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Improving the mechanical properties of magnesium and a magnesium alloy through severe plastic deformation

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Abstract

Pure Mg and Mg alloys generally exhibit only limited ductilities at ambient temperatures. Experiments were conducted to evaluate the potential for improving the mechanical properties of pure Mg and an Mg–0.9% Al alloy at room temperature by subjecting these materials to severe plastic deformation through the procedure of equal-channel angular pressing (ECAP). It is shown that ECAP may be applied successfully to these materials at elevated temperatures and this leads to grain refinement due to the occurrence of recrystallization during the pressing process and to significant improvements in the strength and ductility of these materials. Since these improvements are apparent after only a single pass through the ECAP die, it is concluded that the introduction of ECAP provides a simple and effective procedure for improving the ambient temperature mechanical properties of materials, such as hcp metals, where the measured ductilities are generally limited.

Keywords: Equal-channel angular pressing; Magnesium; Mechanical properties; Severe plastic deformation; Ultrafine grain size

1. Introduction

Considerable interest has developed recently in the potential for processing materials using severe plastic deformation (SPD) [1]. The principle of SPD is that it gives a very large strain in the sample and the rearrangement of the dislocations introduced by straining leads to a very substantial grain refinement down to, typically, the submicrometer or even the nanometer scale [2]. Thus, SPD offers the possibility of refining the grain size to levels that are significantly smaller than those produced using conventional thermomechanical processing.

Two different procedures are generally used for SPD [3]. First, there is equal-channel angular pressing (ECAP), in which a bulk sample is pressed through a die and a strain is induced without any change in the cross-sectional dimensions of the work-piece. Second, there is high-pressure torsion (HPT), in which a sample is subjected to torsion straining under a high pressure. There is experimental evidence suggesting that greater grain refinement may be achieved using HPT, but, nevertheless, there are advantages with ECAP because it gives large bulk samples and there is the possibility of scaling-up for commercial applications using multi-pass facilities where large strains are introduced in a single pressing [4].

In principle, Mg alloys have many potential applications because of their low density and good machinability, but, as a consequence of their hcp structure, they generally exhibit only limited ductility at ambient temperatures. To date, there has been no attempt to improve the room temperature mechanical properties of Mg and Mg-based alloys through the use of ECAP, although there are reports of improved high-temperature properties after ECAP with the AZ91 alloy (Mg–9% Al–1% Zn–0.2% Mn) where the grain size was reduced to ~ 0.5–1 μm [5–8]. The present investigation was initiated both to examine the potential for grain refinement in samples of pure Mg and a dilute Mg–Al

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alloy and also to investigate the subsequent mechanical properties of these materials at room temperature. As will be demonstrated, ECAP is reasonably effective in decreasing the grain size of pure Mg and an Mg–Al alloy through recrystallization during pressing, and the as-pressed materials exhibit significant improvements in both strength and ductility.

2. Experimental materials and procedures

Ingots of pure Mg (99.9% purity) and an Mg–0.9 wt% Al alloy were supplied by Ube Industries, Ltd (Ube, Japan) with an initial thickness of ~ 25 mm. These pieces were rolled to a thickness of 12 mm at a temperature of ~ 400°C and then cut into rods with dimensions of $12 \times 12 \times 60$ mm^3. All rods for both materials were machined to diameters of 10 mm and the rods were then annealed for 1 h at temperatures of 500°C and 400°C for the pure Mg and the Mg–0.9% Al alloy, respectively. Following the annealing treatments, the grain sizes were measured as ~ 400 μm in pure Mg and ~ 100 μm in the Mg–0.9% Al alloy.

All samples were pressed using an ECAP die having a channel diameter of 10.3 mm and with an internal angle Φ between the two parts of the channel of 90°; the ECAP facility is illustrated schematically in Fig. 1. As indicated in the illustration, the angle Ψ representing the outer arc of curvature at the point of intersection of the two channels was ~ 45°. It can be shown that these values of Φ and Ψ lead to an imposed strain of ~ 1 on each passage of the sample through the die [9]. The solid die was constructed from SKD61 tool steel and it was placed within a furnace so that there was a heater surrounding the die and another heater immediately below the die. The temperature for each pressing was controlled using a thermocouple that passed through the wall of the furnace and was then inserted in a hole drilled into the die to a point within ~ 5 mm of the channel wall at the initial point of bending; the location of the thermocouple is shown in Fig. 1. Samples were pressed at temperatures of 200, 300 and 400°C using a plunger attached to a hydraulic press operating at a pressing speed of 18 mm s^{-1}; during pressing, the temperatures were continuously monitored and controlled to within ± 5°C. For each set of pressings at each temperature, the die was brought up to the required temperature over a period of ~ 1 h and then held at temperature for ~ 10 min for stabilization. For each separate pressing, the sample was sprayed with a lubricant used for die casting and it was then placed in the channel after the die had reached the required temperature. It was held in the die during the temperature stabilization for ~ 10 min and it was then pressed through the die and removed using a dummy Al sample. For each subsequent pressing at this temperature, the sample was held at temperature in the die for ~ 10 min to ensure temperature stability.

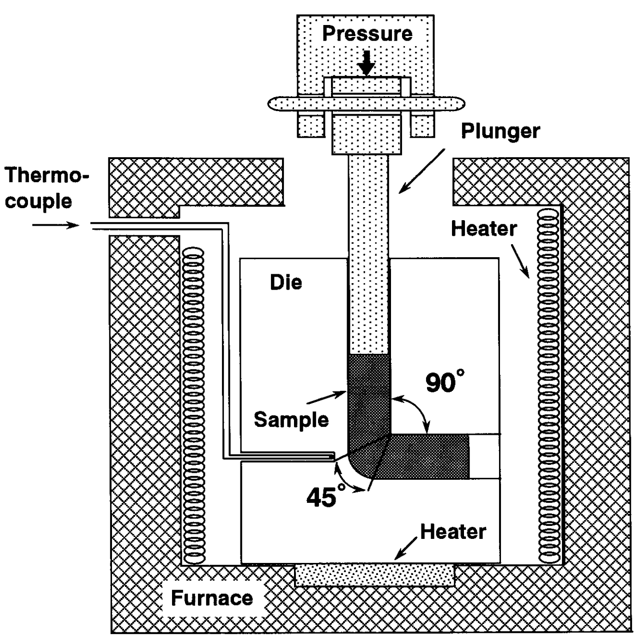

Fig. 1. Schematic illustration of the facility used for ECAP.

All pressings were conducted using processing route B_C, in which the sample was removed from the die and then rotated by $+90$° in the same direction between each pass [10]. This procedure was adopted because there is evidence from experiments on pure Al that it leads most expeditiously to an array of equiaxed and ultrafine grains separated by high-angle grain boundaries [11]. Pressings were conducted to a maximum of four passes through the die; for pure Mg at 400°C and the Mg–0.9% Al alloy at 200°C, the samples were taken to only two passes because of the occurrence of cracking at larger numbers of passes.

Following ECAP, the pressed samples were sliced into discs with thicknesses of ~ 5 mm, with the disks taken near the centers of the rods and oriented perpendicular to the longitudinal axes. These discs were used to observe the grain structures on the X plane where this plane is defined as lying perpendicular to the direction of pressing [10]. The discs were polished using abrasive SiC papers and 0.3 μm Al_2O_3 and they were etched for ~ 15 s at room temperature using a solution of 1% HNO_3, 24% $C_2H_6O_2$ and 75% water. The average grain size was determined for each condition by recording the sizes of more than 100 grains at places where the grains were surrounded by well-defined grain boundaries.

Tensile specimens were machined from the as-pressed samples with gauge sections of $2 \times 3 \times 5$ mm^3 and with the gauge lengths lying parallel to the longitudinal axes. These specimens were tested at room temperature using a machine operating at a constant rate of crosshead displacement with an initial strain rate of 3.3×10^{-4}

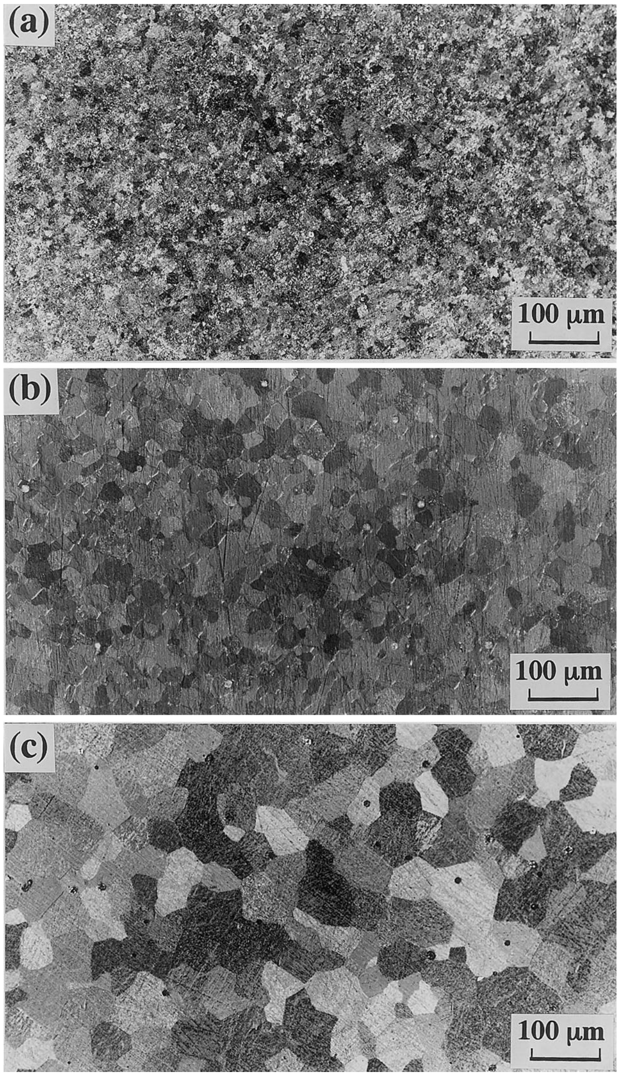

Fig. 2. Typical microstructures in Mg–0.9% Al after ECAP for two passes at temperatures of (a) 200°C, (b) 300°C and (c) 400°C.

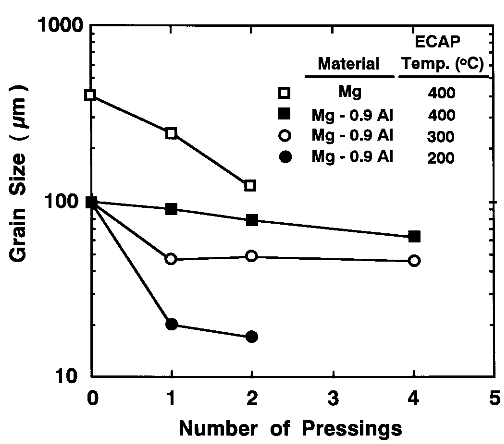

Fig. 3. Grain size versus number of pressings through the ECAP die for all pressing conditions.

s^{-1}. The variation of load was recorded for each test and these stress–strain curves were used to determine the 0.2% proof stress, the ultimate tensile stress (UTS) and the elongation to failure.

3. Experimental results

3.1. Microstructures after ECAP

Typical microstructures are shown in Fig. 2 for the Mg–0.9% Al alloy after pressing for two passes to a strain of ~ 2 at temperatures of (a) 200°C, (b) 300°C and (c) 400°C. The average grain sizes were measured for these three conditions as ~ 17, ~ 48 and ~ 78 μm for pressing temperatures of 200, 300 and 400°C, respectively. These measurements show, therefore, that ECAP is effective in reducing the grain size of this Mg-based alloy. However, the recorded grain sizes are significantly larger than in dilute Al–Mg alloys, where the as-pressed grain sizes are < 1 μm [12]. Inspection of Fig. 2 shows that the grains are reasonably equiaxed and homogeneously distributed for each pressing temperature. This homogeneity after only two passes suggests that recrystallization occurs during ECAP, and the advent of recrystallization is consistent also with the occurrence of larger grain sizes in the Mg alloy by comparison with pure Al [13] and commercial Al-based alloys [14].

Fig. 3 records the variation in the average grain size with the number of pressings for Mg after pressing at 400°C and for the Mg–0.9% Al alloy after pressing at temperatures from 200 to 400°C. This plot demonstrates that the presence of dilute Al reduces the grain size of Mg in the unpressed condition and, in addition, the grain size further decreases with additional pressings in ECAP under all experimental conditions. It is also apparent that ECAP is especially effective in reducing the grain size when the pressing is conducted at lower temperatures, where the occurrence of grain growth is limited.

3.2. Mechanical properties at room temperature

Plots of true stress versus true strain are shown in Fig. 4 for pure Mg tested at room temperature at an initial strain rate of 3.3×10^{-4} s^{-1}. It is evident from these curves that there is a significant increase in the strength and ductility of Mg after one or two passes through the ECAP die, since failure occurs at a true strain of only ~ 0.004 in the unpressed condition (labeled 0 p in Fig. 4). Similar curves are shown in Fig. 5 after ECAP of Mg–0.9% Al at 200°C (upper), 300°C (center) and 400°C (lower). Again, there is a very significant improvement in strength and ductility even after one pass through the die.

The values of the 0.2% proof stresses and the UTS are summarized in Fig. 6 for all pressing conditions. These results show that, by comparison with pure Mg, the addition of dilute Al increases both the proof stress and the UTS through ECAP and they are consistent with the grain sizes recorded in Fig. 3, including the very significant effect after a single pass through the ECAP die. The large differences in the values of the 0.2% proof stresses and the UTS after ECAP demonstrate the occurrence of significant strain hardening in these materials. A similar plot is shown in Fig. 7 for the

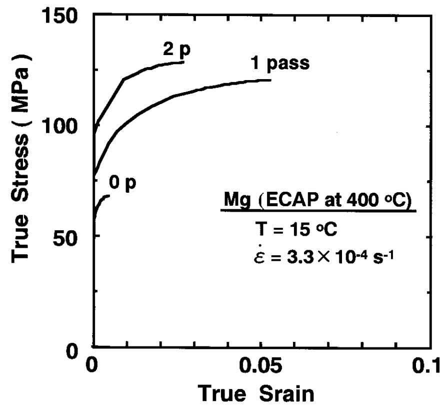

Fig. 4. True stress versus true strain at room temperature for samples of pure Mg in the unpressed condition (0 p) and after ECAP for one pass and two passes at 400°C.

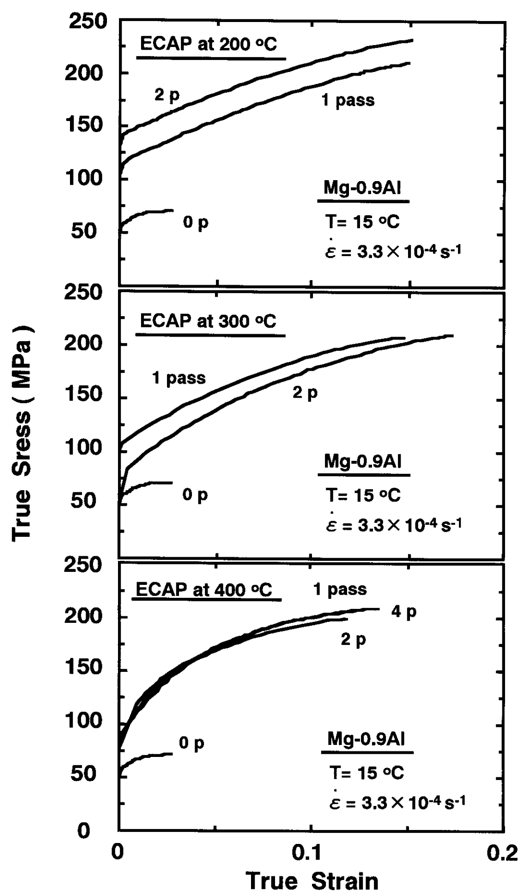

Fig. 5. True stress versus true strain at room temperature for samples of Mg–0.9% Al in the unpressed condition (0 p) and after ECAP at 200°C (upper), 300°C (center) and 400°C (lower).

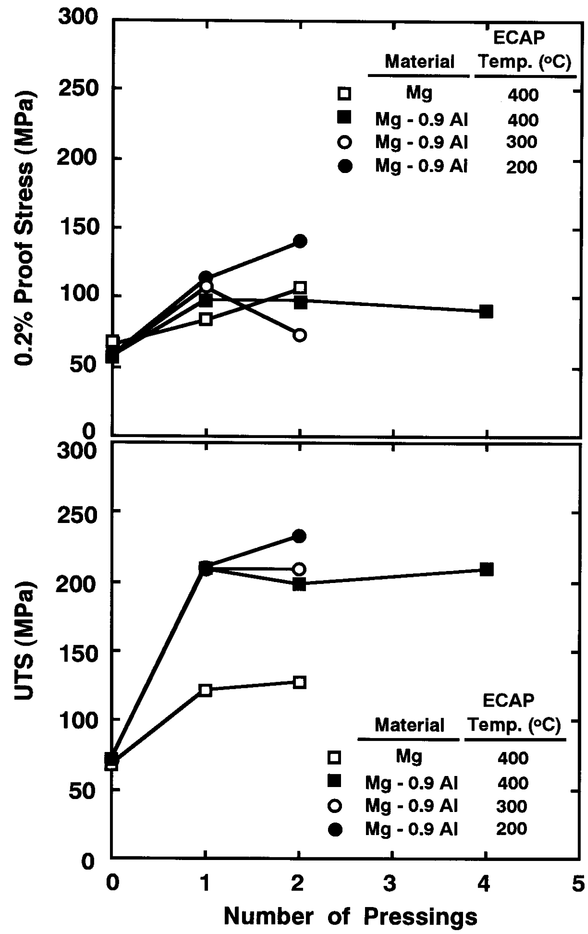

Fig. 6. Variation of the 0.2% proof stress (upper) and the UTS (lower) with the number of pressings for all pressing conditions.

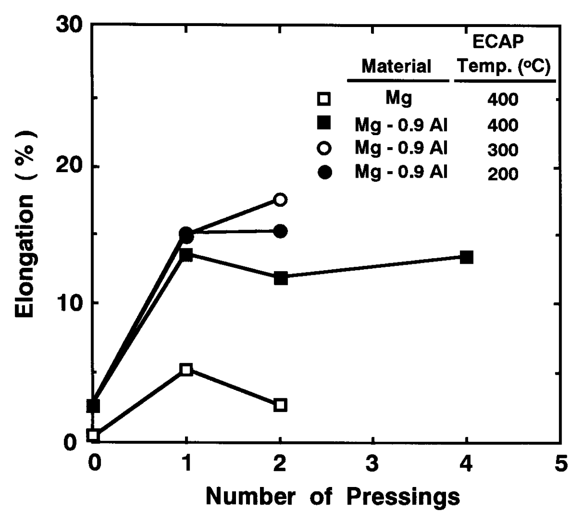

Fig. 7. Elongation to failure versus number of pressings at room temperature for all pressing conditions.

measured elongations to failure for each pressing condition. It is concluded from this plot that the presence of dilute Al leads to higher ductility in the unpressed condition and that ECAP through one or more passes is especially effective in increasing the elongations to failure.

4. Discussion

The results from these tests demonstrate conclusively that ECAP is effective in improving the mechanical properties of pure Mg and an Mg–0.9% Al alloy at room temperature. Pressing refines the grain size even after a single pass through the ECAP die, and this leads to increases in both the strength and ductility of these materials. However, there is a significant difficulty in conducting ECAP on pure Mg and Mg alloys. On the one hand, the restricted number of slip systems, and therefore the limited ductility, in hcp metals requires that the pressing is conducted at an elevated temperature. On the other hand, as shown in Fig. 3, smaller grain sizes are achieved when ECAP is performed at the lowest possible temperature.

To determine whether these results are consistent with the standard Hall–Petch relationship, in which the yield stress is related to the square root of the grain size [15,16], Fig. 8 shows a plot for both materials of the 0.2% proof stress against $d^{-1/2}$ where d is the grain size; the arrows in Fig. 8 indicate the data points for samples in the unpressed condition. These plots show consistency with the Hall–Petch relationship and they confirm that the additional strengthening introduced by ECAP is a direct consequence of the grain refinement. This trend is also confirmed in Fig. 9, which plots the elongation to failure against the grain size for each pressing condition; again, the arrows indicate the unpressed materials. Thus, contrary to the characteristics of many strain-hardened metals, ECAP leads to a grain refinement through recrystallization and this gives rise to an increase in both the strength and the ductility of pure Mg and the Mg–0.9% Al alloy at room temperature.

An important question concerns the inability to achieve submicrometer grain sizes in these two materials through ECAP. By contrast, earlier reports of ECAP with the AZ91 alloy gave grain sizes in the range ~0.5–1 μm after pressing at a temperature of 175°C [5–8]. This difference is probably due to the much higher Al addition in the AZ91 alloy, since this should further decrease the grain size in the unpressed material and thereby permit ECAP at lower temperatures. In the present investigation, brittleness of the unpressed samples precluded the pressing of pure Mg below a temperature of ~400°C and the Mg–0.9% Al alloy below ~200°C. The inherent brittleness in these materials is a consequence of the limited number of slip systems in hcp metals and a failure to fulfill the von Mises criterion of five independent slip systems for homogeneous polycrystalline deformation. Thus, there is a build up of stress concentrations at selected points within these materials during pressing and, if these stress concentrations are not dissipated through pressing at a high temperature, they lead to a brittleness that is especially acute when the grain size is large.

The reduced grain sizes introduced by ECAP in pure Mg and the Mg–0.9% Al alloy are a consequence of recrystallization during the pressing process. This recrystallization leads to an equiaxed and homogeneous grain structure, as shown in Fig. 2, and to a marked

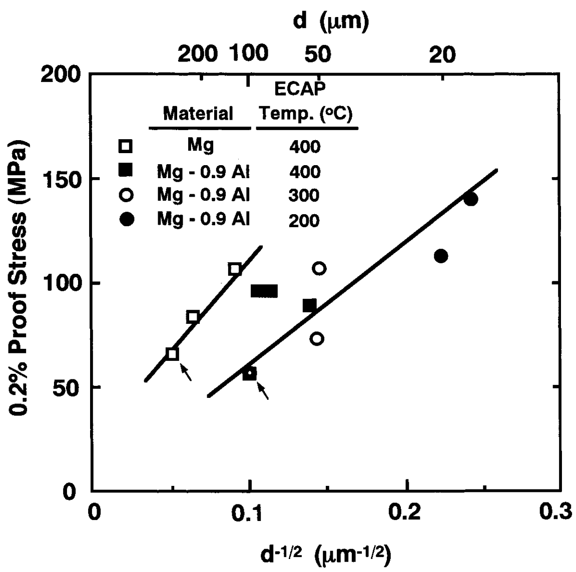

Fig. 8. Values of the 0.2% proof stress versus the reciprocal of the square root of the grain size for all pressing conditions; arrows indicate unpressed samples.

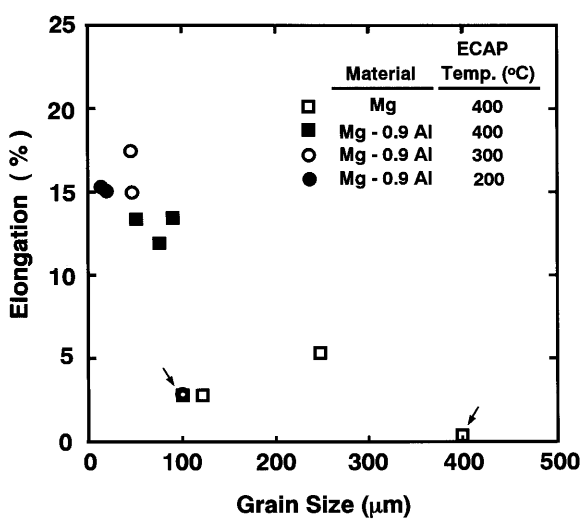

Fig. 9. Elongation to failure versus grain size for all pressing conditions; arrows indicate unpressed samples.

improvement in the room temperature mechanical properties. This is because the strength is increased through the Hall–Petch relationship and because the ductility is increased because more grains contribute to the macroscopic deformation and the stress concentrations are accordingly reduced and spread over a wider area. It is therefore concluded that ECAP provides a simple and effective procedure for improving at ambient temperatures the mechanical properties of materials, such as Mg-based alloys and other hcp metals, where the potential commercial applications are often limited because of the exceptionally poor ductility.

5. Summary and conclusions

(1) ECAP was successfully applied to pure Mg and an Mg–0.9% Al alloy, but only at temperatures of at least 400°C and 200°C respectively for these two materials.

(2) The grain sizes of the materials were reduced under all pressing conditions due to the occurrence of recrystallization during pressing, which led to a homogeneous distribution of essentially equiaxed grains.

(3) For both materials, the use of ECAP gave a significant improvement in both strength and ductility; furthermore, this improvement was achieved after only one pass through the ECAP die, and there was little or no additional improvement with subsequent passes.

(4) It is concluded that the use of ECAP provides a simple and effective procedure for improving the mechanical properties of hcp metals at ambient temperatures.

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