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The effect of grain refinement by warm equal channel angular extrusion on room temperature twinning in magnesium alloy ZK60

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The effect of the grain structure produced by warm equal channel angular extrusion (ECAE) on twinning under subsequent room temperature deformation of magnesium alloy ZK60 was investigated. It was shown that a bi-modal grain structure, characteristics of which are determined by the ECAE temperature and the number of passes, has a strong effect on the tendency to room temperature twinning. The highest density of twins generated during room temperature deformation was found in material pre-strained by ECAE at 300°C that exhibited a uniform recrystallised grain structure with an average grain size large enough for twinning to occur.

1. Introduction

A number of observations, mainly of the behaviour of body centred cubic (bcc) metals, show that twinning can be suppressed by pre-straining at a higher temperature [1], although Boucher and Christian [2] reported that very small pre-strain increased the amount of twinning in niobium, while raising the twinning stress. The amount of pre-strain required to suppress twinning is reported to decrease as the temperature of testing increases and also with the rate of the strain subsequently imposed: it may depend on the form and homogeneity of the substructure produced [1]. Little information is available on the effect of pre-strain on hexagonal closed packed (hcp) metals, magnesium in particular. Reed-Hill [3] observed almost 50% increase in room temperature ductility of zinc specimens pre-strained transversely at low temperature (77 K). He attributed this to nucleation of many twins, which grew broader under tension at room temperature. The lack of such twins in material deformed slowly at room temperature without pre-strain was attributed to difficulty in the nucleation of twins. The measured twinning stress decreased slightly as temperature decreased in most deformation modes in hcp metals, except for $\{10\bar{1}1\}$ twinning in which an increase of twinning stress was reported. In polycrystalline Zr, room temperature deformation at "moderate" strain rates is by $\{10\bar{1}0\}$ prismatic slip and $\{10\bar{1}2\}$ twinning with occasional $\{11\bar{2}1\}$ twins, whereas at 77 K the amount of $\{10\bar{1}2\}$ twinning increases, with many more $\{11\bar{2}1\}$ twins and some $\{11\bar{2}2\}$ twins.

Grain size, d, is another variable that governs the amount of deformation twinning. The lower yield stress, determined by both slip and twinning in polycrystalline metals and alloys, often, but not necessarily, obeys a Hall-Petch type of relation [1]. For instance, Voehringer [4] found that the twinning stress in a Cu-5 at%Sn alloy followed the Hall-Petch relation, whereas twinning stress in a Cu-15 at%Zn alloy increased linearly with d^{-1} rather than $d^{-1/2}$. The latter behaviour is consistent with the analysis of the relationship of twinning stress and twin geometry given by Friedel [5]. The effect of grain size is somewhat complicated by the possibility that grains of mixed size, as commonly obtained in the thermomechanical processing of Mg alloys, may not behave according to the law of mixtures, but may rather exhibit superior properties, as reported by Ma's group [6] in reference to Cu.

In fact, so many variables, some strongly interdependent—temperature, strain rate, amount of pre-strain, grain size, composition and presence of dispersed phases—may influence twinning [1, 3] that only general trends can be predicted.

Because ZK60—a magnesium-based alloy under consideration—has an hcp structure, with $c/a \sim 1.6$, the basal plane is the only close-packed one, although the prismatic planes $\{1\bar{1}00\}$ may become active in some orientations, as may the less closely packed pyramidal planes $\{1\bar{1}01\}$ at higher temperatures. The most likely Burgers vector at room temperature, **c**, provides only four independent slip systems [7] which are insufficient to satisfy the Taylor requirement of five independent active slip systems for compatible plastic flow in polycrystals. It has been suggested, though, that compatible deformation is achieved by four independent slips and local stress relief by twinning [8]. In the case of deformation by twinning, the number of independent modes arising from a single twin system ranges from zero to five, depending on the sense of the direction of twinning

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shear with respect to the state of internal stress, which is variously tensile and compressive in a polycrystal subjected to homogeneous stress. Five independent modes are therefore available if the local stress state is sufficient for both tension and compression twins to operate [7]. The contribution of twinning to general plastic flow is to reorient grains more favourably, so that total plastic strain can be increased substantially by the occurrence of second order twinning, e.g. $\{1\bar{1}01\}$ twinning in Mg, followed by $\{10\bar{1}2\}$ twinning [7]. Using Mg as an example, Reed-Hill [3] found that the volume fraction of twinned material varies linearly with total strain when strains are not too large (say, $<10\%$). Although the twinned fraction continues to increase at higher strains, albeit at a lower rate, the greater difficulty in distinguishing twinned and untwinned regions makes quantitative measurement difficult. The growth in volume twinned during deformation is a result of increase in the number and size of twins.

The effect of chemical composition makes for further variability in behaviour. Twinning may carry interstitial solutes into non-equivalent sites in bcc, fcc and hcp metals, so that the effect of interstitial solutes is generally to decrease and, eventually, to eliminate twinning [1]. Substitutional solid solution often increases the tendency to twinning in bcc metals and also has a strong effect in fcc metals [1], but a similar analysis does not seem to have been made for hcp metals. The effect of size distribution and volume fraction of second phase particles as well as the role of structural factors such as coherency have not been investigated systematically [1]. Ageing or precipitation in many alloys tends to delay twinning or suppresses it altogether [1].

Grain refinement by methods of severe plastic deformation has become important as a means of enhancing the properties (notably strength and ductility) of many alloys [9]. The present work arose from an investigation into the way in which pre-straining by warm equal channel angular extrusion (ECAE), that results in grain size reduction influences deformation twinning in subsequent room temperature deformation either by further ECAE or by uniaxial tensile straining. While twinning represents only one of the possible deformation modes operating concurrently with dislocation glide, the occurrence of twins in the microstructure will be the main focus of the present paper.

2. Experimental

Samples of 20×10 mm^2 cross-section and 70 mm length were machined from a continuously cast billet of ZK60 (Mg-4.95%Zn-0.71%Zr). The samples were homogenised for four hours at 460°C in alumina powder, to reduce surface oxidation. The homogenisation conditions were chosen on the basis of previous time-temperature homogenisation experiments to avoid incipient melting and possible grain growth. As a result of homogenisation, grain boundary precipitates were dissolved, leaving regions rich in zirconium. The initial microstructure consisted of equiaxed grains with average diameter of 45.5 μm and isolated zinc-zirconium particles dispersed within the grains or in clusters.

The samples were subjected to 8 passes of ECAE (Routes A and C, cf. [9]) with a 120° die, at a ram speed of 0.5 mm/s. The temperatures used were 200, 250 and 300°C. After deformation by warm ECAE, the specimens went through a single pass of room temperature ECAE (this time with a 90° die). The occurrence of twins was evaluated by microscopy.

Severe plastic deformation of ZK60 is known to produce a grain structure with a minimum average grain size of about 1 μm [10] or larger [11]. The microstructure obtained in the present work was of a similar scale. Therefore, the evaluation of microstructure and counting of twins was possible with an optical microscope (Olympus, JSM-840), while rotation of grains was studied by scanning electron microscopy (LEO-1530). Transmission electron microscopy (Philips CM20) was used to check the possible occurrence of twinning in small grains with diameter less than 2 μm.

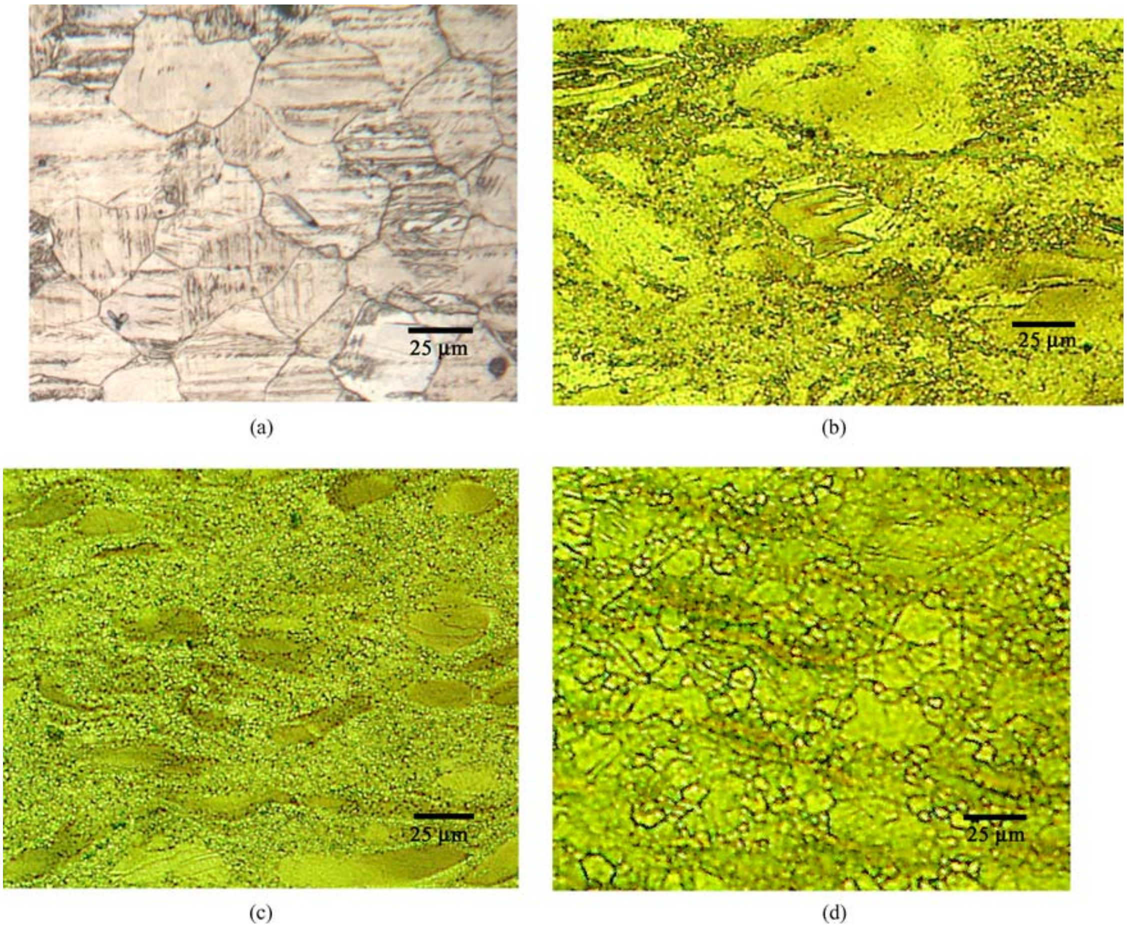
3. Experimental results

3.1. Evolution of microstructure under warm ECAE

For both routes and all temperatures employed, warm ECAE resulted in a bi-modal microstructure with two populations of grains distinctly different in size, where large elongated grains were embedded in regions of small recrystallised grains. The initial microstructure (Fig. 1a) and the microstructures obtained after eight ECAE passes at different temperatures are shown in Fig. 1 (Route A) and Fig. 2 (Route C).

It is evident from the figures that overall grain refinement upon ECAE was observed for both routes and at all temperatures. An increase in processing temperature shifts the grain size distribution toward a larger fraction of small recrystallised grains. Due to a significant difference in the grain dimensions in the two populations, the average grain size of large and small grains could be assessed separately, using the linear intercept method. The mean grain size (Fig. 3) and the area fraction of large grains (Fig. 4) depend on temperature, ECAE route and number of passes. A general trend seen in these diagrams is that the area fraction of large grains and their average diameter decrease with temperature and the number of passes for both routes. This tendency was more pronounced for Route A, as for Route C the scatter in the grain size measurement was greater due to grain rotation. It was observed that dynamic recystallisation set in earlier, i.e. required a smaller number of ECAE passes, at higher temperatures. The volume fraction of dynamically recrystallised grains also increased with temperature.

The degree of grain refinement increases with the number of ECAE passes. Due to severe deformation introduced after 8 ECAE passes, the mean size of large grains (in the transverse cross-section) dropped from 45.5 μm to 26.7 ± 2.5 μm for Route A and to 29.7 $\pm$ 4.1 μm for Route C. A slight dependence on the temperature of warm ECAE pre-straining is seen in Fig. 3. At the same time, a new population of small recrystallised grains emerged. Their average diameter ranged from 2 μm for ECAE at 200°C to 4 μm for ECAE at



(a) (b) (c) (d)

Figure 1 Microstructures of warm ECAE deformed specimens vis-à-vis the initial microstructure (Route A) (a) initial, (b) after 8 passes at 200°C, (c) after 8 passes at 250°C, (d) after 8 passes at 300°C.

300°C. As already mentioned, the area fraction of large grains was found to be smaller for higher ECAE temperatures, cf. Fig. 4, a very small number of large grains being retained after pressing at 300°C.

3.2. Effect of warm ECAE pre-straining on twinning behaviour

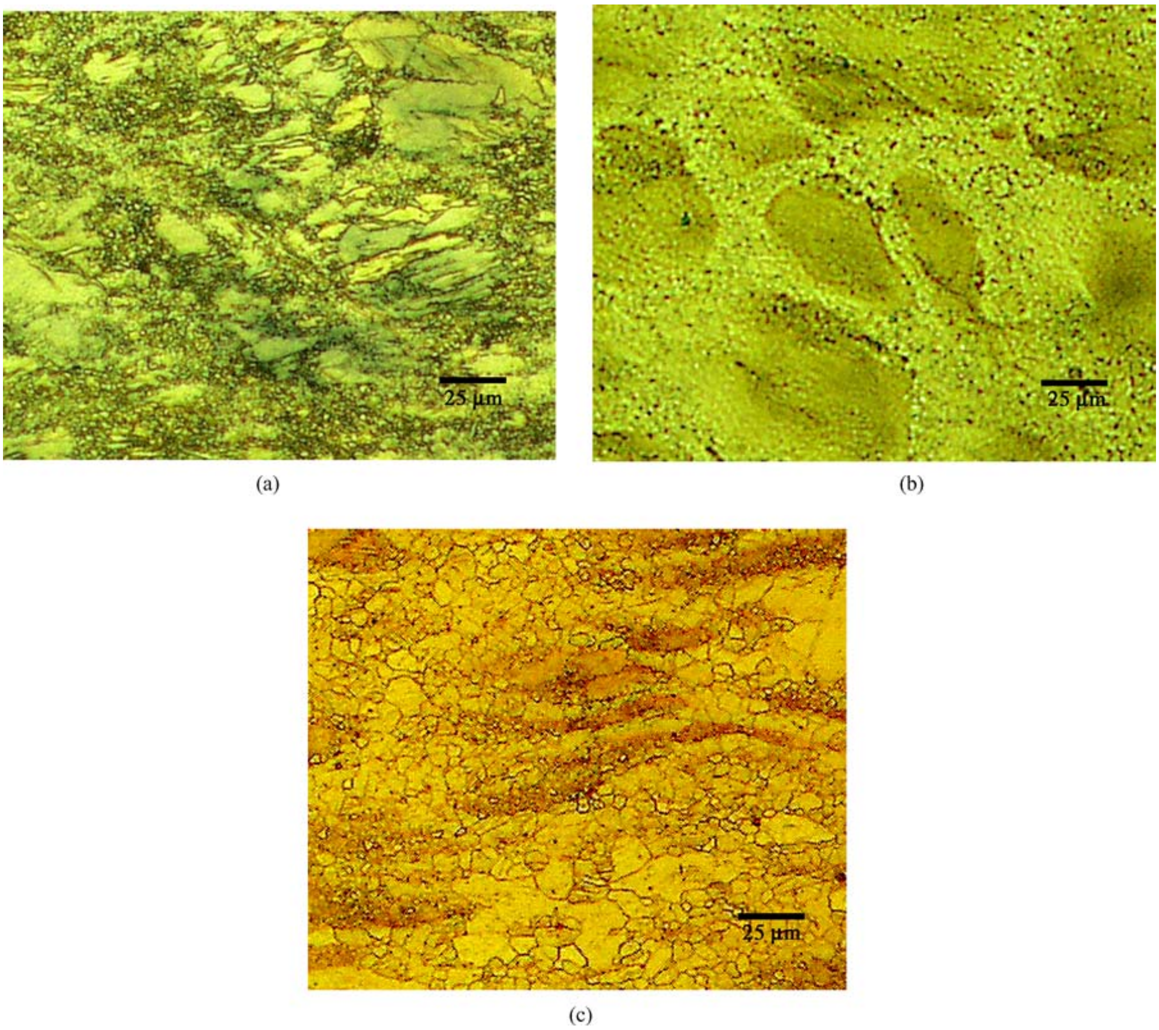
3.2.1. Warm ECAE

While the main focus of this study was the investigation of twinning under *room temperature deformation* following warm ECAE, it is worth reporting that twinning occurred under warm ECAE itself, as well. Evidence for that is seen in Fig. 5 that shows microstructures of specimens deformed by 8 passes of ECAE under Route A at three different temperatures. Deformation twins in large grains are clearly recognisable at all three temperatures. The occurrence of twins is obviously reduced with increasing ECAE temperature, as could be expected [1]. Indeed, direct count of twins in large grains shows that the density of twins (*defined as their number per unit area of large grain regions*) drops almost to zero as the temperature of warm ECAE increases from 200 to 300°C, Fig. 6.

The evolution of the density of twins in large grains with the number of warm ECAE passes was also studied. Measurements were taken after each even pass number. It was found that the twin density acquired within the first two passes either stayed constant, as in the case of ECAE at 200°C, or exhibited an apparent decrease with the number of passes, as in the other two cases. This decrease can be attributed to dynamic recrystallisation at higher temperatures that is believed to be enhanced in the presence of twins [12]. Indeed, transmission electron microscopy (TEM) provides evidence of the occurrence of small recrystallised grains that appear to have nucleated at the twin boundary seen in the micrograph, Fig. 7.

The density of twins detected after two ECAE passes (about 3000 and 4500 mm^{-2} for routes A and C, respectively) was nearly the same for 200° and 250°C, while for 300°C, values two order of magnitude smaller were recorded.

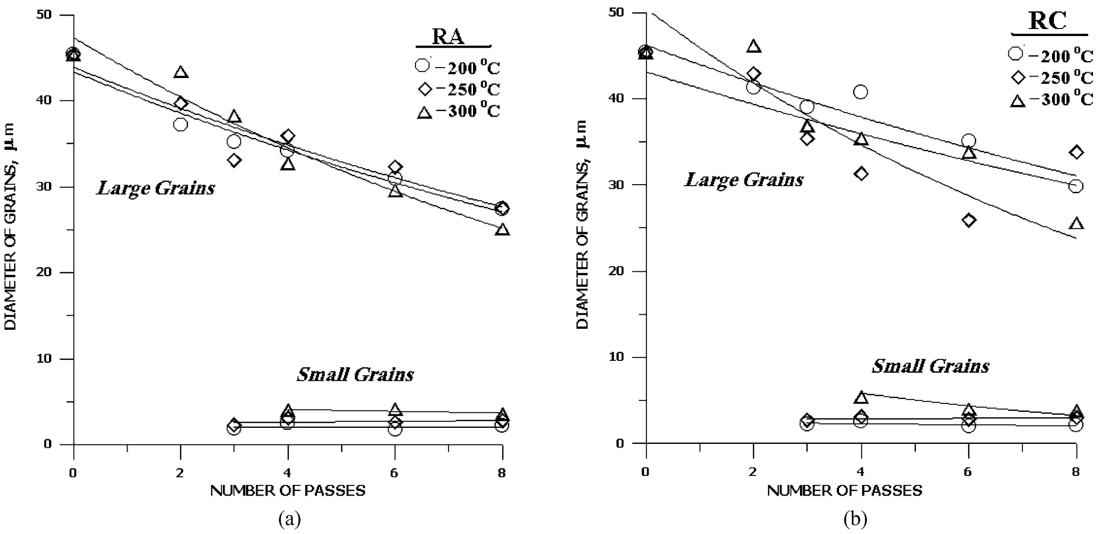
Possible influence of texture on twinning was also investigated. The incoming continuously-cast material had a weak texture with basal planes normal to one transverse direction and the poles of prismatic planes aligned in a weak fibre texture around this direction. After eight passes of ECAP via Route A, the poles of the basal planes were aligned along an arc about 30° to the extrusion direction and the prismatic planes rotated toward the shear plane. Higher temperatures (250 and 300°C) changed the texture relatively little, but temperature and the use of Route C had a light tendency to promote axial symmetry about the extrusion direction, most notably that of the distribution of prismatic planes. The pole figures showing the texture after 8 passes of ECAE at 300°C using routes A and C and illustrating the mentioned tendency are presented in Fig. 8.



(a) (b)

(c)

Figure 2 Microstructures of warm ECAE deformed specimens (Route C) (a) after 8 passes at 200°C, (b) after 8 passes at 250°C, (c) after 8 passes at 300°C.



(a) (b)

Figure 3 Average grain size as a function of number of ECAE passes. The average diameter of large and small grains is shown for three ECAE temperatures. (a) Route A and (b) Route C.

This observed small effect of ECAP on texture is consistent with the findings of Ferrasse *et al.* [13], who found that although a strong texture developed under some circumstances in the first two-to-four passes of ECAP of an aluminium alloy, texture subsequently weakened and even approached random orientation. The strength and nature of initial texture had little effect on this behaviour. There was generally little difference between routes, although Routes A and B_c were most effective in randomizing texture and Route C produced and retained the strongest one. It should be noted that the work of Ferrasse *et al.* refers to cold ECAP. Ferrasse

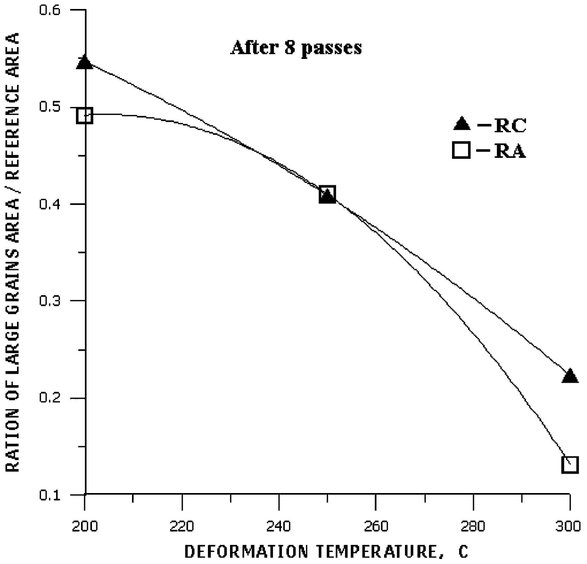


Figure 4 The area fraction of large grains as a function of ECAE temperature.

et al. refer to the effect of dynamic recrystallisation and grain/subgrain rotation as contributors to the weakening of texture at high strains. As those mechanisms are promoted by elevated temperature, warm ECAP cannot be expected to exercise a substantial influence on texture. This is confirmed by the observation that texture changed little with route and hence that the main factor governing the influence on twinning during deformation by hot ECAP was the temperature itself.

3.2.2. Room temperature ECAE of pre-strained samples

All samples pre-strained by ECAE at 200°, 250° and 300°C, as well as a homogenised undeformed specimen, were subsequently deformed at room temperature. The aim of these tests was to elucidate the effect of grain refinement achieved by pre-straining on the deformation mechanisms operating at room temperature. Room temperature deformation was performed by ECAE conducted in the same apparatus, but using a 90° die.

The results of optical microscopy show that twinning does occur at room temperature both in homogenised undeformed material and in the specimens that underwent warm ECAE (Fig. 9). It is seen clearly that at all three temperatures twinning occurred in large grains. Evidence of twinning in small grains was only seen in the case of pre-straining at the highest temperature used (300°C). The average grain size of 4 μm for the small grain population was the largest in this case. The density of twins (number of twins per unit area) was about

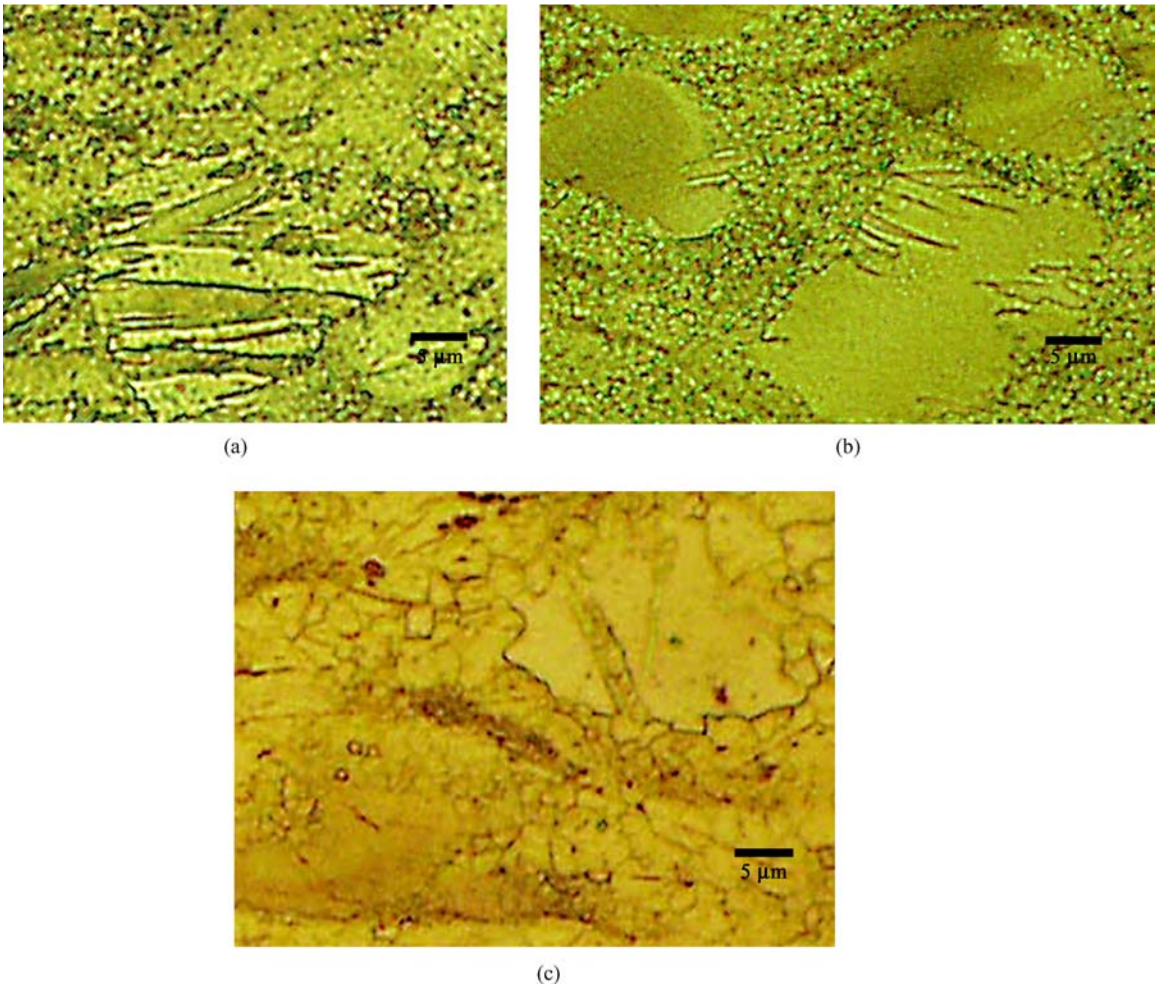


Figure 5 Microstructures of specimens deformed by warm ECAE (8 passes, Route A, transversal section) showing twins in large grains: (a) –200°C, (b) –250°C, and (c) –300°C.

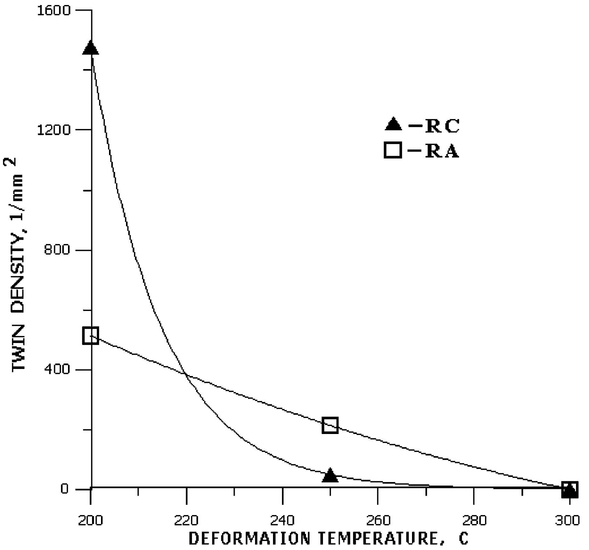


Figure 6 Density of twins in large grains after eight passes as a function of the ECAE temperature (Squares – Route A, triangles – Route C).

Figure 7 The microstructure after warm ECAE (300°C) showing large grain (∼40 μm) crossed by a twin and small grains (indicated in photo by arrows) where recrystallisation was initiated at the twin boundary.

3400 mm^{-2} (Route A) and 2400 mm^{-2} (Route C). (*It should be noted that in these values the total area was used as a reference, as the area fraction of large grains could be neglected.*)

Pre-straining the material by warm ECAE was seen to result in recrystallisation leading to the emergence of small twin-free grains under both ECAE routes used. The subsequent *room temperature pressing* was partially accommodated by twinning, the more so, the higher the warm ECAE temperature, Fig. 9. This is especially evident for Route C, where this trend is more pronounced. The increment in the overall twin density associated with room temperature ECAE as a function of pre-straining temperature is presented on Fig. 10. The aforementioned study of the texture after 8 passes of ECAE at various temperatures showed little change with temperature and only a minor dependence on the route, so that the increase in twin density in room temperature deformation with the temperature of pre-strain by ECAE is not attributed to difference in texture. Despite the presence of a significant population of large grains in the material pre-strained at 200°C, the increment in twin density upon room temperature deformation was fairly low. This may be rationalised as follows. The small grains are probably too small for twins to be generated (see Section 4 below), while the large grains have already been massively twinned during the pre-strain stage. As dynamic recrystallisation was nearly complete after pre-straining at 300°C, and the area fraction of large grains dropped appreciably, Fig. 4, the increase in the overall density of twins was associated with the occurrence of twinning in small grains. It should be noted, however, that the average grain size in the 'small' grain population (4 μm) was not so small any longer. The case of pre-strain at 250°C is intermediate between these two cases.

3.3. Deformation mode in case of fine grains

To observe the deformation mode in fine-grained material obtained by eight ECAE passes at 300°C a compression test was also conducted. A grid of fine (about 1 μm thick) lines was engraved with a diamond stylus onto the electrochemically polished specimen surface. After a room temperature compression test, the deformed scribed lines were observed in a field emission gun scanning electron microscope, cf. Fig. 11.

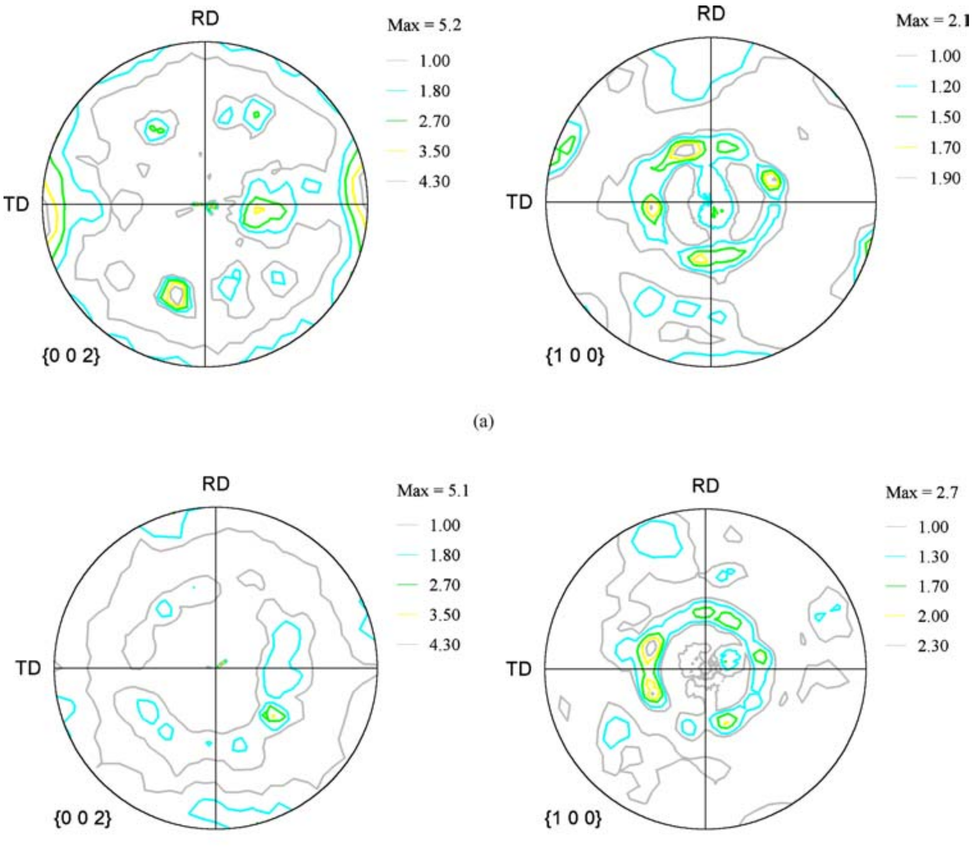
The trace of a scribed line remained continuous after deformation; a change in direction appears to provide evidence of grain rotation. Lack of steps on the scribe line suggests that grain boundary sliding did not occur.

Observation of twinning in small grains by optical microscopy presented above was confirmed by scanning electron microscopy, as seen from Fig. 11 that exhibits twins. This evidence suggests that room temperature deformation of the small grain microstructure produced by 300°C ECAE involves twinning (in the fraction of the grains that are about 3–4 μm or larger) as well as grain rotation.

4. Discussion

The results relating to the effect of warm ECAE on the occurrence of twinning suggest that deformation twinning at room temperature is suppressed by grain refinement associated with pre-sraining. The main effect of grain size on the occurrence of twinning can be seen in the gradual suppression of twinning activity with increase in the fraction of small grains. Dislocation glide then becomes the predominant mechanism of plastic deformation. Twinning under warm ECAE was observed chiefly in large grains. Small grains exhibited twinning only in specimens that underwent ECAE at 300°C—when they actually were fairly large (around 4 μm in diameter on average) and could be categorised as 'small' in a relative sense only.

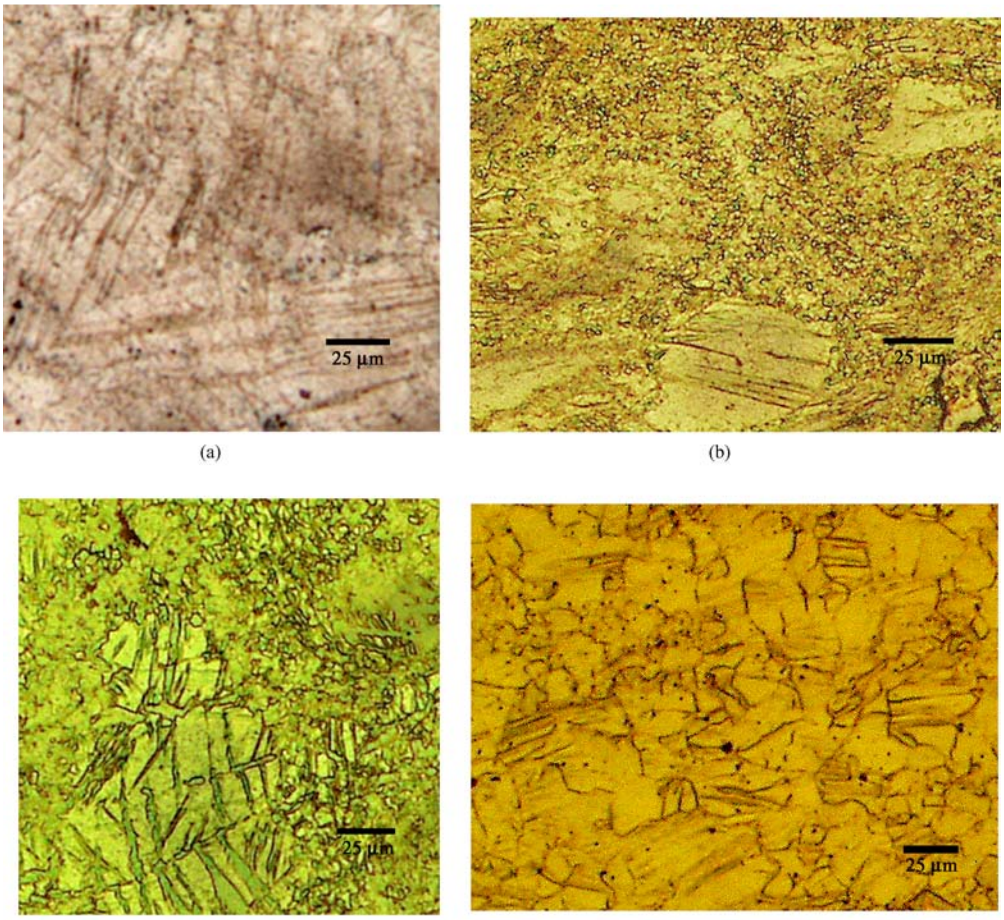
The fact that reduction in grain size apparently led to suppression of twinning can be rationalised by looking



(a)

(b)

Figure 8 Pole figures showing the texture after 8 passes of ECAE at 300°C: (a) Route A and (b) Route C.



(a)

(b)

(c)

(d)

Figure 9 Microstructure of samples after room temperature ECAE: (a) homogenised material, (b) pre-strained by 8 ECAE passes at 200°C, (c) pre-strained by 8 ECAE passes at 250°C, and (d) pre-strained by 8 ECAE passes at 300°C. (Pre-straining: Route A).

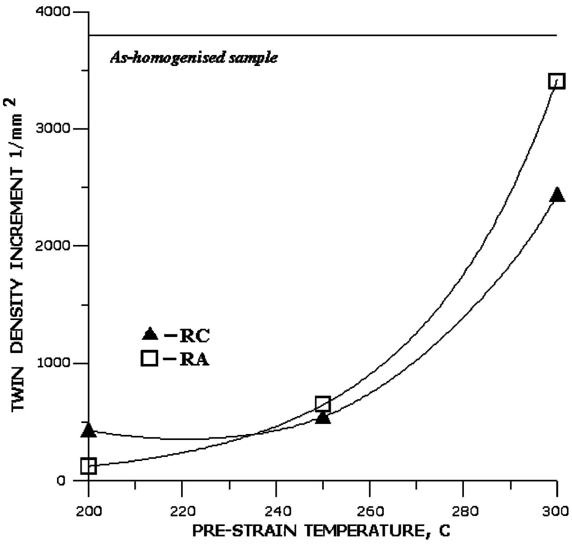


Figure 10 Increment in density of twins after room temperature ECAE as a function of the ECAE pre-strain temperature (Routes A and C).

at the relation between the stress σgnd the dimensions of a twin in equilibrium, considered by Friedel [5] (cf. Equation (6.18)):

$$\sigma \cong \frac{\mu}{2} s \cdot \frac{h}{L} \tag{1}$$

Here μ is the shear modulus, s is the shear produced by the twin (of the order of 0.1–1 [5]), $L$is the length of the twin and h its thickness. If the stress required for twinning, $\sigma_{\mathrm{TWINNING}}$, is assessed on the basis of this equation, the most favourable condition for twinning corresponds to L being identified with the grain size d:

$$\sigma_{\mathrm{TWINNING}} \cong \frac{\mu}{2} s \cdot \frac{h}{d}. \tag{2}$$

The $1/d$ dependence of the twinning stress is known from the experimental literature [1], although a Hall-Petch-like $1/\sqrt{d}$ dependence was also reported, again in Ref. 1. As the stress required for dislocation glide controlled plastic deformation follows a $1/\sqrt{d}$ dependence, it is obvious that for sufficiently small d, the twinning stress given by Equation 2 will become larger than the stress required for dislocation glide, σ_{GLIDE}. Obviously, twinning will not be the preferred deformation mode then. With data on the grain size dependence of strength for coarse grained ZK60 compiled from literature [14], the Hall-Petch relation for dislocation glide controlled deformation can be written as

$$\sigma_{\mathrm{GLIDE}} = 197\ \mathrm{MPa} + 99\ \mathrm{MPa} \cdot \mu\mathrm{m}^{1/2}/\sqrt{d} \tag{3}$$

(d in μm). The dependence of the twinning stress, Equation 2, and the dislocation glide stress, Equation 3, on the average grain size is shown on Fig. 12. The twinning stress was assessed for $s = 1$ and $h = 0.1$ μm. (The latter value is suggested by the order of magnitude of the twin width seen in Fig. 7). The diagram shows that twinning prevalent at large d is to give way to dislocation glide in the range of small d. The critical grain size at which this cross-over occurs is seen to be about 3–4 μm. Of course, this particular value depends on the magnitude of the parameters chosen and is indicative only. From the occurrence of room temperature twinning in a small grained microstructure produced by ECAE at 300°C and lack of its occurrence in the case of material pre-strained at lower temperatures, together with the data on the average grain size for these cases, it can be conjectured that the cross-over between twinning and dislocation glide occurs, indeed, at the grain size of about 3–4 μm.

The fact that the propensity for twinning decreases with increasing degree of grain refinement is also confirmed by the considerations presented by Meyers and co-workers [15, 16]. These authors represented the twinning stress of a range of metallic materials, including hcp ones, by a Hall-Petch-type relation and demonstrated that the Hall-Petch constant for twinning is larger than the corresponding constant for dislocation glide. The predominance of dislocation glide over twinning as the deformation mechanism for small grain sizes follows as a direct consequence of their analysis.

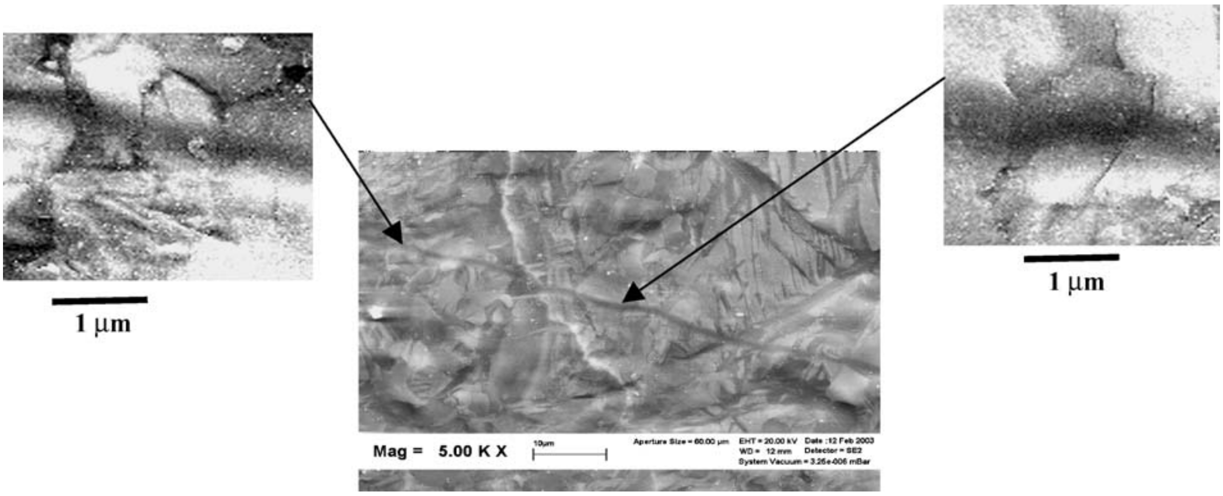


Figure 11 Trace of a scribed line after compression test.

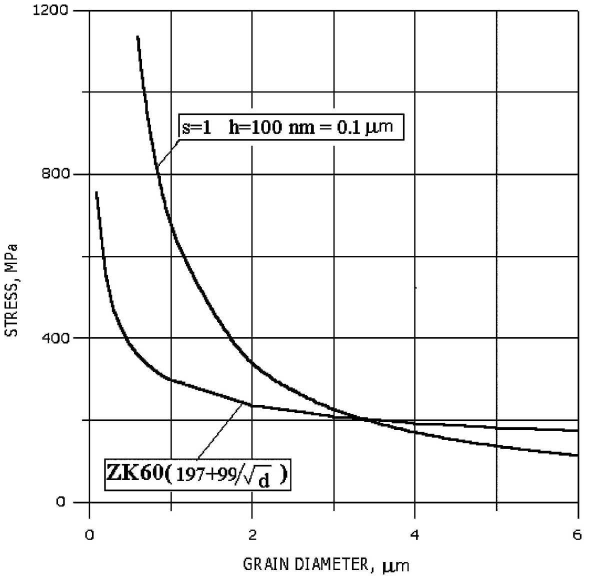


Figure 12 Grain size dependence of the twinning stress and the dislocation glide stress.

Figure 13 Micrograph showing small grains (~ 2 μm) in a sample after 8 passes of warm ECAE (300°C) followed by a single room temperature pass.

To verify the statement that no twinning occurred in the smallest grains produced by ECAE, micrographs of selected grains were taken by transmission electron microscopy, Fig. 13. No evidence of twinning was found, confirming that other deformation mechanisms were active to accommodate room temperature strain.

5. Conclusions

The experimental observations made in this work lead us to conclude that:

- Equal channel angular extrusion of alloy ZK 60 at elevated temperatures leads to a bi-modal grain structure consisting of large deformed and small recrystallised grain fractions. The overall grain refinement is owed to a decrease in the grain size of large grains and the emergence of recrystallised small grains.
- The fraction of small grains increases with the number of passes and the ECAE temperature. Thus, after 8 ECAE passes at 300°C, the grain structure is no longer bi-modal, but is rather uniform and fine-grained, deformed large grains representing a 'minority' fraction.
- Twinning occurs during the first two ECAE passes; the twin density stays constant at 200°C but decreases with subsequent passes for the other two temperatures. Together with the fact that the recrystallised grain area increases with the ECAE temperature, this observation suggests that twins are the preferred recrystallisation sites.
- Twinning under room temperature ECAE in material severely pre-strained at elevated temperatures was less pronounced than in undeformed homogenised material. The density of deformation twins produced at room temperature decreased on lowering the pre-strain temperature, owing to the fact that the smallest recrystallised grain size was produced at the lowest pre-strain temperature.
- It can be conjectured that there exists a critical grain size below which deformation twinning at room temperature is suppressed. For the material tested, this critical size should be about 3–4 μm.

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References

1. J. W. CHRISTIAN and S. MAHAJAN, *Progr. Mater. Sci.* **39** (1995) 1.
2. N. A. BOUCHER and J. W. CHRISTIAN, *Acta Met.* **20** (1972) 581.
3. R. E. REED-HILL, 'Role of Deformation Twinning in the Plastic Deformation of a Polycrystalline Anisotropic Metal", *Deformation Twinning* [Proc. Conf.], edited by R.E. Reed-Hill, J.P. Hirth and H.C. Rogers, Gainesville, Florida, 21–22 March, 1963 (Gordon and Breach, New York 1963) p. 295.
4. O. VOEHRINGER, in 2nd Internat. Conf. on Strength of Metals and Alloys (ICSMA-2) (ASM, Metals Park, Ohio, 1970) p. 294.
5. J. FRIEDEL, "Dislocations" (Pergamon, Oxford, UK, 1964) p. 178.
6. YINMIN WANG, MINGWEI CHEN, FENGHUA ZHOU and EN MA, *Nature* **419** (2002) 912.
7. M. H. YOO, *Met. Trans A* **12A** (1981) 409.
8. U. F. KOCKS and D. G. WESTLAKE, *Trans TMS–AIME* **239** (1967) 1107.
9. R. Z. VALIEV, R. K. ISLAMGALIEV and I. V. ALEXANDROV, *Progr. Mater. Sci.* **45** (2000) 103.
10. V. N. CHUVIL'DEEV, M. YU. GRYAZNOV, A. N. SYSOEV and V. I. KOPYLOV, "Low Temperature Superplasticity of Microcrystalline High Strength Magnesium Alloys Processed by ECAP", in Proceedings of 9th International Symposium

on Physics of Materials, ISPMA9, Prague 1–4 September 2003, edited by F. Chmelik and P. Lukac (to be published in Journal of Metals and Compounds).

11. S. R. AGNEW, G. M. STOICA, L. J. CHEN, T. M. LILLO, J. MACHERET and P. K. LIAW, "Equal Channel Angular Processing of Magnesium Alloys, Ultrafine Grained Materials II", edited by Y. T. Zhu, T. G. Langson et al., TMS Meeting, 2002, p. 643.
12. O. SITDIKOV, R. KAIBYSHEV and T. SAKAI, "Dynamic Recrystallisation Based on Twinning in Coarse-Grained Mg" (Materials Science Forum, Trans Tech Publications, Switzerland, 2003) Vols. 419–422, p. 521.
13. S. FERRASSE, V. M. SEGAL, S. R. KALIDINDI and F. ALFORD, *Mater. Sci. Eng. A*, **368** (2004) 28.
14. M. MABUCHI, K SHIMOJIMA, Y. YAMADA *et al.*, "High Strength and High Strain Rate Superplasticity in Magnesium Alloys" (Materials Science Forum, Trans Tech Publications, Switzerland, 2001) Vols. 357–359, p. 327.
15. M. A. MEYERS, O. VÖHRINGER and V. A. LUBARDA, *Acta Mater.* **49** (2001) 4025.
16. M. A. MEYERS, D. J. BENSON, O. VÖHRINGER, B. D. KAD, Q. XUE and H.-H. FU, *Mater. Sci. Eng.* **A322** (2002) 194.

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