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Deformation and Anelastic Recovery of Pure Magnesium and AZ31B Alloy Investigated by AE

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Extruded pure magnesium and AZ31B alloy were compressed parallel and vertical to extrusion direction. Deformation behavior was investigated in monotonous compression by acoustic emission (AE). Anelastic recovery behavior was observed in cyclic compression-quick unloading-recovery process. AE method was applied in each recovery process to investigate detwinning behavior at different strain levels. It was found that twinned samples vertically compressed easily detwin compared to parallel samples in both pure magnesium and AZ31B alloy. Compared to pure magnesium, twinning in AZ31B alloy is more stable and shows weaker psuedoelasticity. A model of strain dependence on cumulative AE counts from detwinning was proposed and fitting results are in good agreement with experimental data.
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1. Introduction

Pseudoelasticity of magnesium and its alloy in cyclically deformation process has been thought to be related to twinning-detwinning conventionally.[1)] During cyclic deformation process, the anelastic recovery with non-linear behavior in unloading will lead to hysteresis loops. The pseudoelasticity in magnesium is very similar to the stress induced martensitic transformation (MT) process which is also due to twinning and detwinning in loading and unloading processes, respectively.[2)] Detwinning is the shrinking or narrowing of twinning as applied stress is retreated or lowered. A lot of researches about cyclic transition behavior of MT by AE have been conducted widely, because both twinning and detwinning generate strong AE signals and dynamic internal structure evolution can be analyzed effectively by AE.[3)]

Due to hcp structure, extruded magnesium and its alloy demonstrate different mechanical responses when deformed along different direction, which then inevitably impose strong effect on psuedoelasticity of extruded materials. In our previous research,[4)] the psuedoelasticity of pure magnesium was investigated by AE. Anelastic recovery strain, ε_r, the strain difference before and after the anelastic recovery process, was directly obtained by unloading with very high speed. It has been found that the detwinning in anelastic recovery process was closely related to twinning process of previous history. In the present research, the basal plane textured pure magnesium and AZ31B alloy are compressed parallel and vertical to the extrusion direction. Twinning behaviors in both pure magnesium and AZ31B alloy are compared and analyzed in detail. A model for relationship between applied strain and cumulative AE counts from detwinning is proposed.

2. Experimental Procedures

Extruded pure magnesium and AZ31B alloy produced by Osaka Fuji Corp. were selected as present research materials. Samples were with grain size of about 30–50 µm for pure magnesium, and 25–40 µm for AZ31B alloy, respectively. Microstructure was observed by polarized optical microscope. Specimens were machined into cylindrical size of ϕ15 mm × 15 mm and then compressed parallel and vertical to the extrusion direction.

Samples in compression were conducted at a strain rate of about $1.67 \times 10^{-4}\,s^{-1}$. Cyclic compression was performed from strain level of about 0.1% to about 6%. Then, the specimen was unloaded with a speed of about $0.56\,s^{-1}$ and recovered for about 60 min at each strain level, in which AE measurement was applied to investigate detwinning behavior in anelastic recovery process.

In the present research, a low noise type AE sensor was (M304A, Fuji Ceramics, Japan) used in experiments and closely contacted to the sample surface by polymer flocculant in a special compression jig as shown in Fig. 1. Used AE system was µDISP (PAC, USA) with a threshold of 55 dB in compression and 40 dB in recovery as well as a high pass filter of 100 kHz effectively getting rid of noises from

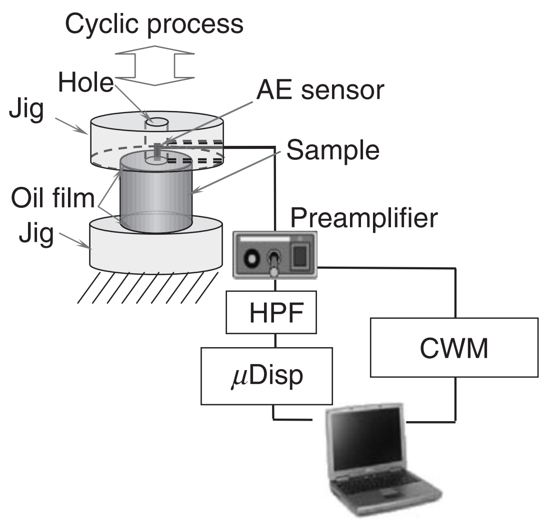

Fig. 1 Schematic figure of experiment. AE sensor is directly placed on the sample surface. CWM AE system is used to obtain STFT of AE signals and µDisp AE system is used to obtain AE RMS and AE count.

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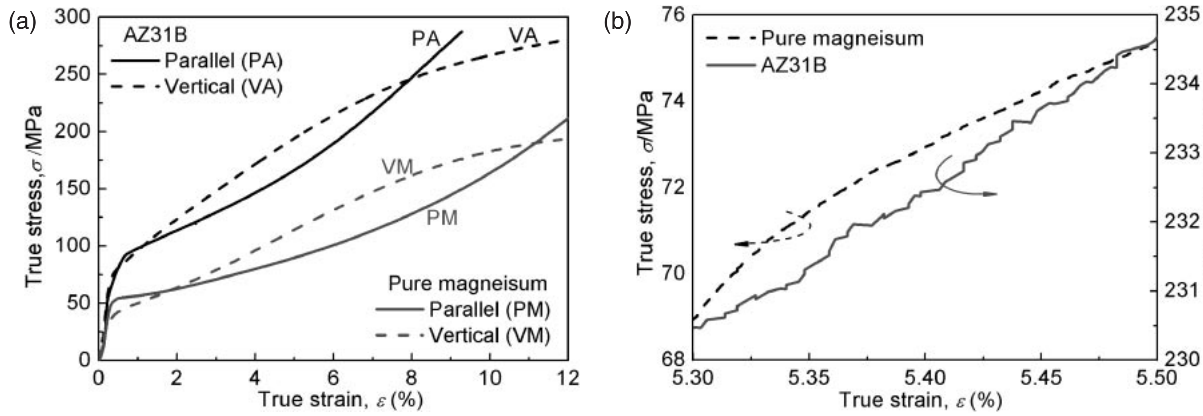

Fig. 2 (a) Deformation curves of the four kinds of samples and (b) the details of deformation curves for parallel samples of pure magnesium and AZ31B alloy in the strain rage of 5.30 to 5.50%.

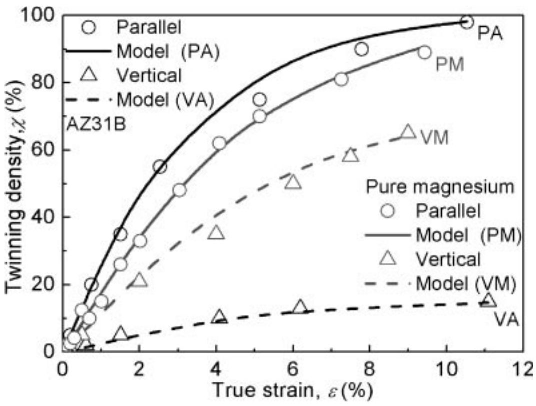

Fig. 3 Strain dependences of twinning area fraction, χ, for the four kinds of samples.

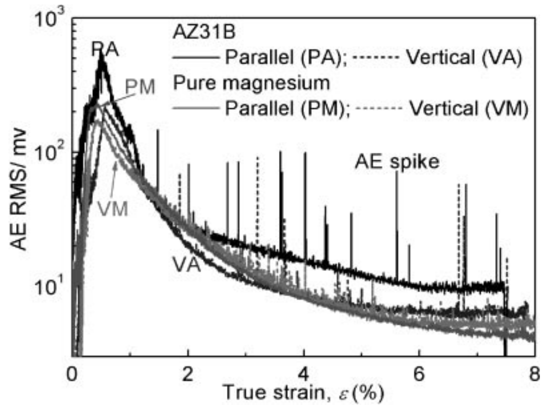

Fig. 4 Strain dependence of RMS voltage of AE signals of the four kinds of samples. Strong AE spikes appeared in the samples of AZ31B alloy above strain of about 2.0%.

environments. The frequency distributions of AE signals as a function of time (Short time Fourier transform, STFT) in deformation will be observed by Continuous Wave Memory (CWM) AE system.[5)]

3. Results and Discussion

3.1 Deformation behavior

Typical deformation curves compressed parallel and vertical to extrusion direction are shown in Fig. 2. Compression along extrusion direction exhibits lower initial hardening rate than vertical samples in both pure magnesium and AZ31B alloy, because $\{10\bar{1}2\}$ twinning system is easily facilitated in parallel samples. However, after strain of approximately 4–5%, the strain hardening rate increases rapidly in parallel samples because of exhaustion of twinning. The extruded pure magnesium and its alloy exhibit strong basal texture, where majority of c-axes are aligned normal to extrusion direction. Present in-plane compressive stress along extrusion direction results in twinning, while vertical samples with contracting alone c-axes are relatively difficult to form twinning, although lower twinning density is observed in the vertical samples (Fig. 3). There are three explanations that can be offered for higher hardening rate in parallel samples at higher strain levels: Twinning introduces additional barriers to dislocation movements,[6,7)] which is in some extent equivalent to reducing the grain size. Secondly, $\{10\bar{1}2\}$ twinning in magnesium reorients the lattice to hard crystallographic orientations,[8)] and the other twinning systems or non-basal slipping systems with higher critical resolved shear stress (CRSS)[1)] have to be activated. Finally, the transition of dislocations as they pass through twinning front can lead to high hardening rate within the twinning interior.[9)]

The twinning densities as a function of true strain are shown in Fig. 3. The fraction of twinning area is followed by the sigmoidal equation[10)] for the parallel samples,

$$\chi = 1 - \exp[-\gamma(\varepsilon/\varepsilon_0)^{\beta}] \tag{1}$$

where the value of ε_0 is theoretical strain when twinning area fraction is unity, and β is the rate exponent. The value of ε_0 is necessarily greater than the true strain ε. The nonlinear fit results by selecting appropriate value of β, ε_0 and γ show a good agreement with the experimental data. In vertical samples, a factor χ_0 should be added to eq. (1) since the maximal value of twinning density is lower than unit,[10)]

$$\chi = \chi_0\{1 - \exp[-\gamma(\varepsilon/\varepsilon_0)^{\beta}]\}. \tag{2}$$

The root-mean-square (RMS) voltage of AE signals in compression process of above four samples is shown in Fig. 4. Similar changing trends are observed in all samples: RMS voltage of AE signals reaches peak values near the yield point and then decreases gradually with increasing

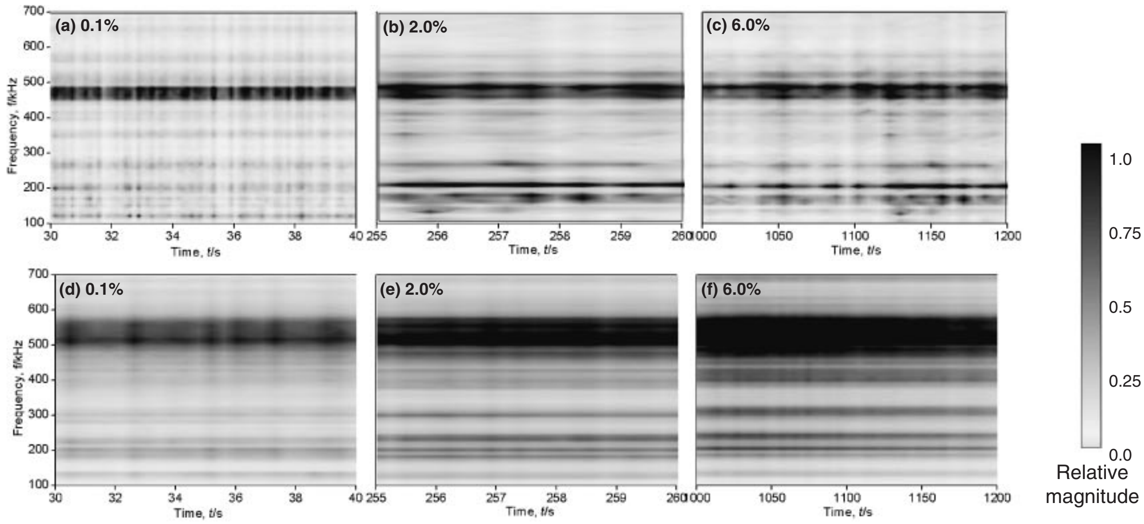

Fig. 5 STFT of AE signals of the parallel samples at strain of 0.1, 2.0 and 6.0%, (a), (b) and (c) for pure magnesium and (d), (e) and (f) for AZ31B alloy.

strain level. In the initial stage of deformation process (strain <2%), the AE signals for all parallel samples are stronger than corresponding vertical ones, because twinning is easily activated in parallel samples and especially twinning nucleation can produce strong AE signals.[3)] AE signals in vertical sample are relatively weak compared to the parallel sample, because twinning activity in vertical sample is relatively low. At high strain level (strain>2.0%), no strong AE spike could be observed in deformation of pure magnesium, while strong AE spikes are observed in both parallel and vertical samples of AZ31B alloy. In present research, the grain size in both pure magnesium and AZ31B alloy is nearly in the same order (average grain size, 30 μm for AZ31B alloy, 35 μm for pure magnesium). The twinning behaviors in both kinds of samples are thought to be similar. So there must be a mechanism not existing in pure magnesium which induces the strong AE spikes in AZ31B alloy. In facts, deformation of magnesium alloys accompanied with AE spikes have been reported previously.[11–15)] Such AE spikes are thought to be correlated with the individual regions of plastic instabilities inside of the sample, that is, PLC (Portevin-Le Chatelier) effect. The PLC effect is originally observed in the serrated flow curves of materials. Serrations result from the dynamic interaction of solute atoms with mobile dislocations temporarily arrested at forest dislocations.[12–15)] The forest dislocations in magnesium are dislocations in non-basal slip systems. The ratio of the critical shear stress for prismatic slip to that of basal slip depends on concentration of solute. In the case of the Mg–Al-Zn system, this ratio decreases strongly with increasing concentration of Zn solute atoms. It is speculated that the presence of Zn atoms leads to an increased density of forest dislocations. The role of Zn, on the other hand, is to enhance prismatic slip, enabling the rapid development of forest dislocations.[11)] The Al atoms in solid solution, due to their larger concentration, determine the dynamics of pile up formation, and make the alloy susceptible of PLC effect. As shown in Fig. 2(b), the deformation curve of pure magnesium shows a relative smooth one compared with the AZ31B alloy. The vibration of the stress at high strain level is reasonably thought to be related to the above PLC effects. There is a high possibility of that the AE spikes are related to such PLC effect closely.

Short time Fourier transitions (STFT) of AE signals at strains of 0.1, 2.0 and 6.0% for parallel samples of both pure magnesium and AZ31B alloy are shown in Fig. 5. The horizontal axis, vertical axis and the contrast of the legend are time, frequency and the relative magnitude of AE signals, respectively. Only one frequency peak of about 500 kHz is clearly observed (Fig. 5(a) and (d)) at strain of about 0.1% in pure magnesium and AZ31B alloy. At strain of about 2.0 and 6.0%, other frequency peaks with lower values are also observed in both samples (Figs. 5(b), (c), (e) and (f)). The signals with lower frequency are thought to be related to dislocation slipping, while the higher frequency peak of about 500 kHz is related to twinning formation process.[4)] It is well known that frequency of AE signal is determined by the event rise time of AE sources.[3)] The formation of twinning with relatively higher CRSS is accompanied with a large decrease of stress and the formation velocity can exceed transverse velocity of sound,[16)] while the slip velocity of dislocation is only about 0.3–0.4 times of the transversal sound velocity.[17)] It is thought that both the dislocation slip distance and twinning nucleation distance are the nearly same and in the order of grain size.[17,18)] The frequency for twinning nucleation is expected to be 2.5–3 times of that for slip. The above characteristics in the formation of twinning and slip results in longer event rise time or lower frequency for dislocation slip and the short event rise time or higher frequency for twinning nucleation. It has to be mentioned that, twinning growth and twinning nucleation are all jumpwise formation processes and supposed to have same frequency peak, although the amplitude of AE signals in twinning nucleation is much stronger than that from the twinning growth for the higher CRSS in the twinning nucleation process.[19)] From Fig. 5, the following conclusion may be obtained: in the initial deformation of parallel samples, the deformation is mainly realized by the twinning formation because only one twinning frequency peak around

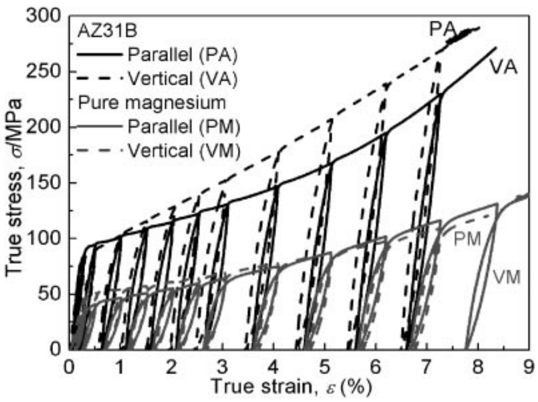

Fig. 6 Cyclic compression curves for pure magnesium and AZ31B alloy of vertical and parallel samples. Clear hysteresis loops are observed in all the samples.

500 kHz is observed, while at higher strain level, the deformation is realized by both dislocation motion and twinning. However, it has to be mentioned that, a little difference between pure magnesium and AZ31B alloy could be observed: a frequency peak of about 400 kHz, between the twinning peak and dislocation slip peak, although not so obvious, is observed in AZ31B alloy. This frequency peak is a unique characteristic of AZ31B alloy in the deformation process compared to pure magnesium at high strain level. This frequency peak is thought to be related to the PLC effect of AZ31B alloy as mentioned before. It has been described in the above that the PLC effects is due to the interaction between the solute atoms and the mobile dislocations. The transverse distance of this distance is thought to be shorter than that of the general dislocation slip. Then, the frequency from PLC effect is reasonably to have high frequency components. However, the PLC effect is also a result of dislocation slip. It is speculated that the frequency peaks with lower values are also related to this effects more or less.

3.2 Anelastic recovery behavior

The cyclic compression curves of pure magnesium and AZ31B alloy for both parallel and vertical samples are shown in Fig. 6. Detwinning in unloading or the anelastic recovery results in broad hysteresis loops when the samples were reloaded again. However, no quantitative information could be obtained by just analyzing these hysteresis loops. In the present research, the unloading speed was selected at very high order of about $0.67\,s^{-1}$. The anelastic recovery process lags behind elastic recovery, and the anelastic recovery process can be analyzed independently (Fig. 7(a)). The strain was selected from low level to higher level cyclically, and the samples were recovered for about 60 min in each recovery process as shown in Fig. 7(a). The AE behaviors and strain recovery behavior in anelastic recovery process of pure magnesium and its alloys have been discussed in other research previously.[4] Emitted cumulative AE counts N and anelastic recovery strain ε_r are obtained as shown in Fig. 7(b) and (c) for pure magnesium and AZ31B alloy after recovered for 60 min at different strain level, respectively.

For pure magnesium, ε_r increases greatly with increasing strain level before strain of about 2.0%, however, the increasing rate decreases later as if the recovery process was interrupted by some factors not favoring the recovery process. N released in each recovery process grows greatly with the increase of strain. However, after strain of about 2.0%, the cumulative AE counts decreases gradually. The different changing behaviors of these two parameters show that anelastic recovery includes some mechanisms other than AE event formation. The AE signals in anelastic recovery process are reasonably thought to be due to detwinning process, because the detwinning process is a very important source of AE and the anelastic recovery is related to detwinning closely.[19] The anelastic recovery strain is due to at least two mechanisms, detwinning and reversal motion of dislocations where the recovery by dislocation motions is a thermal activated process[20] and the elastic energy released is

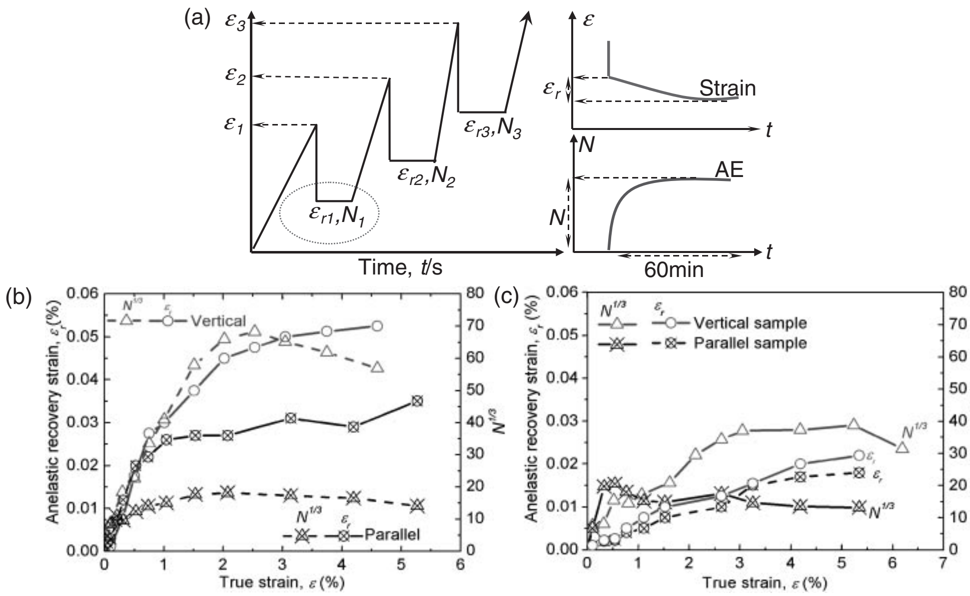

Fig. 7 (a) Schematic figure for the cyclic compression process, and the strain dependence of anelastic recovery strain, cumulative AE counts for (b) pure magnesium and (c) AZ31B alloy.

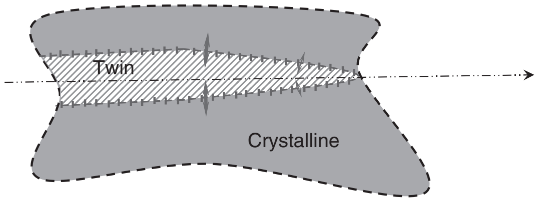

Fig. 8 Schematic figure of the twinning interface structure in which edge dislocations arranged with Burgers vector vertical to the interface can slip normal to the twin interface.[22]

too weak to be detected by present AE system. In AZ31B alloy, a similar changing behavior of these two parameters is observed as in pure magnesium. However, values of both ε_r and N in AZ31B alloy are lower, which shows a lower trend of anelastic recovery in AZ31B alloy. Because the grain size between pure magnesium and AZ31B alloy is in the same order, the high stacking fault energy (SFE) (about 200 mJ/m^2) in pure magnesium[14] is thought to be an important factor to make the twinning unstable and easy to shrink compared with AZ31B with low SFE (27–30 mJ/m^2)[19] for the addition of alloying elements.

It is interesting to observe that both N and ε_r in the vertical samples are higher than that from parallel samples for both pure magnesium and AZ31B alloy (Fig. 7). Detwinning is thought to be closely related to the number of unstable twinning before recovery. In the parallel samples the twinning is much easy to form and the stability of twinning is very high. In the vertical samples, twinning is difficult to form, and the formed twinning is fine and unstable and prone to detwin when applied stress is retreated or lowered.[20] Fine grain size induces easy shrink of twinning.[21] So, the psuedoelasticity in vertical samples is more obvious than that of the parallel samples in both pure magnesium and AZ31B alloy.

3.3 Model for detwinning

During the deformation process, very high stress (stress concentration) was generated in immediate vicinity of twinning, and these stresses and the associated strain energy arise from the resistance of matrix to macroscopic change of shape in twinned volume. When the applied stress is retreated or decreased, internal stresses inside of the twinning would result in an opposite effect of deformation by detwinning. The dislocations of twinning interface with the burgers vector normal to the interface are thought to be a critical prerequisite because the dislocations inside of the interface are edge type which can move parallel to their burgers vectors as schematically shown in Fig. 8.[20]

The cubic root of cumulative AE counts $N^{1/3}$ in the anelastic recovery process is proportional to anelastic recovery strain by detwinning as described in previous research.[4] $N^{1/3}$ is thought to be related to the following three parameters in a specific sample when compressed from a previous strain level ε_1 to next strain level ε_2 in the cyclic compression process: the internal stress σ_i which is the driving force of detwinning, supposed to be proportional to the applied stress σ_a, the increment of twinning area fraction from a previous cycle to next cycle $(\chi_2 - \chi_1)$, which is thought to be proportional to the anelastic recovery strain by detwinning on the basis of that the detwinning is a reversal direction of the twinning process,[22] as well as the fraction of the twinning strain rate before recovery, $(\frac{d\chi}{d\varepsilon})_{\chi=\chi_2}$, which is thought to be related to the initial detwinning rate because the newly formed twinning is in the most unstable state.[11,22] Then the true strain dependence of $N^{1/3}$ can be expressed by

$$(N^{1/3})_{\chi=\chi_2} = V\sigma_a(\chi_2 - \chi_1)\left(\frac{d\chi}{d\varepsilon}\right)_{\chi=\chi_2}, \qquad (3)$$

where V is a constant related to properties of AE sensor and samples such as stacking fault energy (SFE) and the volume of the sample etc.[3] The twinning area fraction in the cyclic compression process follows to the eqs. (1) and (2). The calculated results from eq. (3) demonstrate a good agreement with the experimental data as shown in Fig. 9.

4. Conclusions

(1) The textured pure magnesium and AZ31B alloy compressed parallel and vertical to the extrusion direction, exhibited different deformation behaviors. It was found that the deformation behavior is closely related to twinning.
(2) The STFT of AE signals of both parallel pure magnesium and AZ31B alloy showed that AE signals are mainly due to the twinning formation process in the initial stage of compression process. Strong AE spikes were observed at high strain level in AZ31B alloy due to PLC effect.

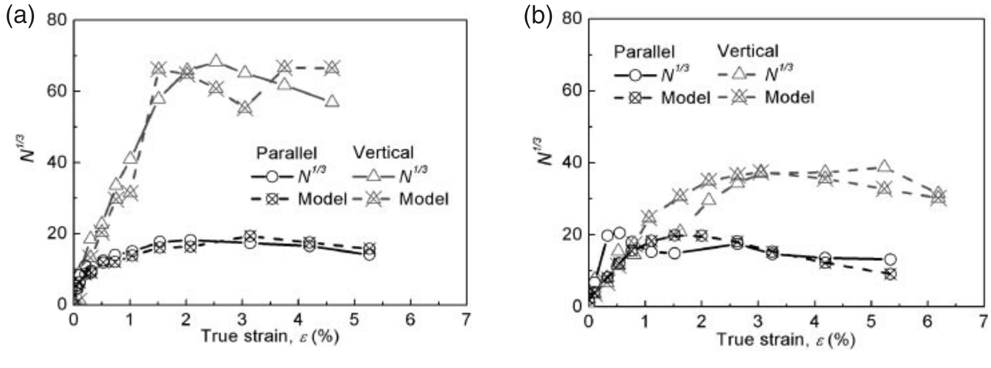

Fig. 9 Both experimental results and the calculated ones from the model for (a) pure magnesium and (b) AZ31B alloy.

(3) The detwinning AE counts in anelastic recovery process of vertical samples were found to be much more than that from the parallel samples. It was supposed that twinning in vertical sample was unstable and easier to detwin compared to parallel samples in both pure magnesium and AZ31B alloy.
(4) Stronger trend of the psuedoelasticity in pure magnesium was found than that of the AZ31B alloy in both parallel and vertical samples.
(5) A model for relationship between the applied strain and the detwinning cumulative AE counts was proposed, and the fitting results gave well agreement with the experimental data.

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