Activation energies for the grain growth of an AZ31 Mg alloy after equal channel angular pressing

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Equal Channel Angular Pressing (ECAP) is one of the methods proved to be very effective in fabricating relatively large bulk materials with ultra-fine grain sizes [1, 2]. In ECAP, the sample undergoes plastic deformation when it passes through the intersecting corner. By pressing the same sample repetitively through a die, very high strain can be accumulated on the sample without change in its cross sectional area. The ECAP process might be very effective in enhancing the workability and strength of Mg alloys due to grain refinement for their extensive usage in industries. Therefore, it is necessary to examine the effect of temperature on the stability of microstructure in the ECAPed Mg alloy following the static annealing for solid state forming such as forging, hot pressing, etc.

In the present work experimental research has been conducted on activation energies for grain growth of 1–4 passed magnesium alloy at different temperatures of static annealing after ECAP. ECAP was conducted for an extruded AZ31 alloy with an initial grain size of 48 μm using a die designed to give an approximate strain ε of $\sim$1. Repetitive pressing of the same sample was carried out with route B_c [1], in which each sample was rotated 90° around its longitudinal axis between the passages. In order to maximize grain refining effect, the first and second pressings were conducted at 593 K and the third and fourth pressings were conducted at lower temperatures, 523 and 473 K, respectively. The ECAPed samples were sliced and used to observe the microstructures on the cross sectional plane perpendicular to the pressing direction.

Fig. 1a–d show the grain size distribution of the (a) 1-passed, (b) 2-passed, (c) 3-passed, and (d) 4-passed AZ31 ECAPed alloys, respectively, obtained from the image analysis results. The average grain size has been largely decreased from $\sim$48 to $\sim$2.5 μm after 4 passes. Microstructures after ECAP are initially inhomogeneous and large grains are still present up to 3 passes. Fig. 1c and d clearly show that the ECAPed microstructure has become reasonably homogeneous and equiaxed after 3 pressings, suggesting that dynamic recrystallization during pressing or static recrystallization during heating to processing temperature prior to subsequent pressing occurred.

Stability of grain structure at elevated temperatures was investigated by annealing a series of 1–4 passes ECAPed samples for 30 min in a range of temperatures from 473 to 748 K. Fig. 2 shows a plot of grain size, d, against the absolute annealing temperature, T. For the 1, 2 and 3 passed samples, the grain growth occurred gradually with annealing temperatures. For the 4 passed sample, however, a relatively small grain growth is observed in the lower temperature regime (473–673 K), but marked grain growth is observed in the higher temperature regime (673–748 K). This result implies that the fine-gained microstructure produced by the current ECAP technique becomes unstable above $\sim$673 K (=0.74 T_m, where T_m is the absolute melting temperature). This temperature, therefore, can be considered to be the upper limit for solid state forming processes of the ECAPed AZ31 alloy. The upper limit temperature, however, would be lower than 673 K when dynamic grain growth that occurs during plastic forming is concerned.

In order to investigate the grain growth mechanisms during static annealing it is necessary to determine the activation energy for grain growth. Grain growth behavior in the investigated temperature range was assumed to follow the general equation for grain growth;

$$d^{\mathrm{n}} - d_{\mathrm{o}}^{\mathrm{n}} = K_{\mathrm{o}} t \exp(-Q_{\mathrm{g}}/RT) \tag{1}$$

where d is the grain size at a given annealing time, d_o is the initial grain size, K_o is a constant, t is the annealing time, Q_g is the activation energy for grain growth and RT has its usual meaning, and n is 2 when a parabolic relationship for grain growth is assumed. Based on the above equation, Q_g can be obtained by plotting $(d^2 - d_o^2)$ against $(1/T)$ in a semi-logarithmic scale at the same annealing time. Such a plot is made for the 1–4 passed samples annealed at different temperatures in Fig. 2, in which initial grain size d_o is 8.1, 6.3, 4.3, and 2.5 μm for 1, 2, 3, and 4 passed samples, respectively.

From Fig. 3, the measured activation energy values Q_g for 1, 2, and 3 passed samples were 34.4, 33.2, and 33.5 kJ/mol, respectively, in the entire testing temperature range, suggesting that the values are almost same with the average value of 33.7 kJ/mol. The activation energy is much lower than that for lattice self-diffusion (Q_L) in pure Mg (=135 kJ/mol) [3] or that for grain boundary diffusion (Q_{gb}) in pure Mg (=92 kJ /mol) [4]. The average value corresponds to $\sim$0.25 Q_L. However, the 4 passed sample has two different values in the testing temperature range. One is 31.3 kJ/mol (=0.23 Q_L) in the range 473–673 K, which is of similar value to that of 1, 2 and 3 passed. And, the other is 109.2 kJ/mol (=0.81Q_L) in the range 673–773 K, which is lower

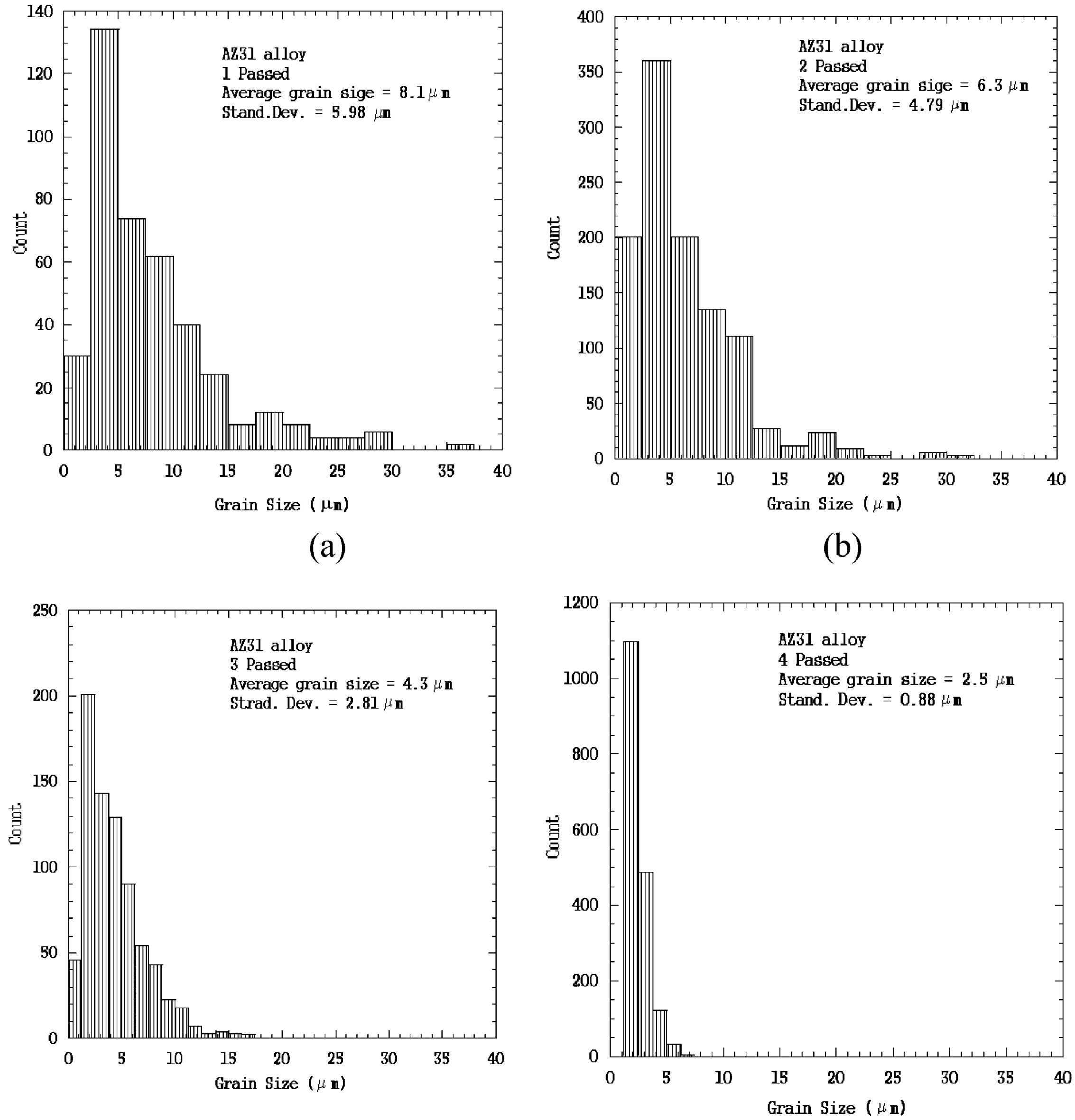

Figure 1 Grain size distribution of (a) 1-passed, (b) 2- passed, (c) 3-passed, and (d) 4-passed ECAPed AZ31 alloys.

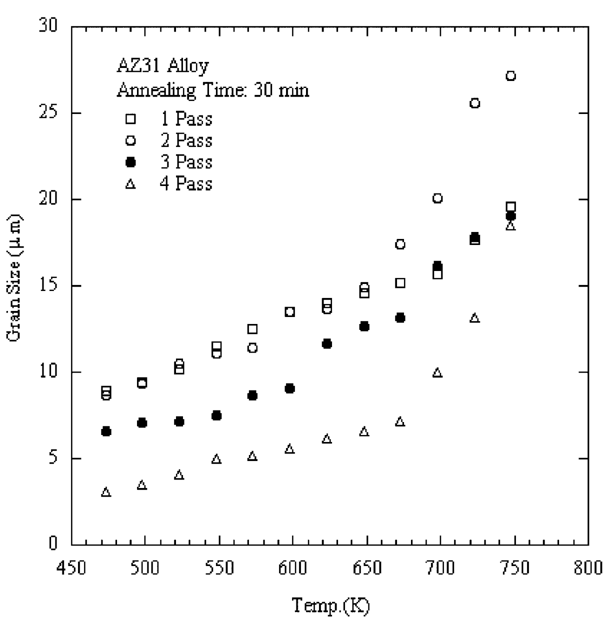

Figure 2 Grain size against annealing temperature for annealing time of 30 min.

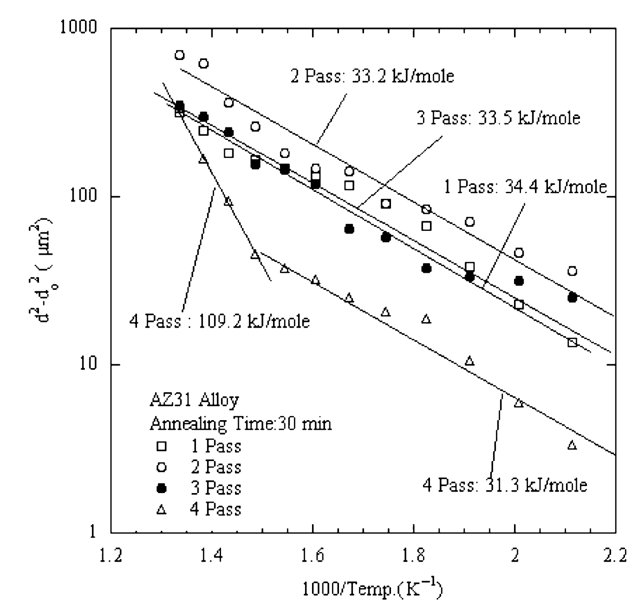

Figure 3 Plot of log $(d^2 - d_o^2)$ against $1/T$ for the estimation of the activation energy for grain growth of the ECAPed AZ31 during annealing.

TABLE I Activation energies for grain growth of ECAPed AZ31 alloys

Samples	Q_g (kJ/mol)
1 pass	34.4 (473–748 K)
2 pass	33.2 (473–748 K)
3 pass	33.5 (473–748 K)
4 pass	31.3 (473–673 K), 109.2 (673–748 K)

than that for lattice self-diffusion (Q_L) in pure Mg [3] but higher than that for grain boundary diffusion (Q_{gb}) in pure Mg [4]. The measured activation energies were summarized in Table I.

There have been several researches on activation energy for the grain growth in ECAPed alloys [5–7]. The ECAPed alloys exhibit a similar trend. The activation energy for static grain growth in the ECAPed Al-3%Mg alloy with unrecrystallized grains measured in the low temperature range, Q_g, is $\sim$0.21 Q_L and that with the recrystallized grains measured in the high temperature range, Q_g, is $\sim 0.63 Q_L$ [5]. It is also consistent with the report on the same alloy that the activation energy with unrecrystallized grains, Q_g, is $\sim 0.18 Q_L$ and that with the recrystallized grains, Q_g, is $\sim 0.63 Q_L$ [6]. The activation energy for static grain growth in the ECAPed low carbon steel with unrecrystallized grains in the low temperature range, Q_g, is $\sim 0.38 Q_L$ (self-diffusion of Fe in α-iron) and that with the recrystallized grains in the high temperature range, Q_g, is $\sim 0.82 Q_L$ [7]. The abnormally low Q value measured in the low temperature range is attributed to the unrecrystallized microstructure with non-equilibrium grain boundaries containing a large number of extrinsic dislocations [5]. This is because the non-equilibrium grain boundaries induced by severe plastic straining may exhibit higher atomic mobility compared to the equilibrium grain boundaries.

From the similarity of the activation values of the these materials, it can be deduced that for the 4 passed ECAPed AZ31 alloy, the grain growth occurs in the range 473–673 K in unrecrystallized condition while it does occur in the range 673–773 K in recrystallized condition. And, for 1, 2, and 3 passed samples the growth occurs in unrecrystallized condition in the entire testing range. As mentioned previously, after 3 passes dynamic or static recrystallization occurred. Up to 3 passes, thus, the ECAPed samples are expected to have unrecrystallized microstructure with non-equilibrium grain boundaries containing a large number of extrinsic dislocations.

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