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Developing a strategy for the processing of age-hardenable alloys by ECAP at room temperature

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ABSTRACT

It is well known that age-hardenable alloys are generally difficult to process by equal-channel angular pressing (ECAP) at room temperature because they invariably fail by catastrophic cracking or segmentation. Experiments were conducted on two supersaturated Al–Zn–Mg alloys with the objective of developing a strategy for the processing of age-hardenable alloys at room temperature. The results from these experiments demonstrate that successful pressing may be undertaken by conducting the pressing operation very quickly (typically within <10 min) following a water quench from the solution heat-treatment temperature. It is also shown that there is a significant increase in strength in the alloys even when the ECAP is performed through only a single pass. This latter result has important implications for industrial processing.

1. Introduction

The processing of metals through the introduction of severe plastic deformation (SPD) is attractive because it introduces significant grain refinement in bulk solids [1]. Much attention has been directed recently to the SPD procedure of equal-channel angular pressing (ECAP) in which a sample, in the form of a rod or bar, is pressed repetitively through a die where it is constrained within a channel bent through a sharp angle to impose a high strain [2]. Some materials, such as hexagonal close-packed alloys, are difficult to process by ECAP because of the limited number of slip systems and the potential for segmentation of the billet and multiple cracking when pressing at room temperature (RT) [3]. These problems may be limited or even avoided by increasing the processing temperature and/or the strain rate sensitivity of the material and/or the channel angle within the die [4–6]. However, an increase in the pressing temperature leads to larger grain sizes. Similar difficulties arise in the ECAP processing of age-hardenable aluminum alloys at RT where the formation of metastable precipitates limits the deformability of the billets. In the case of age-hardenable Al–Zn–Mg alloys, there is an additional difficulty because there is evidence that successful processing may depend upon the inter-relationship between the solution heat-treatment and the subsequent aging prior to SPD processing [7–9].

The present investigation was initiated with the objective of developing a strategy that may be used to successfully process age-hardenable alloys by ECAP at room temperature. The experiments were conducted using two Al–Zn–Mg (7xxx) alloys. The aging and precipitation characteristics of this alloy system are described in the following section and the experimental procedures and results are given in the subsequent sections.

2. Aging and precipitation characteristics in the Al–Zn–Mg system

The chemical composition and the aging conditions influence the decomposition of supersaturated solid solutions (SSS) in the Al–Zn–Mg ternary system [10–14]. The decomposition process near room temperature takes place by the formation of Guinier–Preston (GP) zones but at higher aging temperatures there is an intermediate metastable η'-phase and at even higher temperatures

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there are stable or equilibrium η-phase precipitates. Only a limited number of results are available describing the early stages of decomposition in these alloys [13,15]. It is well known that the precipitation and strength of supersaturated Al–Zn–Mg alloys may be effectively changed by the application of a small pre-strain introduced using conventional tension or compression [16]. Recently, processing by ECAP was used to change the microstructures and improve the mechanical properties of supersaturated alloys [7–9,17–19]. However, the application of SPD to precipitation-hardened Al–Zn–Mg alloys is more complicated. On the one hand, because of the hardening effect of precipitates in the form of GP zones and/or other metastable particles, the samples may break during processing by ECAP at RT. To avoid this problem, supersaturated Al–Zn–Mg samples are generally processed at elevated temperatures. On the other hand, at high temperatures there is additional precipitation and a recovery of the ultrafine-grained microstructures.

Recent results have shown that the application of ECAP for 8 passes at a temperature of 473 K leads to the formation of stable η-phase ($MgZn_2$) precipitates in supersaturated Al–Zn–Mg alloys so that, despite the development of a fine-grained microstructure having an average grain size of ~500 nm, the room temperature strength of samples processed by ECAP decreases significantly relative to samples aged only at RT [19]. These data demonstrate, therefore, that the application of SPD to supersaturated alloys at elevated temperatures may lead to a decrease in the overall strength rather than to additional strengthening as in pure metals and solid solution alloys.

Concerning the processing of age-hardenable Al–Zn–Mg alloys at RT, different pre-aging treatments have been performed to avoid cracking due to the strong strengthening effect of the GP zones during subsequent SPD processing [7,8]. For example, some samples were directly aged at 553 K for 5 h before SPD [7] while other samples were subjected to a slow furnace cooling instead of the usual quick water quenching from a solution temperature of 749 K to RT [8]. However, these treatments lead to an over-aging of the samples through the formation of relatively coarse and stable η-phase particles [10–12] and this usually decreases the strength of the Al–Zn–Mg materials. When an Al–Zn–Mg–Cu sample was cooled slowly in a furnace and subsequently processed by equal-channel angular rolling at RT, the maximum Vickers hardness, HV, was ~100 [8]. This value is much lower than the HV value of ~200 obtained for an almost similar composition after water quenching, subsequent naturally aging and processing by ECAP at RT [9]. These experimental results confirm that, in order to enhance the strength of supersaturated Al–Zn–Mg alloys by ECAP, it is reasonable to deform the materials at RT without any artificial pre-aging when the main precipitation hardening is due to GP zones.

Accordingly, the present investigation was conducted in order to critically evaluate the inter-relationship between natural aging and processing by ECAP at RT using two different Al–Zn–Mg alloys.

3. Experimental materials and procedures

Billets of two polycrystalline Al–Zn–Mg alloys, having compositions in wt.% of Al–4.8Zn–1.2Mg–0.14Zr and Al–5.7Zn–1.9Mg–1.5Cu, were initially subjected to a solution heat-treatment for 30 min at 743 K and then water-quenched to give an SSS. Following quenching, the samples were naturally aged at RT for different periods of time prior to processing by ECAP. Cylindrical billets of 10 mm in diameter and 70 mm in length were pressed at RT for different numbers of passes using a solid ECAP die having a channel angle of 90° and processing route B_c in which the billets are rotated in the same sense around their longitudinal axes by 90° between each pass [20]. The pressing was conducted at a constant displacement rate of 8 mm s^{-1} and the outer arc of curvature at the intersection of the two parts of the channel was 20° thereby giving an imposed strain of ~1 on each separate pass [21].

Indentation and microhardness measurements were undertaken after mechanical and electrolytic polishing using a Vickers indenter in a Shimadzu-DUH 202 depth-sensing ultra-microhardness machine operating with 2 N maximum load. The hardness measurements were taken along two randomly selected diameters on the cross-sectional planes and the reported values are the averages from at least 20 individual measurements. The error bars on these average values were estimated as <5%. Dislocation densities were determined by X-ray diffraction line profile analysis using a special high-resolution diffractometer (Nonius FR591) with Cu $K\alpha_1$ radiation. The line profiles were evaluated by the extended Convolutional Multiple Whole Profile (eCMWP) fitting procedure [22]. The TEM foils were taken from the center of the cross-section perpendicular to the axis of the exit channel for the last ECAP pass. Small disks were prepared with diameters of 3 mm and thicknesses of ~0.15 mm and they were mechanically ground and then thinned to perforation at 253 K using a twin-jet electropolishing unit with a solution of 33% HNO_3 and 67% CH_3OH. These disks were examined using a JEOL 200CX transmission electron microscope (TEM) operating at 200 kV.

4. Experimental results

In order to understand the kinetics of precipitation and its influence on the mechanical properties of the alloys, indentation measurements were carried out on the samples after natural aging at RT for different time periods, t_a. Typical indentation load–depth (F–h) curves are shown in Fig. 1 for the Al–Zn–Mg–Zr and Al–Zn–Mg–Cu alloys in the early stages of natural aging. Considering both the positions and the shapes of the indentation curves, the experimental results demonstrate there is an increasing hardness of the samples with increasing aging time due to the early formation of GP zones after quenching. It is also apparent that characteristic steps appear in the indentation load–depth (F–h) curves at the beginning of natural aging for both alloys where this is a typical plastic instability similar to the Portevin–Le Chatelier effect or jerky flow [23].

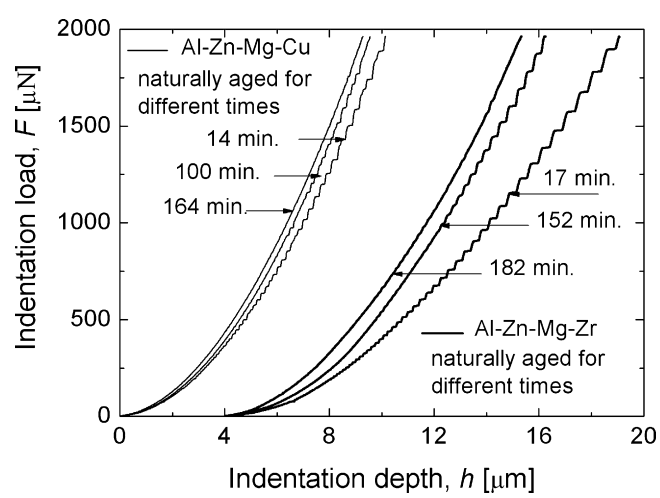

Fig. 1. Effect of natural aging on the indentation load–depth (F–h) curves taken by depth-sensing microhardness tests on supersaturated Al alloys: the curves for the Al–Zn–Mg–Zr alloy were arbitrarily shifted horizontally by 4 μm to provide clarity in presentation.

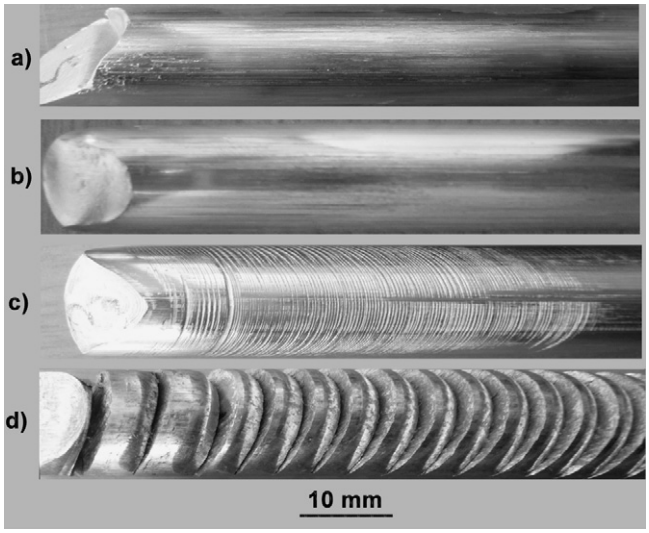

Fig. 2. The surface topography of billets processed by ECAP for 1 pass: (a) the Al–Zn–Mg–Zr alloy and (b) the Al–Zn–Mg–Cu alloy after quenching and pre-aging at room temperature for 10 min and (c) the Al–Zn–Mg–Zr alloy and (d) the Al–Zn–Mg–Cu alloy after quenching and pre-aging at room temperature for 1 week.

The instabilities in the load–depth curves observed in the very early stages of natural aging for both alloys, specifically within 20 min after quenching, are relatively regular and they are similar to those found in the stable Al–3Mg solid solution alloy [23]. However, when the aging time is increased, the steps occur less frequently and become more irregular and finally, after a certain time, t_i, they disappear. The values of t_i are typically between about 100 and 200 min but in the lower concentration Al–Zn–Mg–Zr alloy the steps are retained for a longer period.

The occurrence of plastic instabilities is caused by the interaction of diffusing solute atoms with moving dislocations in the phenomenon of dynamic strain aging (DSA) [24–27]. This implies that the presence of the load-indentation steps is related to a specific solute concentration in the alloys. The formation of these steps of instability at this stage of the process indicates that the GP zones are not sufficiently strong to suppress the DSA effect. The occurrence of these steps characterizes the transition, with a lifetime of t_i, from the SSS to an SSS + GP zone structure where the effect of GP zones on moving dislocations becomes dominant by comparison with that of the solute atoms. Therefore, the values of t_i give important information concerning the changes of the microstructure of supersaturated Al–Zn–Mg alloys in the range of the early formation of GP zones.

Fig. 2 shows four representative billets of the two alloys after a solution heat-treatment and water quenching followed by naturally aging at RT and then processing through 1 pass of ECAP at RT. Fig. 2(a) and (b) corresponds to the Al–Zn–Mg–Zr and the Al–Zn–Mg–Cu alloys, respectively, where there was pre-aging at RT for ~10 min prior to processing by ECAP where the pre-aging corresponds only to the small delay occurring between quenching and pressing. Fig. 2(c) and (d) shows the same two alloys where there was a pre-aging for an extended period of 7 days before processing by ECAP. It is apparent from Fig. 2 that the occurrence of natural aging after quenching, and specifically the length of this pre-aging, has a very significant influence on the development of strain localization and the formation of cracks during ECAP. Thus, in Fig. 2(a) and (b) the surfaces of both alloys are smooth after a short delay corresponding to an aging of only ~10 min at RT whereas in Fig. 2(c) the Al–Zn–Mg–Zr alloy shows the formation of intense shear bands after holding and aging of 1 week at RT. Furthermore, Fig. 2(d) shows that the Al–Zn–Mg–Cu alloy exhibits catastrophic cracking and segmentation when aging is permitted through 1 week at RT. These results confirm, therefore, the difficulty of processing age-hardenable Al–Zn–Mg alloys by ECAP and they further demonstrate the importance of conducting the ECAP almost immediately following a solution heat-treatment and quenching.

As a consequence of the nature of ECAP processing, shear bands are always formed. In the case of pure polycrystalline f.c.c. metals where the dislocations have high mobility, the shear bands are formed only on a microscopic scale and they are not instrumental in promoting failure. Samples of pure Al and Cu [28–30] may be processed easily by ECAP for up to 8 or more passes without the formation of catastrophic cracking whereas for supersaturated alloys it is necessary to consider the effect of precipitation. The macroscopic and almost catastrophic shear bands shown in Fig. 2(d) reflect the combined influence of the presence of GP zones and processing by ECAP.

Fig. 3 shows the Vickers hardness value, HV, as a function of aging time for (a) samples processed by 1 pass in ECAP and (b) as a function of the number of passes in ECAP for samples pre-aged for the shortest time of 10 min: for comparison, the hardness values obtained on the quenched and naturally aged samples without ECAP are also plotted in Fig. 3(a). Considering the effect of natural aging, it is noted that the strengthening effect of GP zones is grad-

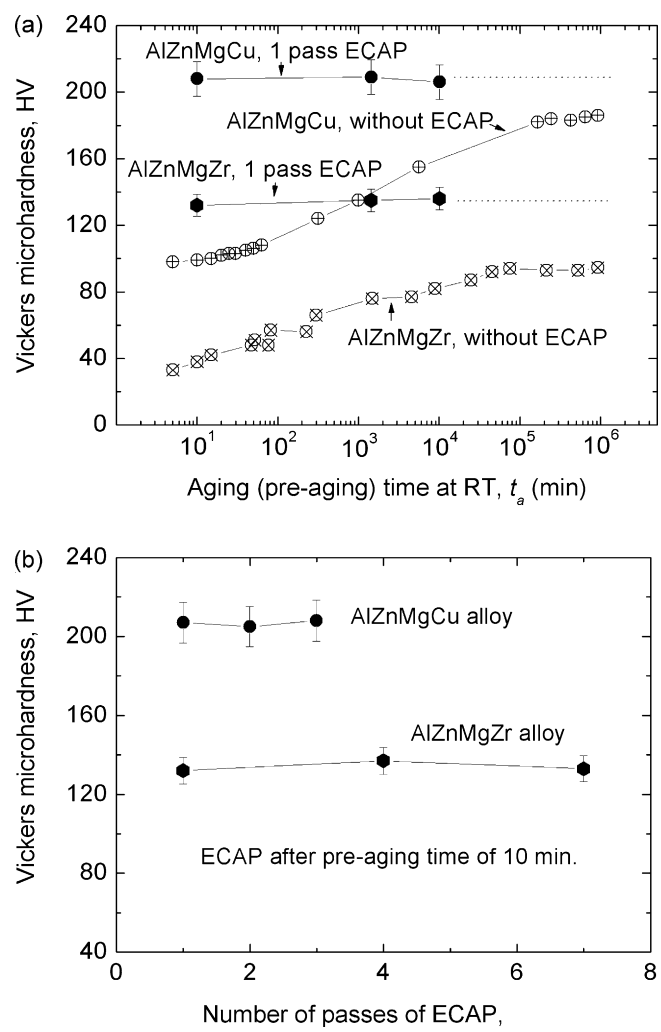

Fig. 3. The values of the Vickers microhardness (a) as a function of pre-aging time for the samples processed by only 1 pass in ECAP and (b) as a function of the number of passes in ECAP for samples pre-aged for 10 min: the hardness values recorded without ECAP are also shown in (a).

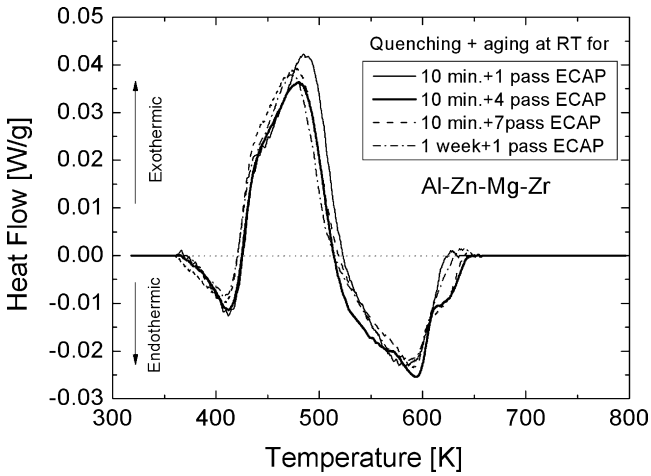

Fig. 4. DSC thermograms taken on Al–Zn–Mg–Zr samples processed by ECAP under different conditions.

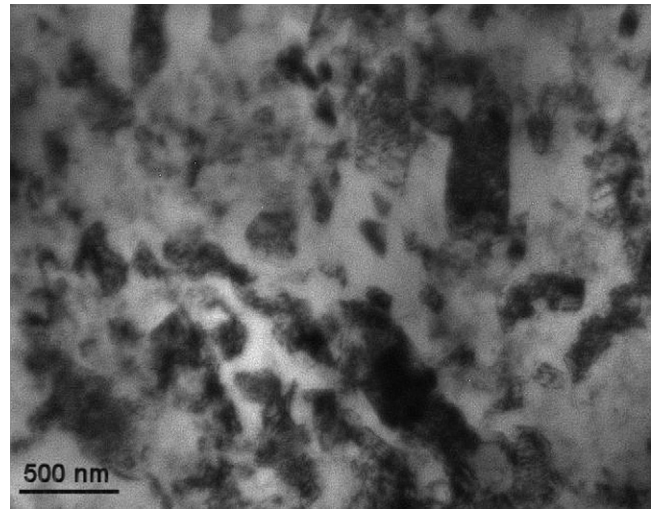

Fig. 5. A TEM image showing the grain structure in the Al–Zn–Mg–Zr alloy after processing through 4 passes in ECAP.

ually enhanced up to about 1 year of aging time through storage at RT and thereafter the strength becomes saturated. The corresponding saturation HV values of the lower concentration Al–Zn–Mg–Zr alloy and the higher concentration Al–Zn–Mg–Cu alloy are ~85 and ~185, respectively.

Fig. 4 shows the differential scanning calorimetry (DSC) thermograms taken on four AlZnMgZr samples naturally aged and processed by ECAP under different conditions. The dislocation densities determined by X-ray diffraction of these samples were found to be the same, $(6.5 \pm 1) \times 10^{14}\,\mathrm{m}^{-2}$, within the experimental error. It can be seen from Fig. 4 that the DSC curves after different pre-aging and ECAP coincide, thereby showing the same precipitation processes consisting of the dissolution of GP zones at the low temperature endothermic peak, the dissolution of the equilibrium η phase particles at the high temperature endothermic peak, and the formation of η'/η precipitates at the exothermic peak (intermediate region). This result demonstrates that similar GP zone microstructures are formed in different naturally aged and ECAP-processed samples which explains, together with the identical dislocation densities, the same strengths gained after ECAP depending neither upon the time period of pre-aging at RT nor upon the number of passes of ECAP. The promoting effect of ECAP on precipitation removed the differences in GP zone structures formed in the samples aged naturally for different times before ECAP.

In practice, it is well established that the application of more passes in ECAP leads to a higher fraction of boundaries having high angles of misorientation and this is important in promoting deformation mechanisms such as grain boundary sliding [31]. The TEM image in Fig. 5 shows the grain structure of the Al–Zn–Mg–Zr alloy processed through 4 passes by ECAP at RT. The average grain size in this condition was measured as ~300 nm and, for comparison, Table 1 lists the average grain sizes and average dislocation densities recorded after processing at RT and after processing through 8 passes at 473 K [19]. It is apparent that processing by ECAP at RT is advantageous because it leads to a smaller grain size, a larger dislocation density and a higher strength. These measurements confirm, therefore, the advantages of processing supersaturated alloys by ECAP at RT to avoid additional precipitation and microstructural recovery.

5. Discussion

The results from this investigation suggest two potential strategies for successfully processing age-hardenable alloys by ECAP. First, the alloys may be successfully pressed at elevated temperatures where GP zones and other strengthening precipitates are not formed. However, a disadvantage of this approach is that it leads to additional precipitation and microstructural recovery in the form of grain growth. Second, the alloys may be successfully processed at RT provided the pressing is performed immediately after quenching or at least within a very short pre-aging lifetime, t_a, of not more than ~10 min so that the GP zones are not strong. This latter strategy is the recommended processing route because it has the advantage of minimizing grain growth.

For processing at RT immediately after, or very shortly after, quenching from the solution heat-treatment temperature, there is a possibility that the high density of dislocations introduced in the first pass of ECAP may subsequently accelerate the formation of GP zones thereby producing a detrimental effect which will become evident in subsequent passes. In the present investigation, where the samples were processed almost immediately (to within ~10 min) after quenching, the lower concentration Al–Zn–Mg–Zr alloy was successfully pressed through 7 passes at RT but the more concentrated Al–Zn–Mg–Cu alloy was successfully pressed through a total of only 3 passes at RT. This is consistent with an earlier report for the Al-7075 alloy where samples were processed by ECAP for 2 passes at RT immediately following quenching [8].

The present experimental results demonstrate that processing by ECAP, even by a single pass, typically improves the strength or hardness by ~10–40% compared to the saturation hardness. Furthermore, for both alloy compositions the strength attained by ECAP is not significantly dependent either upon the time of pre-aging at RT or even upon the number of passes of ECAP. In the

Table 1
Microstructural and mechanical characteristics of the Al–Zn–Mg–Zr alloy after processing by ECAP at RT and at the elevated temperature of 473 K.

ECAP process	Average grain size determined by TEM	Average dislocation density determined by X-ray diffraction	Vickers microhardness
4 passes by route B_c at RT	300 nm	$(6.5 \pm 0.7) \times 10^{14}\,\mathrm{m}^{-2}$	135
8 passes by route B_c at 473 K [19]	500 nm	$(3.2 \pm 0.4) \times 10^{14}\,\mathrm{m}^{-2}$	85

case of the Al–Zn–Mg–Zr alloy aged for 10 min, the results obtained by X-ray line profile analysis and differential scanning calorimetry confirm that neither the dislocation density nor the precipitate structure change during subsequent ECAP processing after 1 pass. This is consistent with reports for other alloys [32,33] showing the strength of these quenched Al–Zn–Mg alloys may be significantly enhanced by pressing through only 1 or 2 passes of ECAP without continuing to larger numbers of passes. This latter result is important in any practical utilization of these alloys because processing through a single pass has been incorporated into several potential industrial processing procedures such as continuous confined strip shearing [34], continuous frictional angular extrusion [35] and equal-channel angular rolling [36].

It is important to note also that the same strengthening is not attained if a strain of ~1 is imposed using alternative processing procedures such as cold-rolling. This is because in ECAP the strain is imposed under a hydrostatic pressure in a single pass and, as shown in other experiments [37], optimum strength and microstructures are achieved most readily when a high strain is imposed in a single processing operation rather than by introducing the same cumulative strain through several small strain increments.

6. Summary and conclusions

1. Experiments were conducted on two supersaturated Al–Zn–Mg alloys in order to optimize the processing parameters for ECAP at room temperature for age-hardenable alloys.
2. The results reveal a successful strategy for processing these materials by ECAP at room temperature. It is shown that pressing may be conducted successfully, without the formation of catastrophic cracking or segmentation, if the processing by ECAP is performed immediately after quenching or at least within a very short pre-aging lifetime (typically no more than ~10 min).
3. The results demonstrate also that high strength is achieved in these alloys even when ECAP is performed through only a single pass. This suggests a potential for incorporating the ECAP processing of age-hardenable alloys in several different industrial processing procedures.

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