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Full Length Article

Grain growth in calibre rolled Mg–3Al–1Zn alloy and its effect on hardness

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Abstract

Calibre rolling of Mg–3Al–1Zn alloy at 300 °C led to development of fine grain size of 3 μm. Subsequent annealing, from 5 to 6000 minutes at 300–450 °C, revealed faster grain growth initially up to 60 minutes, which became sluggish on prolonged annealing. The time exponent for grain growth kinetics (n) suggests bi-linear behaviour with $n = 0.11$ and 0.008 over these time scales. The activation energy, based on various n values, varied over wide ranges that made the understanding of the mechanisms for grain growth difficult. This problem is explained by concurrent evolution of texture and grain boundary structure. The effect of grain growth on hardness at ambient temperature was found to follow the H–P type relationship.

Keywords: Annealing; AZ31 Mg-alloy; Calibre rolling; Twinning; Grain growth

1. Introduction

Magnesium is available abundantly in the nature and, owing to high specific strength, its alloys are becoming popular in aerospace, automobile, biomedical, architecture and electronic industries [1,2]. However, these alloys have limitation of poor mechanical processing because of their common hexagonal close packed (HCP) type crystal structure. There exist several processes that are used for plastic deformation. Rolling is one of them and it is used to form the sheets, bars and rods of various shapes. To improve the mechanical properties of rolled material many changes in the conventional rolling process were introduced [3–5], which could influence the degree of grain refinement. Calibre rolling (CR) process is one of the modified rolling processes. In this technique, a pair of grooved rolls is turned in the opposite directions and the work-piece gets reduced to the desired thickness or cross section over several roll passes by the compressive force. CR is used for mass production of metals with high precision and high strength [6].

The plastic deformation distorts the microstructure, and it causes the thermodynamically unstable state by introducing dislocations and other defects, which leads to increase in stored energy. This energy tends to revert to a stable state on subsequent annealing. This process of annealing results in three phenomena: recovery, recrystallization and grain growth [7]. Since the actual number of dislocations removed during recovery is quite small, the change in mechanical properties is limited. The driving force for recrystallization is the removal of a large number of dislocations, and so the associated stored energy removed is quite large. The reduction in grain boundary area, and thus the reduction in grain boundary free energy with increase in grain size, itself provides the driving force for grain growth in polycrystalline materials. This Mg alloy AZ31 exhibits deformation twins, which possess different interfacial energy from the grain boundaries and can also influence the boundary migration [8] in grain growth process. Various studies [9–11] addressed grain growth in this alloy system. However, no annealing study on grain growth after calibre rolling of this alloy is known. In the present paper, the effects of static annealing at different temperatures and over longer durations were investigated with the aim of investigating the kinetics and mechanisms for grain growth and examining its effect in terms of the Hall–Petch relationship.

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Nomenclature

T	Annealing temperatures
t	Isothermal annealing time
d	Grain size (after annealing)
d_0	Initial grain size
σ_y	Yield strength
k	Rate constant
n	Growth law index (time exponent)
V	Grain boundary migration rate
P	Driving force
M	Intrinsic mobility of a boundary in the pure material
t_0	Time at which grain growth would have started on completion of recrystallisation
Q	Activation energy for grain growth
σ_0 and k_y	The Hall–Petch constants

2. Experimental procedures

Mg-alloy AZ31B in the form of rolled plate of 50 mm thickness and having chemical composition (wt %): Mg–Al 3.0, Zn 1.0 and Mn 0.2 was used. The calibre rolling was carried out at a temperature of 300 °C to 12 × 12 mm^2 rods (~76% reduction) by a series of 5 roll passes in a rolling mill with reduction of ~16% per pass.

The samples of 10 × 5 × 5 mm^3 were cut from the calibre rolled rod. The annealing was carried out at temperatures (T) 300, 350, 400 and 450 °C in a muffle furnace with the accuracy of ±2 °C. The annealing time (t) used was varied to 5, 10, 20, 30, 60, 240, 600, 1440, 2880 and 6000 minutes within the accuracy of ±2 seconds. The specimens were quenched in water immediately after annealing to retain the microstructures attained at high temperature.

Metallographic specimen was prepared as per the ASTM procedure. The etching was carried out with acetic picral. The microstructure was examined by Olympus GX51 optical microscope (OM). Grain size measurement was done by mean linear intercept method and the error bars in mean linear intercept, called grain size (d) here, are reported at 95% confidence level. Electron back scattered diffraction (EBSD) was obtained by scanning electron microscope (SEM) Quanta 3D FEG with EBSD attachment, and TSL software was used for analysis. The misorientation angle and twins were measured in the area 250 × 250 μm^2 with a step size of 0.4 μm.

The microhardness measurement was carried out before and after annealing for all the specimens. The machine used was LM300AT, LECO make. The weight used was 15 gm and dwell time was kept at 15 seconds. Yield strength (σ_y) of the material is determined by the relation [12]:

$$\sigma_y = 3 * Hardness \quad (1)$$

where σ_y is in MPa and the hardness measured is in Hv.

3. Results

3.1. Initial microstructure

The microstructure of the as received plate, as shown in Fig. 1a, consists of equiaxed grains of average size 33.0 ± 3.0 μm, along with the presence of a large number of twins. CR led to grain refinement and the equiaxed microstructure developed is shown in Fig. 1b. This reveals large reductions in number of twins and grain size to 3.0 ± 0.5 μm, Fig. 1b. This grain size will be called as initial grain size d_0 for further study here. The grain refinement was observed in the CR condition due to dynamic recrystallization [3]. The hardness of the as-calibre rolled material was 84.3 Hv.

3.2. Microstructural evolution by annealing

For all the conditions of temperature and time employed for annealing the microstructures remained equiaxed, but with the increased grain sizes. The evolved grain size will be called as d. The grain size measured after annealing at the temperatures of 300 and 450 °C is plotted in Fig. 2 as a function of time. The grain growth from 6 to 20 μm was observed for various times and temperatures of annealing. Microstructures after annealing at 300 and 450 °C for 5 minutes and 6000 minutes are shown in Fig. 3a and b and Fig. 3c and d, respectively. The grain size obtained upon annealing at 300 °C for 5 minutes was found to be 6.1 ± 1.0 μm whereas it became 16.8 ± 3.0 μm after 6000 minutes of annealing. After annealing at 450 °C for the same

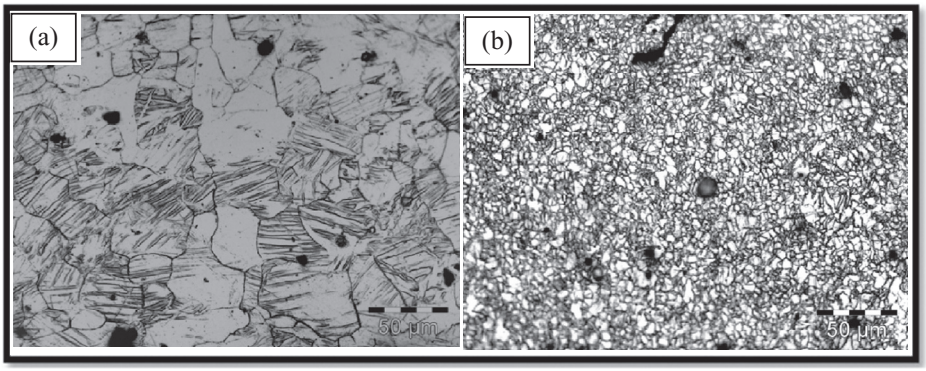

Fig. 1. Optical micrographs of the AZ31 Mg-alloy (a) as-received plate and (b) calibre rolled rod at 300 °C.

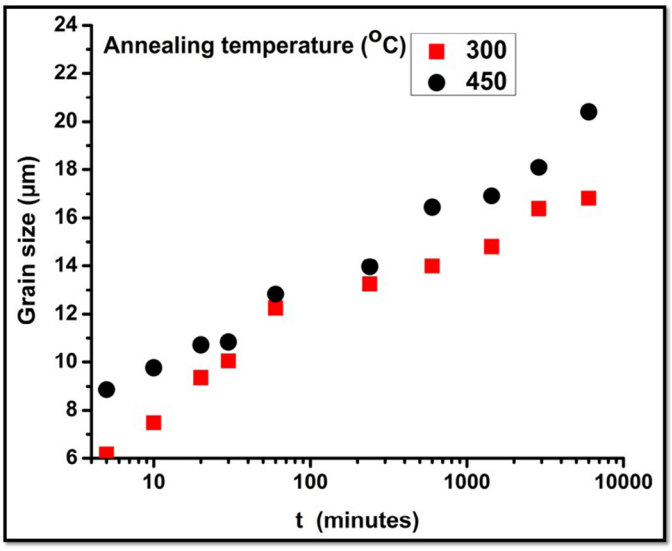

Fig. 2. Grain size measured as a function of annealing time at 300 and 450 °C.

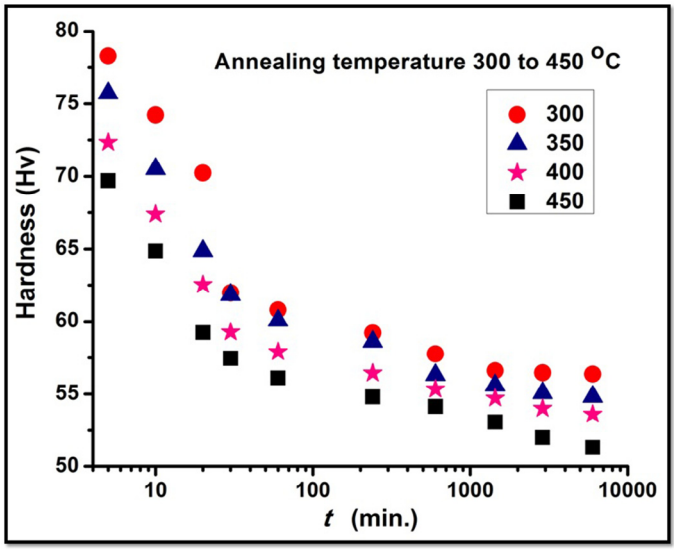

Fig. 4. Hardness measured as a function of annealing time at different temperatures.

durations, these grain sizes were 8.8 ± 1.0 and 20.4 ± 3.0 μm respectively. The grain growth was faster initially up to 60 minutes after which it became sluggish during longer annealing time.

3.3. Effect of annealing on hardness

The hardness measured as a function of annealing time at different temperatures is plotted in Fig. 4, which reveals a rapid reduction in hardness up to first 60 minutes of annealing time and then the change becomes less sensitive to annealing time. The values of micro-hardness after annealing for various lengths of time and at different temperatures were noted to decrease as the grain size increases with the increase in time and temperature of annealing.

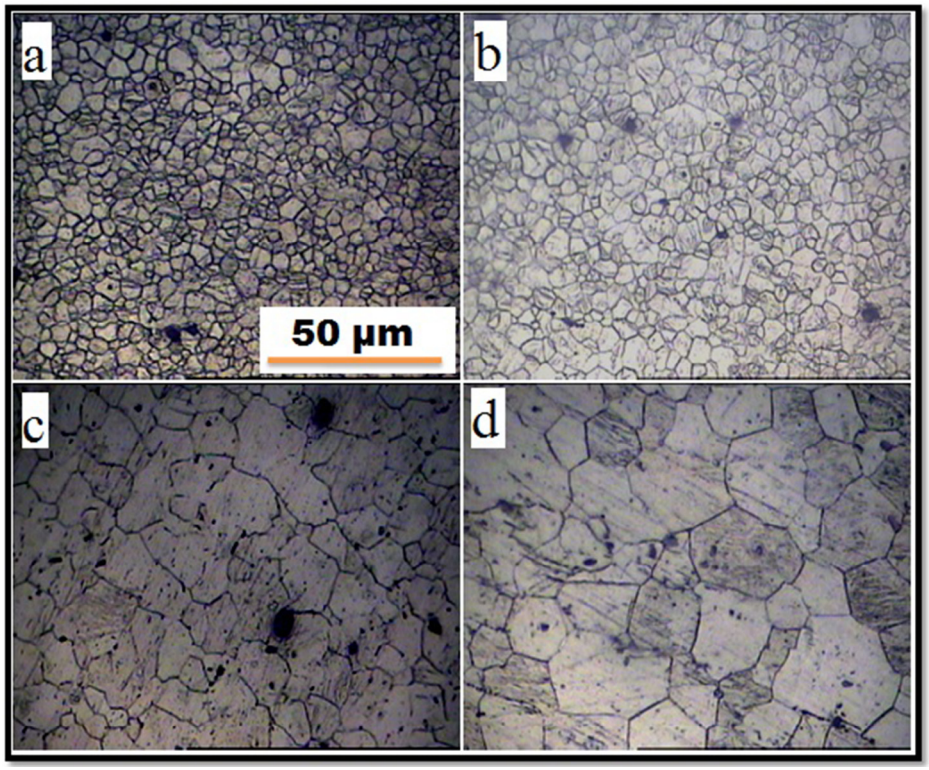

Fig. 3. Optical micrographs after annealing for 5 minutes at temperatures (a) 300 °C and (b) 450 °C; and for 6000 minutes at temperatures (c) 300 °C and (d) 450 °C.

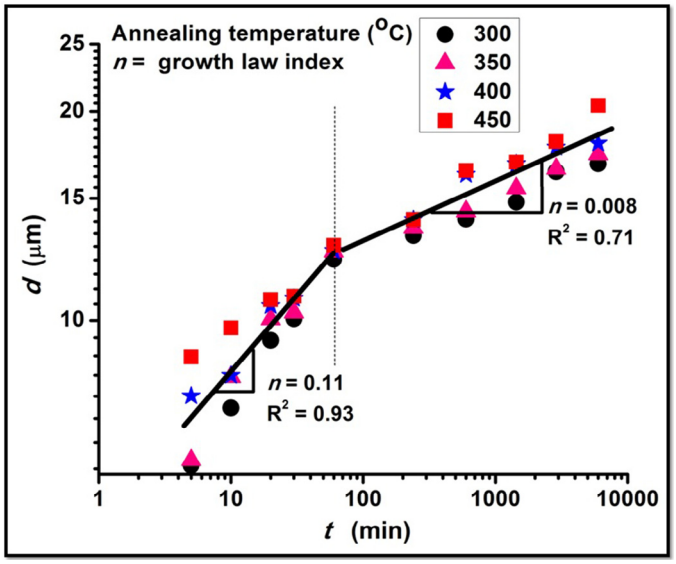

Fig. 5. Plot of log d vs log t, giving growth law index "n" after annealing the samples at temperatures of 300–450 °C and for time from 5 to 6000 minutes.

4. Discussion

4.1. Grain growth kinetics and mechanisms

As plotted in Fig. 5, the variation in grain size with annealing time in log–log scale reveals bilinear behaviour of grain growth kinetics, with the variation in grain size with time exhibiting $n = 0.11$ for the short annealing time (up to 60 minutes) and 0.008 for the longer duration (above 60 minutes).

Normal grain growth data can usually be fitted to an equation of the form:

$$d = kt^n \tag{2}$$

where d is the average grain size, k is a rate constant, t is isothermal annealing time and n is the growth law index (time exponent).

If the measured growth law index is $n = 0.5$, as known for pure metals, then the boundary migration of grain growth obeys the relationship:

$$V = MP \tag{3}$$

where V is the grain boundary migration rate, P is the driving force for a hypothetically pure material and M is the intrinsic mobility of the boundary in the pure material [13,14]. However, these theories do not consider the solute effects on the growth law. Many investigators proposed the theories that consider the solute effects that include:

- The Lücke–Detert theory [15]
- The Gordon and Vandermeer theory [16]
- The Cahn and Lücke–Stüwe theory [8]

The addition of solute, even in the parts per million ranges, was observed to reduce boundary mobility drastically. It is also a common observation that the addition of a small amount of solute increases the measured activation energy for boundary migration to higher values; sometimes much larger than for any identifiable atomistic process.

Rath and Hu [17] pointed out the fact that a linear dependence of grain growth rate on driving force was seldom observed except for grain growth in metals of ultra high purity and at high annealing temperature, near the melting point. These authors also suggested that no meaningful activation energy could be measured for grain growth unless the value of $n = 0.5$ was employed. They suggested that the temperature dependence of the mobility parameter in grain growth could not be regarded as a single thermally activated process. To understand the mechanism involved in grain boundary migration, Q was calculated by considering the variants of n, based on the present work and that reported for pure metals ($n = 0.5$), quasi-single phase ($n = 0.33$) and two-phase ($n = 0.25$) materials [18–20].

The initial grain size d_0 can only be neglected if it is very small in comparison to grain sizes at long annealing times. But, for short annealing time, the d_0 is not small enough compared to d. Therefore, the analysis made by using Eq. (2) is not justified. Using the appropriate d_0 for each of these grain growth stages, Fig. 5, it is found that the data would fit into the equation [21]:

$$d^2 - d_0^2 = k(t - t_0) \tag{4}$$

where t_0 is the time at which grain growth would have started on completion of recrystallisation.

This relationship can be used to measure the activation energy for grain growth (Q) accurately. The difference ($d^2 - d_o^2$) is determined over the range of temperatures. A plot of log ($d^2 - d_o^2$) versus T^{-1} (K^{-1}) yields the activation energy for grain growth to vary at various time periods as illustrated in Fig. 6. However, it shows very high values of the activation energy. Table 2 lists the values of activation energy calculated by considering growth law index (n), constant (t) and constant (d) approaches. It shows very high values of activation energy. However, in the beginning of grain growth the activation energy was found to be 65–91 kJ mol^{-1}, which is close to the anticipated Q of 92 kJ mol^{-1} reported for grain boundary diffusion in magnesium.

There exist very few studies on annealing of Mg-alloy AZ31 upon processing by various methods of plastic deformation, as summarised in Table 1 from the literature [9–11]. The limited studies reported in the literature also exhibit a wide variation in the activation energy for grain growth (29–200 kJ mol^{-1}), as compared to that expected on the basis of grain boundary (92 kJ mol^{-1}) or lattice (135 kJ mol^{-1}) diffusion [1].

As pointed out earlier for the variation in n values of kinetics law [18–20], it could also be reasonable to assume that no meaningful value of activation energy for grain growth might appear for the system like AZ31 Mg-alloy, which contains various alloying or impurity elements [22]. However, it is interesting to note from Table 2 that grain growth at five minutes of annealing, and using the present value of $n = 0.1$ and so also approaching the calculation by considering either constant d or constant time for Q, gives $Q = 64.5$–90.7 kJ mol^{-1}. These values of Q support the grain boundary diffusion to be

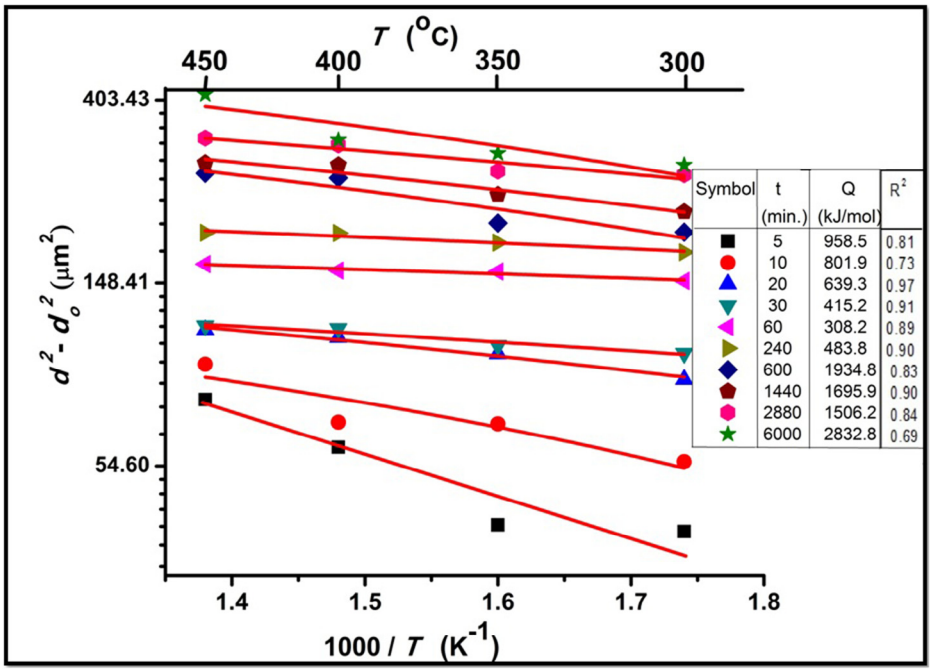

Fig. 6. Arrhenius plot to determine the activation energy for grain growth after annealing the samples at temperatures of 300–450 °C and for time from 5 to 6000 minutes.

the mechanism for grain growth. However, $n = 0.1$ does not support the kinetics of grain growth commonly predicted but this $n \sim 0.1$ happens to be true for a wide range of materials [19]. Further to the theoretical and experimental results on kinetics and mechanisms for grain growth, and the sources of deviations from thereof elaborated in the literature [17–20], in terms of n and Q values, the following experimental results and the inferences emerging could add to our understanding of grain growth in this alloy. (i) Twins formed profusely during CR disappear very fast during subsequent annealing. It is during the

Table 1
Studies on annealing of Mg alloy AZ31 as summarized from literature.

Processing type	Annealing		Grain size (μm)		Activation energy Q (kJ mol^{-1})	Growth law index (n)	Ref.
	Temp. (°C)	Time (min)	Initial (d_o)	Final (d)			
Rolling	375	1440	3	7–39	–	–	[9]
Compression	350–500	50	11	29	200	1.27	[10]
ECAP*	250–500	60	1.9	4–32	94.2 29.5	0.5	[11]
Calibre rolling	300–450	5–6000	3	6–20	12.3–76.48 308.2–2832.8	0.1 0.5	Present study

* ECAP = equal channel angular pressing.

Table 2
Activation energy calculated considering growth law index (n), constant (t) and constant (d).

t (min)	Activation energy Q (kJ mol^{-1})					
	$n = 0.5$	$n = 0.33$	$n = 0.25$	$n = 0.1$	Constant t	Constant d (μm)
5	958.5	10784.9	108943.1	76.4	64.5	90.7 at $d = 07$
10	801.9	10393.3	120434.5	43.7	46.6	414.5 at $d = 10$
20	639.3	9651.7	129722.3	53.3	31.5	520.4 at $d = 12$
30	415.2	6513.1	90849.69	63.5	19.7	2721.1 at $d = 13$
60	308.1	5796.5	96903.01	17.7	12.3	8600.8 at $d = 14$
240	483.8	9893.6	179928.3	12.3	17.7	–
600	1934.8	44267.8	902235.7	19.8	63.5	
1440	1695.9	40493.3	860627.4	31.8	53.2	
2880	1506.1	38919.5	894702.3	46.7	43.7	
6000	2832.8	78933.1	1961359	64.5	76.4	

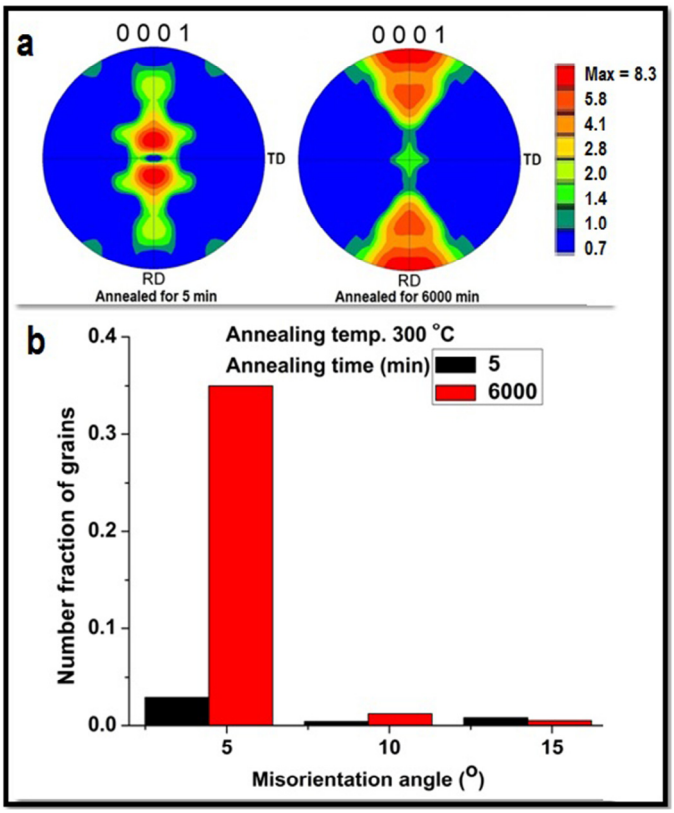

Fig. 7. EBSD analysis of the annealed samples at 300 °C. (a) Pole figure (0001). (b) Misorientation angle versus number fraction of grains.

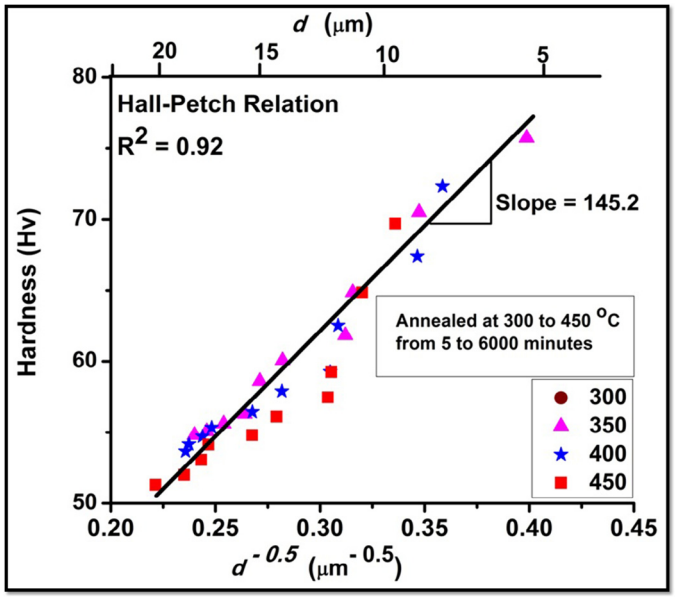

Fig. 8. Hall–Petch relationship after annealing of the samples at temperatures of 300–450 °C and for time from 5 to 6000 minutes, without distinguishing the temperature effect.

process of twinning getting eliminated that the activation energy for grain growth is comparable with that for grain boundary diffusion. The subsequent dramatic change in the value of Q seems to suggest that the structural element of twin boundaries could provide extrinsic grain boundary dislocations to affect the otherwise CR grain boundary structure. Thus, the Q values on longer annealing time may be affected. (ii) Concurrent to grain growth, texture evolution is seen during annealing as illustrated in Fig. 7a by comparing the pole figures obtained upon annealing for 5 min and 6000 min at 300 °C. This change in texture could resist grain boundary migration which could occur via the grain interior. (iii) The examination of grain boundary nature by EBSD, Fig. 7b, also revealed formation of a greater proportion of low angle boundaries (35%), leaving a lesser proportion of high angle grain boundaries (65%) with the increase in annealing time from 5 min to 6000 min at 300 °C. It is known that the rate of migration of high angle grain boundaries is faster than that of low angle boundaries [23,24]. This property, in conjunction with the presence of low driving force due to the substantially reduced grain boundary area, can provide greater resistance to grain growth.

4.2. Contributions of grain size and twinning to strength

The grain size (d) dependence of yield strength (σ_y) is given by Hall–Petch type relationship [25,26]:

$$\sigma_y = \sigma_0 + k_y d^{-0.5} \quad (5)$$

which predicts that as the grain size decreases the yield strength increases. Here, σ_0 and k_y represent the Hall–Petch constants having significance to the strengthening caused by grain interior and grain boundary, respectively. This strengthening by grain refinement is experimentally found to be true over the grain sizes ranging from 1 mm to 1 μm [27]. This relationship is valid for strength, which is also related to hardness. It is seen that the hardness decreases as the grain grows at all the temperatures with increasing time (Fig. 4). Micro-hardness is plotted against grain size ($d^{-0.5}$) irrespective of the annealing temperature in Fig. 8. It is seen to clearly obey the Hall–Petch type relationship ($R^2 = 0.92$), which can be expressed as:

$$H_v = H_0 + k_v d^{-0.5} \quad (6)$$

Following the relationship between yield strength and hardness, viz. Eq. (1), the Hall–Petch type relationship for yield strength can be written by putting the values of σ_0 and k_y in Eq. (5) as:

$$\sigma_y = 59.4 + 421.6 d^{-0.5} \quad (7)$$

Similar plots were considered for grain sizes obtained at annealing temperatures of 300, 350, 400 and 450 °C individually, and the Hall–Petch constants σ_0 and k_y were found to range between 55.8–63.7 MPa and 403.7–447.5 MPa $\mu m^{-0.5}$ respectively. However, no systematic effect of annealing temperature was noted in the variation of σ_0 and k_y. The large value of k_y exhibits the strong grain size dependence of flow stress. Therefore, the reduction in strength (hardness) is attributed to the increase in grain size by annealing. As listed in Table 3, there appear wide variations in the values of σ_0 and k_y in the literature [28–30]. The value of σ_0 is related to the critical resolved shear stress (CRSS) for the easiest (basal) slip system operating

Table 3
Comparison of the present values of σ_0 and k_y in AZ31 Mg-alloy with the literature.

Processing	Grain size (μm)	σ_0 (MPa)	k_y (MPa μm$^{-0.5}$)	Ref.
FSP**	5–10	16.4	119.5	[28]
Rolling	5.2–23	85	200	[29]
Rolling	13–140	115	272	[30]
Calibre rolling	6–20	59.4	421.6	Present study*

* In the present study, σ_0 was found to vary from 55.8 to 63.7 MPa, whereas k_y varied from 403.7 to 447.5 MPa μm$^{-0.5}$ with no systematic effect of annealing temperature (300–450 °C) used for grain growth.
** Friction stir processing.

within the grain volume. The value of k_y depends on temperature, texture, composition, preparation method employed for producing the materials, along with its dependence on the CRSS for the nonbasal (more difficult) slip systems required to operate near the grain boundary. The values of σ_0 and k_y can depend in general on the state of the material developed, in the way it was produced by varying the working temperature or thermo mechanical treatments it was subjected to, as is the case for other mechanical properties in general [22].

A close examination of the values of $\sigma_0 = 59.4$ and $k_y = 422$ MPa μm$^{-0.5}$ in Table 3 reveals that the grain boundary component of strengthening k_y in calibre rolled material is much higher than that reported by other methods of processing of this AZ31 alloy. The source of this enhanced strengthening by grain boundaries is not clear at this stage. However, it appears tempting to think that resistance to grain boundary migration during annealing, requiring much higher activation energy for grain growth, could have its origin in the structural change in grain boundaries. Such change in grain boundary structure, when remains so upon quenching of the annealed material, can contribute to different grain boundary strengthening effect [31]. In fact, Sangal and Tangri [32] reported a difference in the effects of grain boundary strengthening between equilibrium and non-equilibrium boundaries in type 316L stainless steel. In the present work, the AZ31 Mg-alloy contains profound number of twins which increase rapidly with increasing annealing time [33]. Therefore, probably, the generation of twins introduces additional dislocations into the grain boundaries to become stronger than the otherwise equilibrium grain boundary structure.

5. Conclusion

Grain refinement in Mg–3Al–1Zn alloy was achieved from 33 μm in as-mill rolled state to 3 μm upon calibre rolling of about 76% at 300 °C. Annealing of these samples for 5–6000 minutes at 300–450 °C and analyzing the grain growth along with its effect on hardness, at room temperature, leads to the following conclusions:

1. Grain growth occurs from initial grain size of 3 μm up to 20 μm and is faster initially up to 60 minutes, but then becomes sluggish at longer annealing time. The grain growth kinetics law reveals two values of growth law index with $n = 0.11$ for the shorter annealing time (up to 60 minutes) and its marginal decrease ($n = 0.008$) for the longer annealing time (above 240 minutes).
2. The activation energy Q for grain growth was found to be very high for all the growth law indices except $n = 0.1$. However, the activation energy in the beginning of grain growth (65–91 kJ mol^{-1}) supports grain boundary diffusion to be the mechanism. At longer annealing time, the exceptionally higher or lower activation energy values arise from other structural evolution in the material.
3. Micro-hardness varies as a function of grain size following the Hall–Petch type relationship with the equivalent H–P parameters $\sigma_0 = 59.4$ ($H_o = 18.4$) MPa and $k_y = 421.6$ ($k_v = 145.2$) MPa μm$^{-0.5}$, irrespective of annealing temperature. The k_v found in the calibre rolled material is much larger than known in this material that was processed by conventional rolling or friction stir processing.

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