitates retains the fine grain sizes up to temperatures of at least 700 K. These results suggest, therefore, the possibility of achieving superplasticity in tensile testing of the 2024 alloy at high temperatures.

This possibility is confirmed in Fig. 3 in which the elongation to failure is plotted against the initial strain rate for specimens tested in tension at 673 K, where the lower line shows data for the unpressed alloy and the two upper lines show the results obtained for samples taken through eight passes at 298 or 373 K, respectively. Thus, the unpressed material yields maximum elongations that are consistently <200%, but elongations of up to >400% are achieved after processing by ECAP. Furthermore, the highest elongation of ~500% is achieved using an initial strain rate of 1.0×10^{-2} s^{-1} after processing by ECAP at 373 K, where this strain rate denotes the occurrence of high strain rate superplasticity. Figure 3 reveals also a displacement in the peak elongation to a faster strain rate when increasing the temperature for the ECAP processing, where this displacement occurs despite the slightly larger grain size in this material. Since it is well established that the fraction of high-angle boundaries increases in ECAP with increasing numbers of passes through the die (Ref 32), it is reasonable to anticipate that the fraction of high-angle boundaries will also increase more rapidly at higher processing temperature due to the more rapid evolution of the highly deformed structure into an equilibrated microstructure.

It is especially important to note that the present results

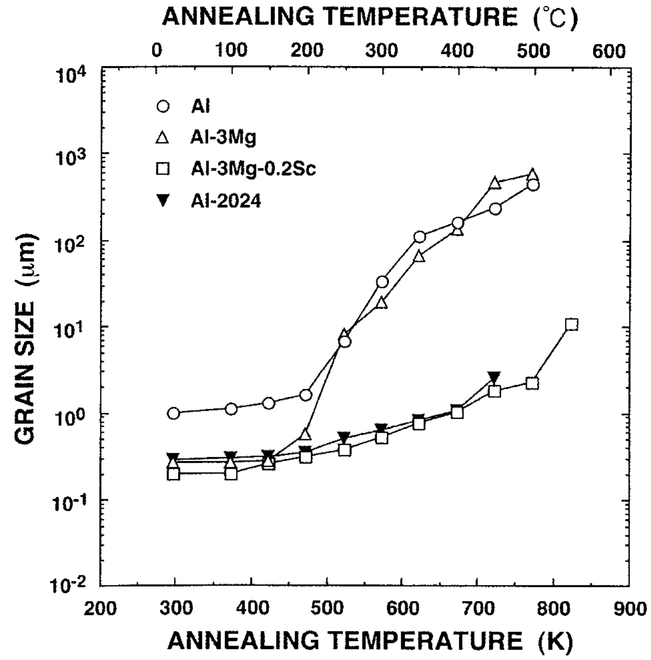

Fig. 2 Grain size versus annealing temperature for the 2024 alloy after ECAP; data are also shown for pure Al, an Al-3%Mg alloy, and an Al-3%Mg-0.2%Sc alloy. Source: Ref 31

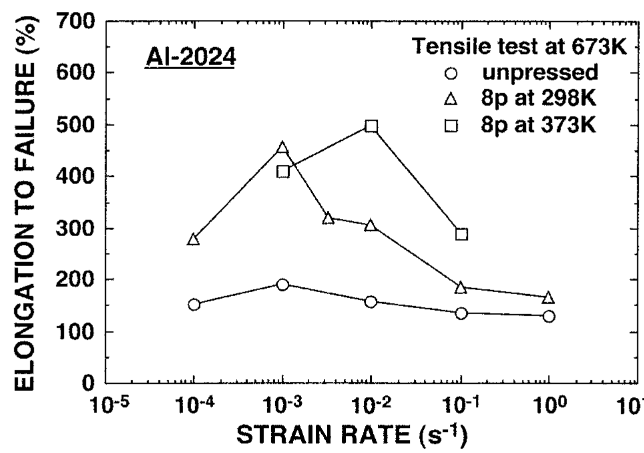

Fig. 3 Elongation to failure versus strain rate for samples of the 2024 alloy tested at 673 K in the unpressed and pressed conditions. Source: Ref 31

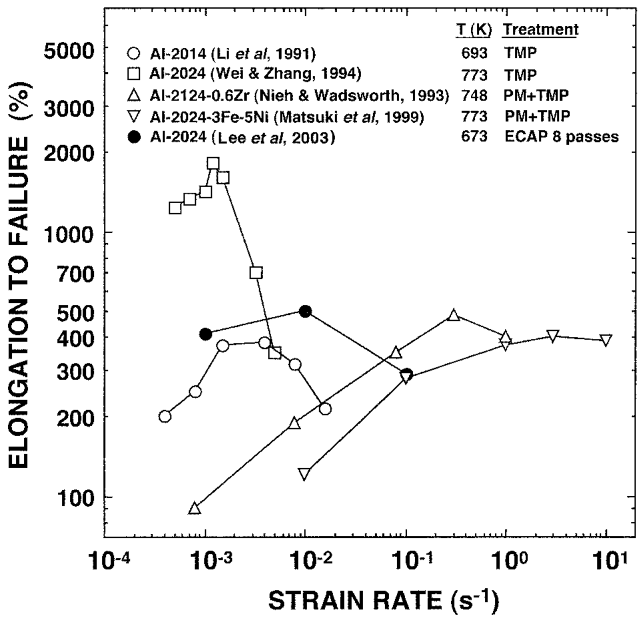

Fig. 4 Elongation to failure versus strain rate for the 2024 alloy processed by ECAP (Ref 31) and for other similar alloys subjected to thermomechanical processing. Source: Ref 33-36

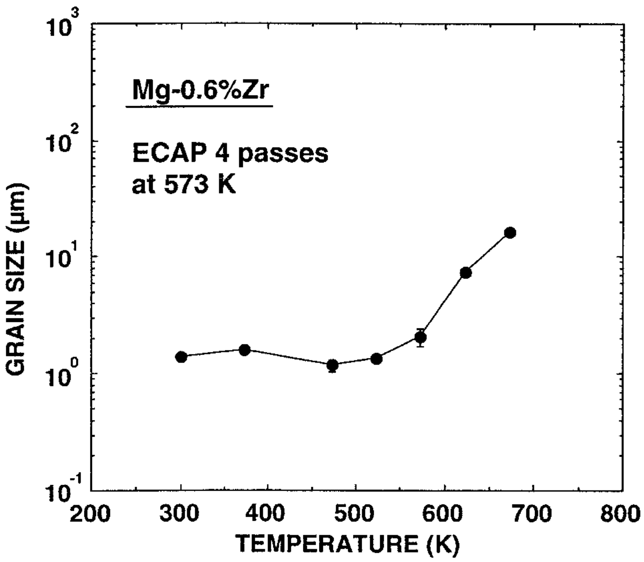

Fig. 5 Grain size versus annealing temperature for a Mg-0.6%Zr alloy after EX-ECAP through four passes at 573 K. Source: Ref 40

achieved through ECAP processing appear very favorable by comparison with other reported results where superplastic elongations were achieved in the 2024 or similar alloys. This comparison is shown in Fig. 4 in which the elongation to failure is plotted against the strain rate for alloys subjected to thermomechanical processing (TMP) and two of the alloys were produced using powder metallurgy (PM) techniques (Ref 33-36). Inspection of Fig. 4 shows that by comparison with the present results obtained on the 2024 alloy, high strain rate superplasticity is achieved in the two alloys produced using PM methods but the other alloys do not exhibit high elongations at very rapid strain rates. Furthermore, the results achieved on the 2024 alloy through ECAP processing are especially encouraging because as a direct consequence of the exceptionally fine grain sizes achieved through severe plastic deformation, superplasticity is attained at a testing temperature of only 673 K which is the lowest temperature recorded for all of the specimens in Fig. 4.

3.2 Mg-0.6%Zr

Magnesium alloys have many attractive features for use in structural applications including low density, good machinability, and high damping capacity. In practice, however, the use of magnesium-base alloys is restricted because they have a close-packed hexagonal structure that gives only limited ductility at ambient temperatures. An early attempt to apply ECAP processing to samples of pure magnesium and an Mg-0.9%Al alloy was unsuccessful (Ref 37): specifically, the grain size of the pure magnesium was reduced only from ~400 to ~100 μm after two passes at 673 K and the grain size of the magnesium-aluminum alloy was reduced only from ~100 to ~17 μm after two passes at 473 K.

Subsequently, it was demonstrated that ultrafine grain sizes may be achieved using ECAP with dilute magnesium alloys by employing a two-step processing procedure in which the samples are initially extruded and then subjected to ECAP (Ref 38). This process, termed EX-ECAP, is effective because the initial extrusion serves to produce a texture in which a majority of the basal (0001) planes lie parallel to the extrusion direction (Ref 39). To date, the EX-ECAP process has been used to produce ultrafine grain sizes in several magnesium alloys: Mg-0.6%Zr (Ref 38, 40), Mg-9%Al (Ref 41), and Mg-7.5%Al-0.2%Zr (Ref 42).

An earlier report provides a detailed description of the application of EX-ECAP to a Mg-0.6%Zr alloy (Ref 40), where this alloy was selected because it has the optimum composition for achieving a high damping capacity. The cast alloy had an initial grain size of ~1.4 mm, but this was reduced to ~55 μm after extrusion at 623 K using a reduction ratio of 36:1, and it was further reduced to ~1.4 μm after ECAP through four passes at 573 K. Figure 5 shows the thermal stability of these ultrafine grains for samples annealed for 1 h over a range of temperatures to 673 K. It is apparent that the grains are reasonably stable up to 573 K, but there is some grain growth at higher temperatures. These results suggest, therefore, the potential for achieving high ductilities in tensile testing at a temperature of 573 K.

The results from tensile testing are recorded in Fig. 6 for four different conditions: the as-received cast condition where the elongation to failure is close to ~100%, the cast and extruded condition where the elongations increase to a maximum close to 200% at the lowest strain rate, the EX-ECAP condition where the sample was subjected to ECAP for one pass at 573 K, giving an elongation close to 400% at the lowest experimental strain rate, and the cast material subjected to ECAP for one pass at 573 K without any intermediate extrusion where the elongation at the lowest strain rate is only ~150%. These results demonstrate the validity of the two-step EX-ECAP process in producing materials capable of exhibiting high elonga-

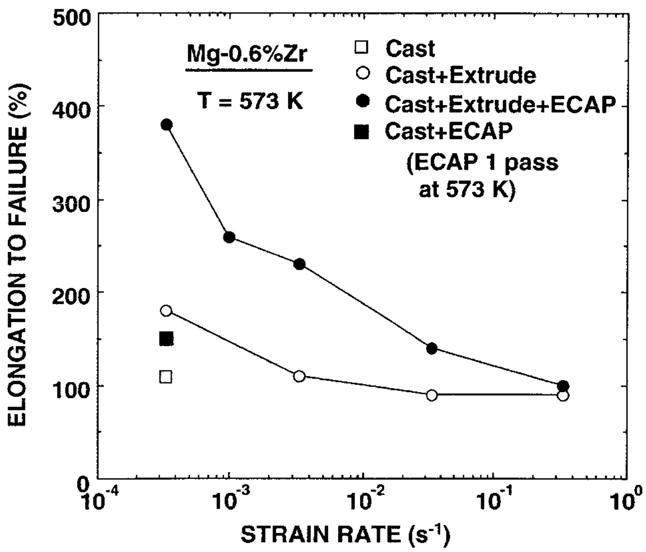

Fig. 6 Elongation-to-failure versus strain rate for samples of the Mg-0.6%Zr alloy prepared by casting, casting and extrusion, casting and ECAP, or casting and the EX-ECAP process. Source: Ref 40

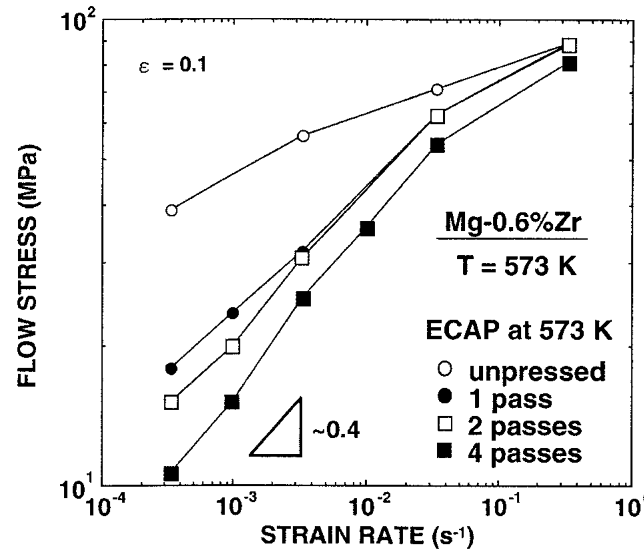

Fig. 8 Flow stress versus strain rate for the Mg-0.6% Zr alloy after using the EX-ECAP process. Source: Ref 40

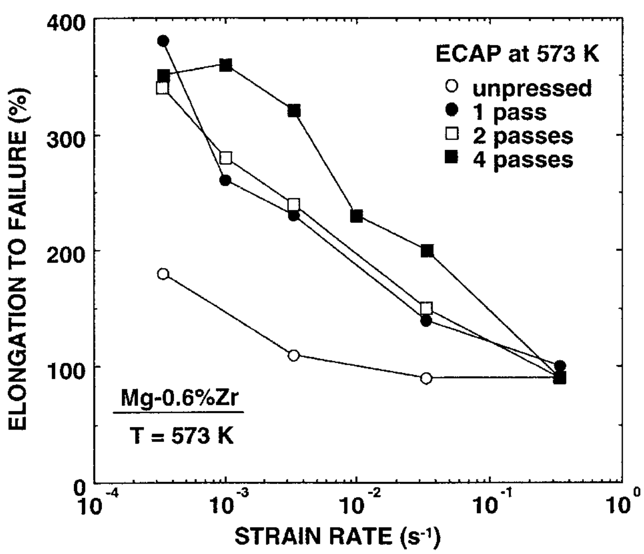

Fig. 7 Elongation to failure versus strain rate for the Mg-0.6%Zr alloy showing the effect of different numbers of passes when using the EX-ECAP process. Source: Ref 40

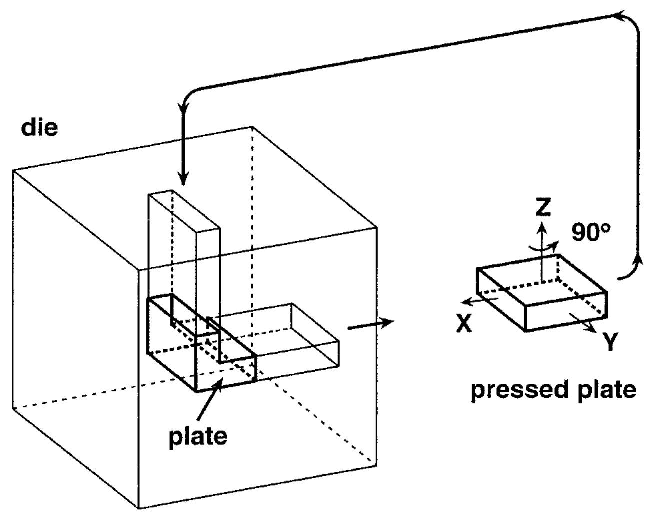

Fig. 9 Schematic illustration of the principle of ECAP with a plate sample. Source: Ref 44

tions to failure. Furthermore, the elongations tend to increase slightly with increasing numbers of passes in ECAP, as shown in Fig. 7, in which samples were pressed at 573 K through totals of 0, 1, 2, and 4 passes. Figure 8 reformats the experimental data for the specimens shown in Fig. 7 by measuring the flow stress at a strain (ε) of 0.1 at a testing temperature of 573 K, and plotting the value logarithmically against the strain rate. These results show the flow stress is reduced significantly through the application of ECAP, and this is consistent with the much smaller grain size produced in these samples. The strain rate sensitivity (m), defined as the slope of the lines in Fig. 8, is given by $m \approx 0.4$ at the lowest strain rates for the samples pressed through 1, 2, and 4 passes where the elongations are a maximum. This value of m is intermediate between the values of $m = 0.5$ anticipated for true superplasticity (Ref 14) and $m = 0.3$ anticipated for a dislocation glide process (Ref 43).

4. Application of ECAP to Plate Samples

The results described in the preceding section demonstrate the potential for using ECAP to achieve superplastic characteristics in bulk metallic alloys. However, the results relate specifically to the pressing of rod or bar samples having circular cross sections, and these samples are not easy to use in industrial SPF operations where the forming operations are generally conducted on sheet samples. It is appropriate, there-

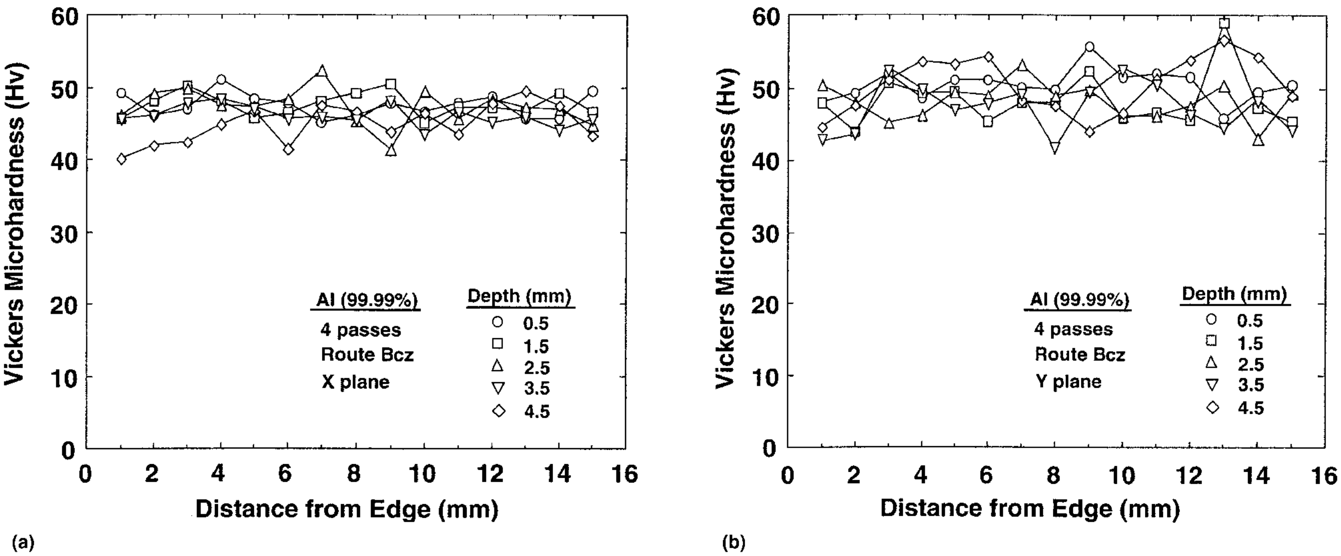

Fig. 10 Vickers microhardness from the edge to the center of the plate at different depths. (a) On the X plane. (b) On the Y plane. Source: Ref 44

fore, to evaluate the possibility of applying the ECAP process directly to plate samples. An earlier report described the principles of ECAP with plate samples (Ref 44).

The pressing of plate samples introduces some significant differences by comparison with the conventional pressing of rods. Thus, it is no longer feasible to use processing route B_C where the sample is rotated by 90° in the same sense around the pressing axis between each pass. The situation for the pressing of plate samples is illustrated schematically in Fig. 9, in which the channel within the die consists of a narrow slit and the plate is pressed through the die contained within this channel. Three orthogonal directions, X, Y, and Z, are defined in Fig. 9, where these directions follow the same convention indicated in Fig. 1. Although processing route B_C is no longer feasible for rotation around the X-axis, samples may be rotated between passes by 90° in the same sense around the Z-axis in the procedure henceforth designated route B_{CZ}, where the second subscript is needed to identify the axis of rotation.

Samples of pure aluminum, in the form of small plates, were pressed through four passes at room temperature using route B_{CZ}, and the homogeneity of the as-pressed samples was evaluated by taking measurements of the Vickers microhardness across the samples on the X and Y planes (Ref 44). These microhardness measurements are shown in Fig. 10 for (a) the X plane and (b) the Y plane, respectively, where the measurements were taken from the edge to the center of the plate at depths ranging from 0.5 to 4.5 mm from the top surface. Inspection of Fig. 10 shows that there is some scatter, but nevertheless all of the points lie within a reasonably narrow band suggesting a high degree of homogeneity within the plate after pressing.

The presence of homogeneity in plate samples after ECAP is supported also by the plots of true stress versus elongation in Fig. 11 in which two curves are shown for an Al-1%Mg-0.2%Sc alloy pressed through four passes using route B_{CZ} and tested in tension at 673 K using an initial strain rate of 1.0×10^{-3} s^{-1}. These two curves correspond to tensile specimens cut parallel

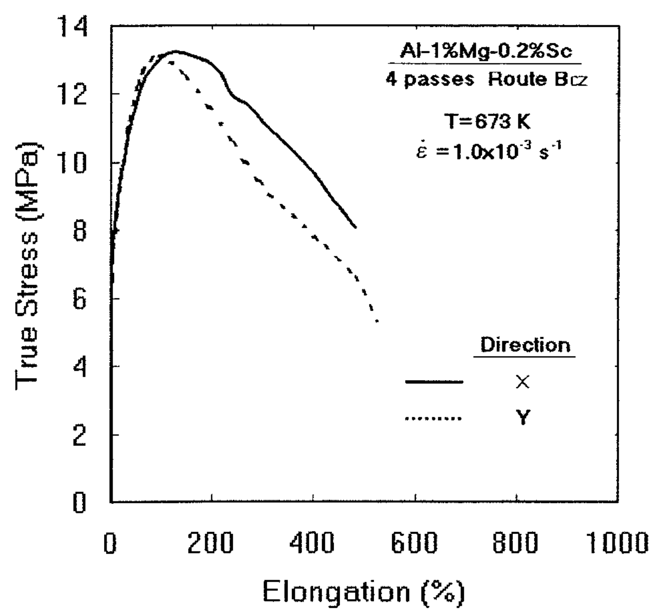

Fig. 11 True stress versus elongation for tensile specimens cut from a plate sample of an Al-1%Mg-0.2%Sc alloy with the samples lying parallel to the X or Y directions. Source: Ref 45

to the X and Y directions, respectively (Ref 45). Since these two curves are almost identical, it is reasonable to conclude that the pressing of plate samples leads to a high level of microstructural homogeneity. Thus, the extension of ECAP to plate samples appears to provide an opportunity for producing bulk materials that can be readily used in SPF operations.

5. Discussion

Processing by ECAP leads to grain refinement and arrays of ultrafine grains that are significantly smaller than those generally produced using conventional thermomechanical processing. In addition, and by contrast to TMP techniques, ECAP is a simple process that can be readily applied to a wide range of materials without the requirement of developing specific and different treatments for each alloy composition. The presence of these exceptionally small grain sizes provides an opportunity for achieving superplastic ductilities, and thus a superplastic forming capability, at strain rates faster than those associated with conventional SPF processes.

The present report demonstrates that ECAP processing is effective with commercial aluminum alloys such as 2024, and also, when combined with an extrusion step in the EX-ECAP process, with magnesium alloys. The demonstration of good tensile ductilities with the aluminum 2024 alloy is important because three aluminum alloys (2004, 5083, and 7475) currently account for approximately 90% of the current usage of aluminum-base alloys in the SPF industry (Ref 46).

Although ECAP processing is usually conducted using small rods or bars with cross-sectional dimensions in the range of ~1.0 to 1.5 cm, it has been shown that the ECAP process can be easily scaled to produce much larger samples (Ref 47). There are also experimental results showing that there is no reduction in the superplastic properties produced by ECAP in rod samples when the material is rolled to form a sheet metal (Ref 48). Alternatively, the extension of the ECAP processing technology to plate samples provides an opportunity for the direct production of metals suitable for use in the SPF industry. It is reasonable to anticipate, therefore, that the utilization of this new technology may provide the opening to significantly expand the so-called techno-economic niche associated with the current SPF technology.

6. Summary and Conclusions

The processing of metallic alloys by ECAP gives exceptional grain refinement and, if these ultrafine grains are reasonably stable at high temperatures, the opportunity to achieve superplastic elongations at high strain rates. Examples are presented for an aluminum and a magnesium alloy.

Processing by ECAP may be extended to plate samples, thereby permitting the production of materials in a form suitable for superplastic forming operations.

Acknowledgment

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