

Microstructure, hardness evolution and thermal stability of binary Al–7Mg alloy processed by ECAP with intermediate annealing

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Abstract: A binary Al–7Mg alloy was processed by equal channel angular pressing (ECAP) at room temperature via route Bc, combined with intermediate annealing. After 6 passes, a high hardness of 218 HV is achieved. Transmission electron microscopy (TEM) observations demonstrate that ECAP leads to a significant grain refinement and ultrafine grains down to 100–200 nm are developed after 5 or 6 passes. X-ray diffraction (XRD) analysis indicates that the major part of Mg atoms are in solid solution in the deformed material, and the possible strengthening effect of Mg solute atom clusters or precipitates is neglected. The high hardness of the 6 pass-treated materials comes mainly from grain boundary strengthening, which contributes about 41% to the total strength, while dislocations and Mg solid solution contribute about 24% and 35% to the remaining strength, respectively. Also, the thermal stability of this severely deformed material was investigated by hardness measurements. The material is relatively stable when annealed at a temperature lower than 250 °C, while annealing at 300 °C leads to a rapid softening of the material.

Key words: Al–Mg alloys; equal channel angular pressing (ECAP); as-deformed microstructure; hardness evolution; strengthening; thermal stability

1 Introduction

Ultrafine grained (UFG) metals and alloys produced by severe plastic deformation (SPD) have generally superior mechanical properties resulting from significant grain refinement together with dislocation strengthening [1]. The equal channel angular pressing (ECAP) technique is especially attractive for producing bulk UFG metals and alloys in comparison with other SPD techniques, as it allows a minimal change of external sample dimensions [1,2].

Al–Mg alloys are among the most widely used and most extensively studied solid solution hardening Al alloys. They are considered especially attractive for production of high strength UFG materials through ECAP [3] because Mg has very high solubility in Al. The role of Mg in obtaining UFG structures in Al matrix by RT-ECAP has been clearly demonstrated [3,4]. The mechanical properties of Al–Mg alloys ($\leq 3\%$ Mg) subjected to RT-ECAP has been investigated in detail,

based mainly on the hardness measurements and compression tests [4–6]. In addition, UFG materials processed by ECAP appear to retain high levels of internal strain and most of the grain boundaries are in non-equilibrium configurations [7], which necessitates further evaluation of their thermal stability [2]. Generally, an increase in Mg content decreases the stacking fault energy, normally resulting in low recovery rates and therefore smaller grain sizes after the SPD process [4]. With the Mg content increasing up to 4%, however, the material presents challenges with regard to workability at ambient temperature with cracking and failure occurring during ECAP [8]. In contrast, by increasing the pressing temperature, the plastic workability of Al–Mg alloys is improved [9,10]. However, hot ECAP leads to a tremendous expense of strength [11]. To the best of our knowledge, yet little effort has been devoted to binary Al–Mg alloys containing above 4% Mg processed by RT-ECAP.

To further exploit the strengthening potential of aluminium alloys containing a high content of Mg, a

combination of RT-ECAP with intermediate annealing, similar to that introduced by FURUKAWA et al [12], has been applied in the present work. With this approach, 6 passes of RT-ECAP have been achieved successfully in the Al–7Mg alloy. The corresponding mechanical properties and the annealing response of the material were investigated and briefly reported.

2 Experimental

Materials used in the present work were taken from cast Al–7Mg ingots supplied by hydro aluminium having chemical composition (mass fraction, %): Mg 7.0, Fe 0.05, Si 0.06, Al in balance. ECAP was performed with route Bc, using a 100 mm × 19.5 mm × 19.5 mm bar in a 90° die, which leads to an imposed strain of about 1.0 per pass. All ECAP experiments were carried out at room temperature. However, the ECAP process at room temperature was interrupted after 3 passes, since severe cracks extended to the center thickness of the samples. By applying intermediate annealing at 300 °C for 120 s on samples between selected pressings, the Al–7Mg alloy was pressed successfully to 5 and 6 passes, respectively. Samples from the uniformly deformed central region of the ECAP billets were chosen for microstructure observation, hardness measurement and post annealing experiment. The intermediately annealed samples were labeled as 5P and 6P (5P=2 passes + annealing +3 passes; 6P=2 passes + annealing + 2 passes + annealing + 2 passes). Intermediate and post-ECAP annealing treatments were performed using two different furnaces depending on the applied temperature range: air circulated furnace (≤ 275 °C), and molten salt baths (~ 300 °C). The temperature error was controlled within ± 3 °C in all cases. Immediately after annealing, samples were water-quenched to room temperature. The TEM foils were prepared by twin-jet electro polishing in a solution of 33% nitric acid–methanol at -30 °C. TEM analysis was performed with a Philips CM30 operating at 150 kV and with a JEOL* 2010 at 200 kV. Samples for X-ray diffraction analysis (XRD) were prepared by standard metallographic techniques followed by electro polishing with a solution of 80% C₂H₅OH + 20% HClO₄ at 20 V for 15 to 25 s at -30 °C. XRD was undertaken with a Siemens D5000 at 40 kV, Cu K α radiation, with 0.02 step size and 20 s/step. Vickers microhardness measurements were performed under a 4.9 N load applied for 15 s. At least six separating measurements were conducted for each condition.

3 Results and discussion

3.1 As-deformed microstructure

TEM observations were carried out in order to investigate the microstructures of the 5P and 6P treated

samples and the corresponding micrographs are shown in Fig. 1. The adjoining selected area diffraction patterns (SAD) in Fig. 1(a) indicate that a submicron sized polycrystalline structure is developed. No Mg-containing precipitates, such as Al₃Mg₂ phase, were detected by diffraction patterns. However, this might be due to their small volume fraction and extremely fine size [13]. It is therefore believed that the effect from Al₃Mg₂ precipitation, if any, should be weak and negligible.

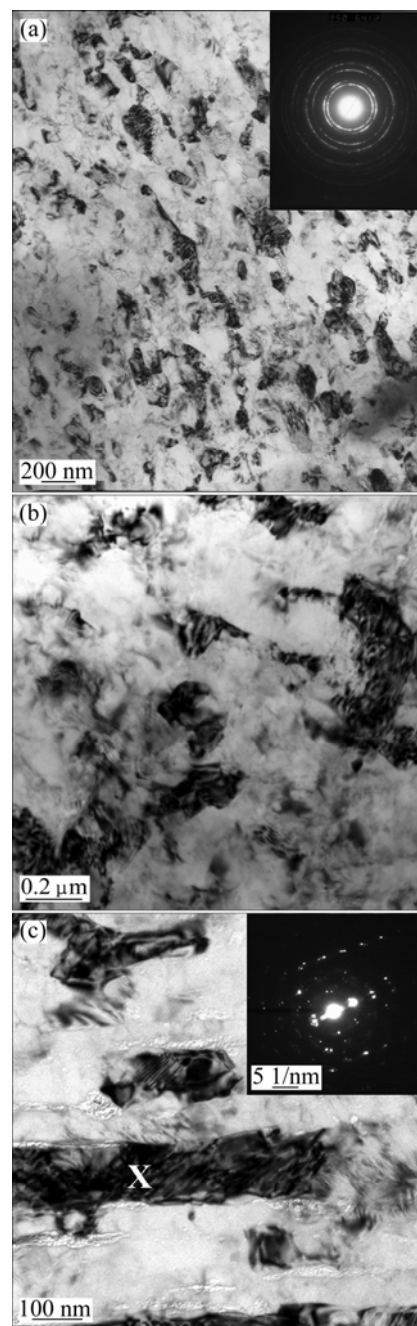


Fig. 1 Typical TEM images of 5P (a) and 6P (b) treated samples and higher magnification micrograph of elongated UFG grains (c) (The insertion in (a) is the corresponding diffraction pattern taken from the same area as in micrograph (a); the insertion in (c) is the corresponding diffraction pattern taken from the elongated grain marked out in the micrograph)

Also, the 6P treated sample has similar microstructures to that of the 5P treated sample, but with an obvious further grain refinement (Fig. 1(b)). Most ultrafine grains in both samples appear elongated, with 50–100 nm in width and 200–500 nm in length. Figure 1(c) shows a higher magnification micrograph of the elongated grains in the 5P treated sample, and shows apparent extinction contours and dislocation tangles inside the grains at the grain boundaries. The inserted SAD pattern in Fig. 1(c) reveals some misorientations inside the elongated grains, indicating the formation of sub-boundaries.

Note that grain boundaries (GBs) frequently appear curved and poorly defined, or show a strong spread in terms of thickness extinction contours (Fig. 1), indicating a high level of internal stresses and elastic lattice distortions due to the presence of a high density of dislocations in boundary regions. Figure 1 also reveals the presence of extensive dislocation tangles in grain interiors and at grain boundaries. Apparently, the elongated ultra-fine grains are in a non-equilibrium state, as evidenced by the fuzzy GBs [14,15].

3.2 Hardness evolution during processing

The evolutions of Vickers hardness (HV) of the 5P and 6P treated samples during the whole ECAP + intermediate annealing procedure are illustrated in Figs. 2(a) and (b), respectively. Obviously, the hardness increases significantly after 2 passes, i.e., from ~77 HV in the as-cast state to ~165 HV in the 2 passes treated sample. The large increase in hardness after 2 passes probably originates from a significant substructure refinement and the introduction of a high density of dislocations. After intermediate annealing, the hardness decreases significantly, though still slightly higher than that in the as-received state. With another 2 or 3 ECAP passes, the hardness increases again, to ~180 HV and ~205 HV in the 4 and 5 passes treated sample, respectively. With another round of intermediate annealing and another 2 ECAP passes performed after 4 passes, a peak hardness of ~218 HV is achieved in the 6P treated sample. It is here interesting to note that a HPT-processed Al–4.1Mg alloy obtained ~191 HV [16]. Hence, the high hardness in the present Al–7Mg alloy is probably strongly affected by the enhanced Mg concentration, facilitating large strain hardening during ECAP.

3.3 Strengthening mechanisms

Undoubtedly, the high hardness obtained in the present material is substantially beyond what can be achieved in conventionally-deformed bulk Al–Mg alloys, as well for ECAP treated Al–Mg alloys with lower Mg concentrations [4,17]. In the present study, the 6P treated sample gives the highest hardness and accordingly,

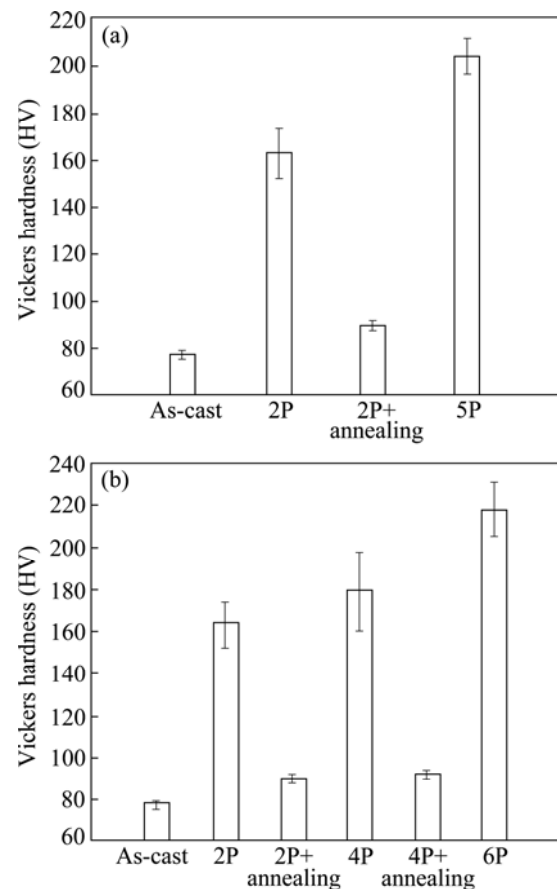


Fig. 2 Evolution of Vickers hardness of 5P (a) and 6P (b) treated samples with increasing passes and intermediate annealing

represents an interesting process scheme and a preferred template for evaluating roles of various potential strengthening factors. However, careful examinations in high-resolution TEM, indicate that no potential contribution to the strengthening seems to occur from precipitated Mg-containing intermetallics.

In the present alloy, where grains are refined into UFG structures with an average size down to 100–200 nm, grain boundary strengthening is expected to have a prominent influence, as indicated by the Hall–Petch relationship [18]:

$$\Delta H_{VGB} = k_H d^{-1/2} \quad (1)$$

where ΔH_{VGB} is the contribution from grain boundaries to the overall strength; d is the grain size; k_H is a constant of HV $\sim 35 \mu\text{m}^{-1/2}$. The average grain size d of the UFG grains in the present material was determined by measuring the length and width of about 100 individual grains from six TEM micrographs. Then the equivalent diameter was calculated to be ~ 150 nm. With an average grain size of 150 nm, the strength contribution from grain boundaries is ~ 90 HV. However, it should be noted that the actual grain boundary strengthening contribution

is probably somewhat lower than that predicted from this value, due to the existence of some coarser grains, which might not be easily detected by TEM.

Magnesium is known to strengthen Al through solid solution hardening, reducing dislocation mobility by atom-dislocation interactions [3,18]. The atom-dislocation interactions consist of both in the form of dislocation “pinning” in lattice positions occupied by solute atoms, and by the diffusion of solute atoms to dislocations, retarding their mobility [18]. According to results of RYEN et al [19] and HUSKINS et al [18], the solute strengthening increment, σ_{ss} , due to a concentration C of solid solution atoms can be expressed as

$$\Delta\sigma_{ss}=HC^n \quad (2)$$

where H and n are constants [18]. At the yield point $\varepsilon_{ys}=0.002$, commercial Al alloys take values $H=13.8$ (MPa/%) and $n=1.14$ under a strain rate of $6\times 10^{-3} \text{ s}^{-1}$ [19]. All the parameters in Ref. [19] are determined from tensile experiments conducted on coarse grained Al-based alloys and the strain rate ($6\times 10^{-3} \text{ s}^{-1}$) in Ref. [19] is about ten times that used in the present work ($5\times 10^{-4} \text{ s}^{-1}$). Hence, one may speculate that this would represent an uncertainty since H and n are sensitive to strain rate. However, according to Ref. [19], the Mg concentration may be disregarded, within the strain rate range of $1\times 10^{-3} \text{ s}^{-1}$ – $1\times 10^{-4} \text{ s}^{-1}$, and the yield stress keeps almost constant.

Before calculating the strengthening contribution from Mg in solid solution, one has to mention that there might be some Mg segregation, according to XRD analysis. The latter indicates a decrease in the lattice parameter from $(4.0860\pm 0.0002) \text{ \AA}$ in the as-homogenized state to $(4.0824\pm 0.0004) \text{ \AA}$ in the as-deformed state. Such a feature could be the result of deformation-induced segregation or precipitation at grain boundaries [20]. However, Mg-containing precipitates were neither observed by TEM nor detected by XRD. Moreover, it is very difficult to calculate the strengthening contribution from SPD-induced Mg segregation. For convenience, one assumed that the 7% of Mg atoms are kept in solid solution. The increment in hardness due to solid solution is therefore estimated to be $\Delta H_{vss} \sim 51 \text{ HV}$ (assuming that $\sigma_b=2.5V_H$, where V_H is $\sim 218 \text{ HV}$ giving a yield strength σ_b of $\sim 538 \text{ MPa}$). Nevertheless, the assumption that 7.0% of Mg is solved may slightly overestimate the contribution from solid solution.

An increase in dislocation density is expected to be the main mechanism responsible for the rapid increase in strength of severely deformed Al–Mg alloys with increasing Mg content [21], as the relationship between strength increment and the dislocation density (ρ) for

plastically deformed metals can be characterized by the Taylor equation [22]:

$$\Delta\sigma_D = M\alpha_1 Gb\sqrt{\rho} \quad (3)$$

where $\alpha_1=0.3$ is a constant, $G=27 \text{ GPa}$ is the shear modulus, $b=2.86\times 10^{-10} \text{ m}$ is the length of the Burgers vector for Al and $M=3$ is the Taylor factor [22]. To the best of the present authors' knowledge, applicable dislocation densities for ECAP-processed Al–Mg alloys are not available in the literature. Also, it is quite difficult to assess reliable dislocation densities for the present material due to the heterogeneous microstructure. However, since all contributions to the present total flow stress are calculated at the onset of yield (0.2% strain) and are referred to as increments in yield strength, ΔH_{vD} may be estimated to be $\sim 76 \text{ HV}$. This is obtained by employing a relationship for the total hardness $H_{vb}=\Delta H_{vGB}+\Delta H_{vD}+\Delta H_{vss}$, where $\Delta H_{vGB}=90 \text{ HV}$, $\Delta H_{vss}=51 \text{ HV}$, $H_{vb}=218 \text{ HV}$. As the possible Mg segregation may cause some influence on the strengthening mechanisms, the contribution from the dislocation strengthening might be somewhat overestimated. Segregated Mg atoms might form clusters at grain boundaries and provide an additional strengthening effect as discussed earlier and as proposed in Ref. [23].

Nevertheless, based on the above analyses, the high hardness of the 6 passes material comes mainly from the enhanced grain boundary strengthening σ_{GB} , i.e., carrying $\sim 41\%$ to the total strength, while the dislocation density and Mg solid solution contribute $\sim 24\%$ and $\sim 35\%$ to the remaining strength, respectively, if the additional strengthening effect of clusters formed by segregated Mg atoms is neglected.

3.4 Hardness evolution upon annealing

To evaluate the thermal stability of the present Al–7Mg alloy, the evolution of hardness of the 5P and 6P treated samples upon annealing at different temperatures for 30 min, and for various time at $\sim 300 \text{ }^\circ\text{C}$, was plotted in Figs. 3(a) and (b), respectively. In Fig. 3(b), the data for the 3P treated sample are incorporated for comparison. After annealing at $\sim 200 \text{ }^\circ\text{C}$ for 30 min, a significant portion of the enhanced hardness from ECAP is still retained, e.g., decreasing from $>200 \text{ HV}$ to $\sim 180 \text{ HV}$ (see Fig. 3(a)). With the temperature increasing to $\sim 250 \text{ }^\circ\text{C}$ and further to $275 \text{ }^\circ\text{C}$, the hardness decreases gradually to $\sim 150 \text{ HV}$, most likely due to a more pronounced dislocation recovery process. At $300 \text{ }^\circ\text{C}$, the decrease in hardness becomes sharp and completes more or less within 90 s (Fig. 3(b)), hence indicative of the occurrence and completion of recrystallization. The hardness evolution of the 5P treated sample at $\sim 300 \text{ }^\circ\text{C}$ is quite similar to that of the 3 passes treated sample, i.e. changing drastically above $\sim 300 \text{ }^\circ\text{C}$. Therefore, $\sim 300 \text{ }^\circ\text{C}$

seems to be more or less than the critical temperature for nucleation of recrystallization. However, further investigations on the recrystallization behaviour are still ongoing.

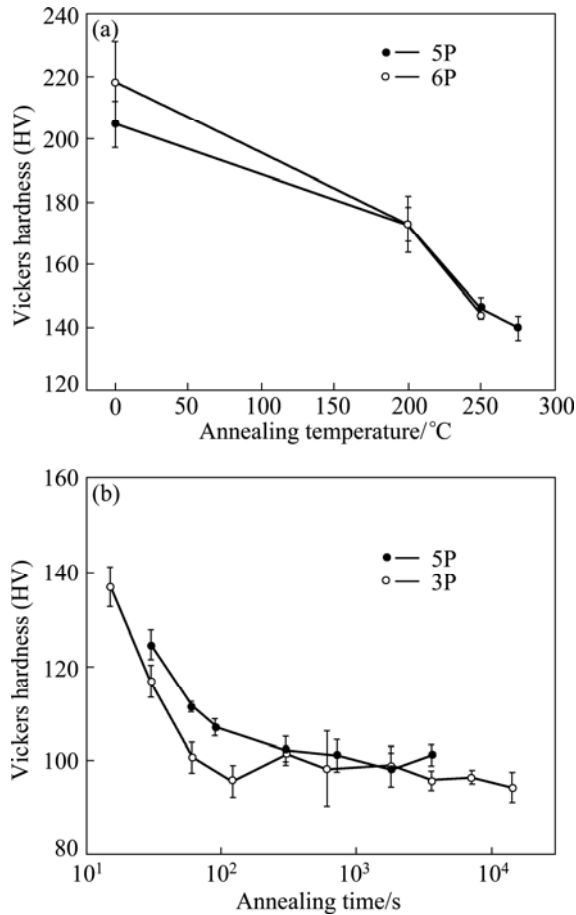


Fig. 3 Change of Vickers hardness with increasing annealing temperature for 30 min (a) and isothermal annealing at 300 °C with increasing time (b) (In Fig. 3(b) the data for the 3 passes treated sample are incorporated for comparison with the 5P treated sample)

4 Conclusions

1) A binary Al–7Mg alloy with enhanced hardness was processed at room temperature by ECAP combined with intermediate annealing. After 5 or 6 passes, ultrafine grains down to 100–200 nm were developed. After 6 passes, a hardness of ~218 HV was achieved.

2) The major part of Mg atoms is in solid solution in the as-deformed material, and hence the possible strengthening effect of Mg solute atom clusters or precipitates is neglected. The high hardness of the 6 passes treated material comes mainly from grain boundary strengthening, which contributes ~41% to the total strength, while dislocation density and Mg solid solution contributes ~24% and ~35% to the remaining strength, respectively.

3) The thermal stability of this severely deformed

alloy was investigated by hardness measurement. The material is relatively stable when annealed at temperatures lower than 200 °C, while annealing at 300 °C leads to rapid softening. The temperature of 300 °C seems to be the critical temperature for nucleation of recrystallization.

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等通道挤压变形结合中间退火制备 Al–7Mg 合金的组织、硬度演变及热稳定性

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摘 要: 在室温条件下采用 Bc 模式等通道挤压变形 (ECAP) 结合中间退火方法制备二元 Al–7Mg 合金。经 6 道次 ECAP 变形后, 该材料硬度高达 HV218。透射电镜 (TEM) 表征揭示该 Al–7Mg 合金的晶粒得到明显细化。经 5 或 6 道次变形后得到的超细晶组织平均尺寸为 100~200 nm。X 射线衍射分析表明 Mg 主要以固溶原子的形式存在。因此, 由于 Mg 偏析或析出相造成的可能强化影响均忽略不计。经 6 道次 ECAP 变形后的 Al–7Mg 合金的高硬度主要来自于晶界强化, 大约占总强度的 41%, 其次, 位错强化及 Mg 原子的固溶强化分别约占总强度的 24% 和 35%。此外, 通过测量硬度变化研究了该 Al–7Mg 合金的热稳定性。结果表明在 250 °C 退火处理时该材料相对稳定, 然而当温度升至 300 °C 时, 该 Al–7Mg 合金的软化趋势明显加快。

关键词: 铝–镁合金; 等通道挤压变形(ECAP); 变形组织; 硬度演化; 强化; 热稳定性

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