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Producing high strength aluminum alloy by combination of equal channel angular pressing and bake hardening

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ABSTRACT

A combination of severe plastic deformation by equal channel angular pressing (ECAP) and bake hardening (BH) was used to produce high strength ultrafine-grained AA6061 aluminum alloy. 2, 4 and 8 passes of ECAP were performed, and the bake hardenability of samples was tested by 6% pre-straining followed by baking at 200 °C for 20 min. The microstructures obtained for various passes of ECAP were characterized by XRD, EBSD, and TEM techniques. The microstructures were refined from an average grain size of 20 μm to 212 nm after 8 passes of ECAP. Maximum bake hardenability of 110 MPa, and final yield stress of 330 MPa were obtained in the specimens processed by 8 passes of ECAP.

1. Introduction

The usage of bake hardened (BH) material is an effective way to reduce the weight of vehicles and obtain higher safety in transportation industry [1–4]. The use of controlled aging during paint baking of deformed materials is known as the bake hardening (BH) technique. During the industrial BH process, a low strength material is required before press forming, and a high strength material is obtained after the paint baking process [5,6]. Among aluminum alloys, Al–Mg–Si alloys are widely used to manufacture body structures of cars, because these alloys attain higher strength after paint baking [7–9].

In general, BH takes advantage of strain aging when the solute atoms segregate to the dislocations and lock them. The procedure to determine the bake hardenability of materials is as follows: (a) the specimen is pre-strained between 2 to 8% at the room temperature, (b) aging is carried out at 170–200 °C for about 20 to 30 min, and (c) the aged specimens are tensile tested at the room temperature. These three steps simulate the forming, panting and baking the paint in the real industrial process of car bodies.

Another effective way to improve the mechanical properties of metallic alloys is by producing ultrafine-grained (UFG) or nanograined (NG) microstructures [10]. Severe plastic deformation (SPD) is a practical approach to attain UFG or NG microstructures. SPD techniques, such as equal channel angular pressing (ECAP), are widely used to produce NG aluminum alloys [11,12].

A combination of a grain refinement process and BH may result in significant increase in the strength of metallic alloys [4,13]. There are only a few works about the BH behavior of aluminum alloys [13], and there is no work concerning the effect of UFG or NG microstructures produced by SPD on the BH behavior of AA6061 aluminum alloy. The main goal of this study was to investigate the effect of UFG structures on the BH behavior and mechanical properties of baked AA6061 aluminum alloy. For this propose, UFG samples of AA6061 were produced by different passes of ECAP process, and then, these samples were subjected to BH. The BH behavior and mechanical properties of UFG samples were compared with those of the coarse-grained (CG) samples.

2. Experimental procedure

In this work, AA6061 aluminum alloy was used for experiments. Before the SPD processing, the initial bars were heated to 550 °C for 5 h, followed by water quenching. Afterwards, the samples were cut to become 10 mm in diameter and 80 mm in length, which are required dimensions for ECAP process to attain UFG samples. The ECAP was conducted using a die made of SKD61 steel grade with an angle of 90° between the channels and a curvature angle of 0°. Samples were coated with molybdenum disulfide (MoS_2) lubricant during the ECAP process [12].

After different passes of ECAP, the grain size was characterized by XRD and EBSD techniques. Williamson–Hall equation was used to calculate the size of the produced grains [14]. The grain size of the alloy was also checked by the EBSD technique; EBSD was used to study the microstructure in areas of approximately 600 μm^2 [15].

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To study the BH behavior of AA6061 aluminum alloy, tensile specimens were cut from the ECAP-processed and un-deformed samples according to ASTM E8. To simulate the BH process, the samples were pre-strained for 6%, followed by baking at 200 °C for 20 min. Finally, the baked samples were tensile tested at the room temperature at a strain rate of $10^{-3}\,s^{-1}$. The BH amount was determined from the difference between the flow stress after pre-straining and the yield stress after baking.

3. Results and discussion

3.1. Microstructures of ECAP-processed samples before BH

The microstructures of samples before the ECAP process had an average grain size of about 20 μm. The microstructures and histograms of the samples processed by ECAP, characterized by EBSD, are shown in Fig. 1(a–c). The results show that the initial CG samples were significantly refined to produce UFG microstructures after the ECAP process. After only two passes of ECAP, there is a significant decrease in the average grain size (~755 nm) of the sample. With an increase in the number of ECAP passes, the grains were considerably finer, for example by applying 8 passed of ECAP, the initial average grain size of 20 μm was reduced to about 212 nm.

The grain size was also measured by XRD method. According to the XRD patterns, the peak of (2 0 0) was dominant compared with other peaks [16]. Also, the intensity of as-received samples is lower than that of ECAP-processed samples. As shown in Fig. 1(d), by increasing the number of ECAP passes, the intensity increases, there for more grain-refinement is achieved.

The average grain size of as-received and ECAP-processed samples determined by XRD and EBSD are summarized in Table 1(a).

3.2. BH behavior of samples

Effect of BH treatment and ECAP on mechanical properties: The BH curves obtained for samples processed by different passes of ECAP are shown in Fig. 2. These samples were pre-strained by 6%, and then baked at 200 °C for 20 min. Fig. 2 shows that the amount of BH of ECAP-processed samples is higher than that of as-received one. This indicates by applying 2 and 4 passes of ECAP followed by BH, there is about 50 and 85 MPa increase in yield stress, respectively. The maximum bake hardenability (110 MPa) and final yield stress (330 MPa) were obtained for the specimens subjected to 8 passes of ECAP and baked at 200 °C. As for the as-received sample, the measured BH and yield stress were respectively about 20 MPa and 100 MPa. These results show that baking after ECAP is an effective approach to improve the strength of aluminum alloys; nevertheless, its effect depends on the number of ECAP passes. The increase in the strength is also due to the formation of fine and UFG microstructures in the samples. It is well known that with grain refinement, the amount of grain boundaries increases. As the baking treatment is a diffusion control process, the diffusion of soluble atoms increases by increasing the volume fraction of grain boundaries [4]. In addition, the precipitation kinetics is increased due to the higher diffusivity in the UFG structures. Therefore, in the case of baked AA6061 with UFG microstructure, the effect of dislocation locking is enhanced and the BH is increased significantly. These subjects are discussed in details by Alihosseini et al. [4] and Dehghani [13].Obviously, a decrease in the grain size from 20 μm (CG state) to 200 nm (UFG state) led to a considerable increase in the bake hardenability and the final strength of the materials; this is clearly evident from the stress–strain curves presented in Fig. 2.

In Fig. 2, the return of yield point after baking indicates that aging is occurred. This is attributed to the diffusion of solute atoms to the dislocations generated during pre-straining and in turn to the formation of solute atmosphere around dislocations, resembling the Cottrell atmosphere. As dislocations are locked in this way, the higher stress (i.e. yield stress) is required to un-pin them.

Effect of bake hardening process on microstructures: TEM micrographs of the samples processed by 4 and 8 passes of ECAP and baked at 200 °C for 20 min are shown in Fig. 3. A low density of very fine precipitates (~25 nm) exists in the micrograph of samples processed by 4 passes of ECAP, Fig. 3(a). The precipitates

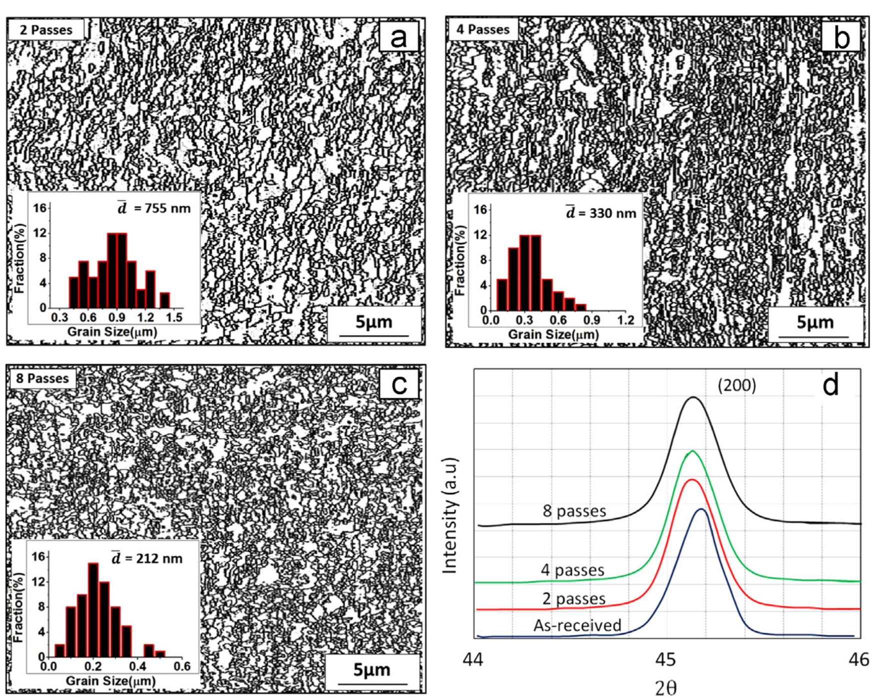

Fig. 1. Microstructures of the samples taken by EBSD after: (a) 2 passes, (b) 4 passes, and (c) 8 passes of ECAP; (d) XRD pattern of the ECAP-processed 6061 samples for the (2 0 0) peak.

Table 1
(a) Grain size of as-received and ECAP-processed 6061 Al samples determined by XRD and EBSD.

Number of ECAP passes		As-received	2	4	8
Grain size	XRD	20 μm	750 nm	330 nm	200 nm
	EBSD	20 μm	755 nm	330 nm	212 nm

(b) Amount of BH gained by solute strengthening ($\Delta\sigma_{Cottrell}$) and precipitation hardening ($\Delta\sigma_{ppt}$).

Number of ECAP passes	λ (nm)	$\Delta\sigma_{Cottrell}$ (MPa)	$\Delta\sigma_{ppt}$ (MPa)	$\Delta\sigma_{BH}$ (MPa)
4	200	51	34	85
8	80	24	86	110

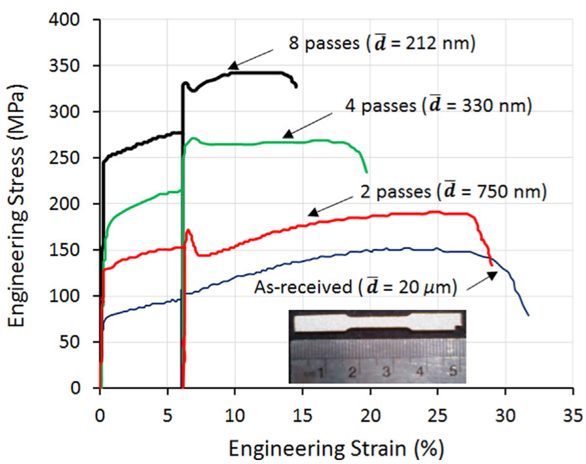

Fig. 2. The stress–strain curves of the ECAP-processed 6061 Al alloy after bake hardening (samples were pre-strained to 6 %, then baked at 200 °C for 20 min).

accumulate especially around the dislocations, and this indicates that the precipitates preferentially nucleate at the sites where dislocations are piled-up. Fig. 3(b) shows very high density of small particles in the micrograph of samples processed by 8 passes of ECAP. The EDX analyses indicate that the precipitates are mainly rich in Mg and Si, thus, they were identified to be Mg_2Si precipitates. The general accepted precipitation sequence during baking/aging is as follows [9,17]: super-saturated solid solution (SSSS) atomic clusters (spherical)→β″(needle)→β′(rod)→β. Because of the short time and low temperature of the baking, the main strengthening phase is β″. The nucleation of precipitates happens with individual solute clustering of Si and Mg [9,18,19]. The increased diffusivity and strong stress field induced by the significant amount of dislocation density during the ECAP processing can promote the kinetics of precipitation.

As reported by Kozeschnik et al. [20], the strengthening due to BH ($\Delta\sigma_{BH}$) involves two steps as follows:

$$\Delta\sigma_{BH} = \Delta\sigma_{Cottrell} + \Delta\sigma_{ppt}, \tag{1}$$

where $\Delta\sigma_{Cottrell}$ is known as the solute strengthening caused by the formation of Cottrell atmosphere around dislocations, and $\Delta\sigma_{ppt}$ is the precipitation hardening. When pre-strained specimens are aged at low temperatures, the solutes of Mg and Si diffuse to the dislocations and lock them, and thereby increase the strength through the formation of Cottrell atmosphere [20]. With respect to $\Delta\sigma_{ppt}$, as the aging/baking proceeds, the solute saturation is reached resulting in the formation of precipitates, i.e. precipitation hardening. Both $\Delta\sigma_{Cottrell}$ and $\Delta\sigma_{ppt}$ are responsible for age hardening. As the concentration of dissolved solute atoms reaches a saturation state, there is a transformation from the cluster hardening ($\Delta\sigma_{Cottrell}$) toward precipitation hardening ($\Delta\sigma_{ppt}$), where the nucleation of precipitates occurs. A high density of fine precipitates can occur in UFG structures resulting in the pinning of grain boundaries [4,13].This could be a major source of stability for the produced nanostructure of the material.

As was mentioned before, there are two main factors that affect BH, and to evaluate their effects separately, it is needed to measure some important features experimentally, such as size, shape, volume fraction and distribution of hardening precipitates. Orowan strengthening is one of the well-known strengthening mechanisms resulting from precipitation hardening [21]. The basic equation for the Orowan precipitation hardening stress is:

$$\Delta\sigma_{ppt} = \frac{Gb}{\lambda}, \tag{2}$$

where G is the shear modulus of the matrix material, b is the burgers vector or lattice parameter of the matrix material, and λ is the inter-particle spacing. To estimate the amount of precipitation hardening, if the Orowan equation gives results that are consistent with the experimental observations, we need to know λ. This parameter can be measured based on TEM micrograph results (Fig. 3). Inter-spacing of particles (λ) is the Mg_2Si spacing. According the TEM micrographs in Fig. 3, the ratio of Mg_2Si particle spacing in the case of 4 passes (λ_4) to that of 8 passes (λ_8) is $\lambda_4/\lambda_8 \approx 2.5$. Considering Eq. (2), we can find:

$$\frac{(\Delta\sigma_{ppt})_8}{(\Delta\sigma_{ppt})_4} = \frac{\lambda_4}{\lambda_8}, \tag{3}$$

where $(\Delta\sigma_{ppt})_4$ and $(\Delta\sigma_{ppt})_8$ are the precipitation hardening stresses of the samples that were ECAPed 4 and 8 passes, respectively. Substituting the amount of λ_4/λ_8 in Eq. (3), the value of $(\Delta\sigma_{ppt})_8/(\Delta\sigma_{ppt})_4$ will be also about 2.5. This means that the precipitation strength of 8 passes ECAP+BH of Al6061 samples can be about 2.5 times higher than that of 4 passes ECAP+BH. In another word, the formation of particle of Mg_2Si with small interparticle-spacing of λ can lead to significant increases in the properties of produced samples.

To calculate the effect of precipitation hardening in different passes of ECAP, we assumed that $G = 27.6$ GPa and $b = 0.23$ nm for Al6061 and the interspacing of particles for these passes are based on the TEM micrographs in Fig. 3; the calculated precipitation hardening stresses for these two passes are $(\Delta\sigma_{ppt})_4 = 34$ MPa and $(\Delta\sigma_{ppt})_8 = 86$ MPa.

Based on Eq. (1): $\Delta\sigma_{Cottrell} = \Delta\sigma_{BH} - \Delta\sigma_{ppt}$, where $\Delta\sigma_{BH}$ is the total BH and can be calculated based on the stress–strain curves shown in Fig. 2. In Table 1(b), the results for BH gained by solute strengthening ($\Delta\sigma_{Cottrell}$) and precipitation hardening ($\Delta\sigma_{ppt}$) are summarized. These results clearly shows that although solute strengthening decreases by increasing the passes of ECAP, precipitation hardening increases significantly which results in increase of total BH. To measure the stresses more precisely further research is needed that is being carried out currently and the results will be presented in our future publications.

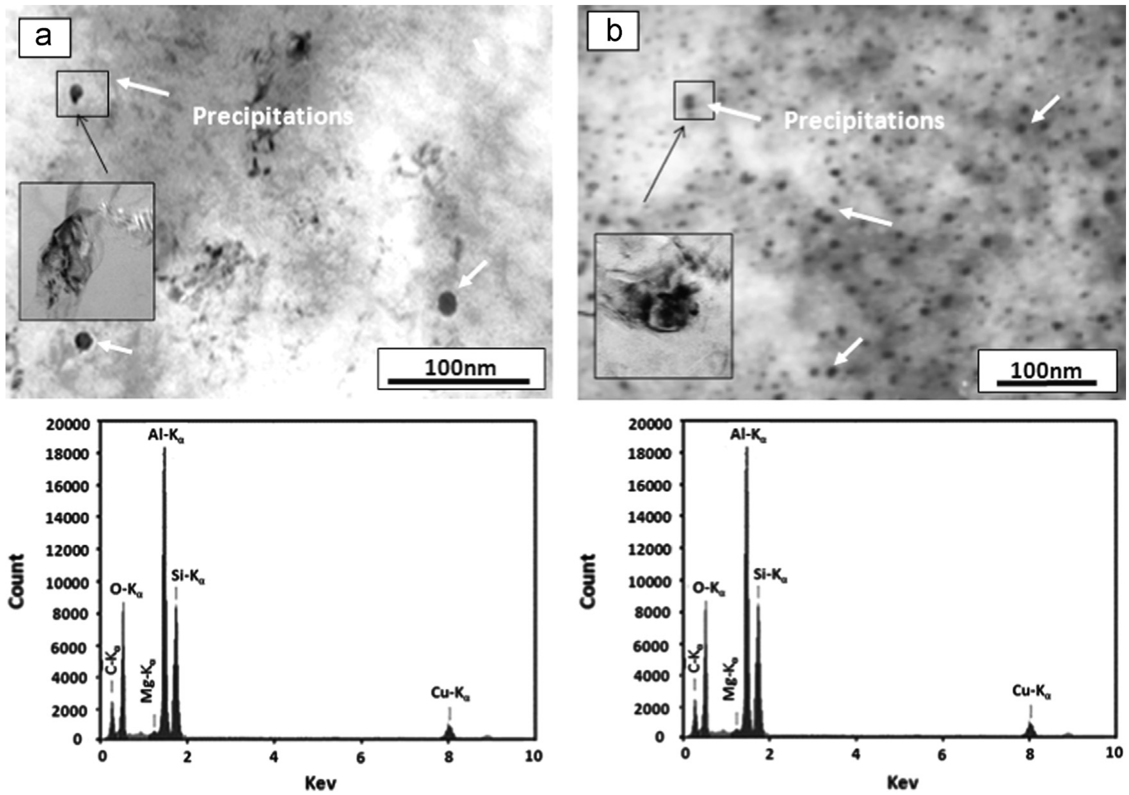

Fig. 3. TEM micrograph of ECAP-processed 6061 Al alloy after baking treatment at 200 °C for 20 min: (a) four ECAP passes, and (b) eight ECAP passes.

4. Conclusions

A combination of SPD by ECAP process and BH treatment was used to study the effect UFG structures on bake hardenability of AA6061 aluminum alloy. The results indicated that the combination of high density of dislocations as well as high volume fraction of grain boundaries in UFG structures will result in more precipitation during the BH process, which is a major factor in increasing the bake hardenability and strength of the material.

Acknowledgments

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