Recovery Behaviour of Pure Magnesium in Cyclic Compression–Quick Unloading-Recovery Process at Room Temperature Investigated by AE

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Anelastic recovery of pure magnesium at room temperature was investigated in cyclic compression-quick unloading-recovery process where acoustic emission (AE) measurement was applied to analyze the dynamic behaviour and mechanism of anelastic recovery process. By analyzing the RMS voltage of AE signals from both the background and the recovery process, it was observed that the recovery process was accompanied with a gradual decrease in the strength of AE signals. The AE signals in recovery processes of different strain levels seem to be due to the same source of detwinning process because the same slope between amplitude and logarithmic AE count of AE signals in different strain levels was found in the strong elastic waves related to detwinning process. The AE behaviors in recovery process were described in details by AE count rate and AE incubation time. The relations between twinning or detwinning and AE counts in both deformation and anelastic recovery process could be expressed by a general equation. [doi:10.2320/matertrans.MC200705]

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1. Introduction

Anelastic recovery of magnesium and its alloys has been thought to be resulted from the detwinning process conventionally when the external stress is retreated or lowered.1–5) The results of Gharghouri et al.1) showed that the hysteresis loops of pure magnesium in cyclic tension and compression are due to {1012} twinning which grows when the materials are stressed and partially reverts when unloaded. The pseudoelasticity in magnesium and its alloys is very similar to the stress induced martensitic transition (MT) in which there is also twinning and detwinning in loading and unloading processes, respectively.5) Lots of researches about the cyclic transition behaviour of MT by AE technique have been conducted because the twinning and detwinning processes are strong AE sources and the dynamic internal structure evolution can be analyzed effectively by AE.5) Magnesium demonstrates different behaviour and mechanism in loading and the following unloading process compared with the other materials without the pseudoelasticity. The fatigue and energy absorption, when magnesium or its alloys are used as the damping structure, are also affected by such cyclic movements of twinning boundaries. Then, precisely understanding the dynamic behaviour and mechanism of the anelastic recovery is an important topic for both engineers and researchers.

Traditionally, pseudoelasticity is analyzed in hysteresis loops. The anelastic recovery strain \( \varepsilon_r \), the strain difference before and after the anelastic recovery, can be determined from the hysteresis loops by using the nominal Young’s modulus.7) However, this method will result in a large error because it was by an indirect measuring method. Besides, the unloading curve of magnesium is composed of both the elastic and anelastic unloading curves which are not separated automatically, and it is difficult to analyze the anelastic recovery behaviour and mechanism independently.6,7)

In the previous research,8,9) the anelastic recovery behaviour of magnesium was obtained by unloading the specimen with relatively high speed so that anelastic recovery lags behind elastic recovery and were analyzed independently. AE measurement was applied to investigate the behavior of anelastic recovery process. The main objective of the present research is to get the dynamic behaviours and mechanism of anelastic recovery in more details in terms of AE measurements. The relationship between recovery by detwinning and deformation by twinning will be discussed for the first time.

2. Experimental

Commercial extruded pure magnesium without heat treatment with purity of 99.95% was selected as the present research materials. Samples are with average grain size of about 35 \( \mu m \) as shown in Fig. 1. Microstructure was observed by optical microscope at the center of the side surface in samples after polishing and etching by nitric acid solution. Cylindrical specimens were machined into size of \( \phi 15 \times 15 \) mm.

![Fig. 1 Microstructure of the pure magnesium in present research.](image-url)
Cyclic compression was performed from the strain level of about 0.1% to about 5% along the extrusion direction. The deformation rate was in an intermediate level of $1 \times 6.7^{10}/C_2^{4}/s$. The specimen was unloaded with a speed of about 0.56/s in order to get a clear separation of anelastic recovery and elastic recovery. AE system used was μDISP (PAC USA) with a threshold of 40 dB and high pass filter (HPF) of 100 kHz. AE sensor was a low noise type (M304A, Fuji Ceramics, Japan) and closely contacted to the sample surface by polymer flocculant in a specially designed compression jig as shown in Fig. 2. The voltage of AE signals, AE count as well as the cumulative AE counts will be used in describing the recovery behavior. The analysis in the relation between recovery and deformation was partially based on the results in previous researches.8,10

3. Results and Discussion

3.1 Cyclic compression curve

Figure 3(a) shows the monotonous compression and cyclic compression-quick unloading-recovery curves. In each cycle, the stress was quickly unloaded to zero and then recovered for about 60 min, which leads to broad hysteresis loops with clear separation of elastic recovery and anelastic recovery (Fig. 3(b)). After recovery, the decrease of peak stress can be observed. The stress decrease is reasonably thought to be due to the recovery in which the internal stress was relaxed. However, the stress will increase greatly as the strain exceeds the previous strain level before unloading and continues to keep the entire shape similar to the monotonous one. The yielding stress in present research showed to be a little lower than the previous results9) because in present research, the grain size of the sample was observed to be a little larger than the previous one. However, the entire deformation curves in these two kinds of samples were very similar. Our previous research8) and other report11) showed that the hysteresis loops during cyclic loading of magnesium and its alloys is due to detwinning process during unloading stage in which the elastic twins disappeared as soon as the applied stress was lowered to a certain level. Reed-Hill et al.11) also observed the similar pseudoelastic behaviour of Zr when compressed at 77 k while unloading at room temperature, pointing out that such psuedoelasticity of Zr can be explained on the assumption that there are stress-induced movements of [1012] twinning boundaries which result in the loading-unloading hysteresis loops. Recently, C. H. Caceres et al.7) reported the similar pseudoelastic behaviour of cast AZ91 magnesium alloy under cyclic loading-unloading process, by an in-situ observation of the surface of the

Fig. 2 Experimental setup for cyclic compression- quick unloading-recovery process and AE measurement.

Fig. 3 (a) Cyclic and monotonous compression curves, and (b) the details of the typical hysteresis loops after quick unloading.
specimen. Present hysteresis loops formed in cyclic process are thought to be in good agreement with the previous results that twinning–detwinning process is related to the hysteresis loops closely. Some of twins partially reverted when applied load was slightly decreased. Twins may become either slightly narrower or shorter with the decrease of applied load. In case of very thin twins, complete reversal seems to occur, but upon reloading, the twin reappeared on the same location. Present results of magnesium were in good agreement with previous results. However, it has to be noted that we observed near linear unloading curve when high unloading rate was applied, which resulted in an elastic recovery stage followed by the anelastic recovery. Present result in anelastic recovery was thought to be somewhat not consistent with the previous explanations1,11,12) that detwinning process occurred simultaneously in the unloading process because the detwinning process lagged behind significantly at high unloading rate in present research.

3.2 AE signals in anelastic recovery process

Figure 4 shows the amplified root mean square (RMS) voltage of AE signals during the recovery stage at strain level of about 4.26% as well as the signals of background. The AE signal strength of recovery decreases to a noise level eventually with increasing time, showing a similar behaviour to the traditional recovery process of some properties at elevated temperature. This result indicates that present AE system is an effective method in investigating the recovery of magnesium in a specific time range. The recovery time dependence of cumulative AE counts \( N \) at a threshold of 40 dB when the sample was recovered for about 60 min at different strain levels are shown in Fig. 5. All AE behaviours in anelastic recovery processes show similar trends in which the AE count rate was highest at the initial stage of the recovery process. The AE behaviours are in some extent similar to the strain recovery behaviour that recovery process begins with highest rate and slows down rapidly with the increase of time.

Figure 6 shows the distribution of AE event count as a function of signal amplitude in the anelastic recovery process at two strain levels. In both graphs, linear behaviours between \( \log(N_e) \) and AE amplitude are observed. From M. Ohtsu13) such kind of linear distribution behaviour between AE amplitude and event count generally demonstrate the single AE generation source. The slopes in the two graphs are observed to be nearly in the same value, showing the same source of AE in the anelastic recovery process at different strain levels.

![Figure 4](image1.png)  
Fig. 4 RMS voltage of AE signals in recovery process (at strain 4.26%) and the noise level in room temperature.

It was supposed that very high stress (stress concentration) was generated during the deformation process in immediate vicinity of deformation twins. These stresses and the associated strain energy arising from the resistance of matrix to the macroscopic change of shape in the twinned volume. When the applied stress is retreated or decreased, internal stresses around the twins would result in an opposite effect of the deformation by detwinning at least partially. Elastic twins should disappear spontaneously or in a very short period after unloading, however some “true twins” in some favorable condition (such as in a position with very high anti-resolved shear stress) should also shrink continuously when the applied stress is decreased or retreated. The detwinning of the “true twins” should proceed in a competitive process of the driving force such as the stress concentration or high strain energy with the resistances from the vicinity such as the dislocations around the interface of the twins, the defects of the matrix etc. With the increase of time, the relaxation of matrix by the annihilation of dislocations, ordering of dislocation configurations or disappearance of point defects will result in a favorable condition for the detwinning process. As described above, the anelastic recovery of pure magnesium was due to (at least partially) the detwinning process. It can be naturally concerned to the martensitic transition in which AE signals were believed to be from the detwinning process. As described by O. A. Bartenev et al.,14) disappearance of twinning in martensitic transition can produce much stronger AE signals than that from the dislocation annihilation or slipping. In present condition, such strong AE signal after deformation can be reasonably ascribed to the detwinning process.

3.3 Relation between AE and anelastic recovery process

Strain recovery curve in Fig. 5 can be accurately described by
\[
\varepsilon = \frac{RT}{\alpha} \ln(t) + \frac{RT}{\alpha} \ln(2\tau) = A \ln(t) + B
\]

where \( \varepsilon \) is the anelastic strain, \( R \) the universal gas constant, \( t \) the recovery time, \( \tau \) the theoretical relaxation time from present strain to zero, and \( A \) and \( B \) constants. The recovery curve at strain of 4.26% drawn in Fig. 7 is shown to be in good agreement to eq. (1) where \( A = -\frac{RT}{\alpha} = -4.68 \times 10^{-3} \), and \( B = \frac{RT}{\alpha} \ln(2\tau) = 3.83 \) are selected. Equation (1) is a traditional form of the recovery in the properties of materials at high temperature in case of the thermal activated process. In present research, the anelastic recovery process of detwinning is thought to be also a thermal activated process if considering the process in the whole aspect, because the detwinning process is driven by the internal stress in which recovery behavior of internal stress at a certain temperature is well known to be a thermal activated process.

The applied strain dependences of the anelastic recovery strain \( \varepsilon \) and the corresponding cumulative AE counts \( N \) after recovered for 60 min in each cycle are described in refer. \(^9\) Before the strain of about 2.0%, \( \varepsilon \) increases greatly with the increase of strain level while the increasing rate decreases in the later stage as if the recovery process was interrupted by some factors not favoring in 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 behaviours of these two parameters show that the mechanisms of anelastic recovery and the AE event formation mechanism must be different. The AE signals in the anelastic recovery process are reasonably thought to be due to the detwinning process, because the detwinning process is a very important source of AE and the anelastic recovery of pure magnesium is related to detwinning closely.\(^{15}\) It is thought that the anelastic recovery strain is due to at least two mechanisms, detwinning and the annihilations of dislocations where the annihilations of dislocations is a thermal activated process\(^{15}\) and the elastic energy released is too weak to be detected by present AE system. From above explanation, it can be known that the anelastic recovery by detwinning and the overall anelastic recovery process (both detwinning and dislocation annihilation) are different. At lower strain level (lower than about 1.0%), the recovery from detwinning and the overall anelastic recovery have a similar behaviour. In the later stage, due to the decrease of the fraction of anelastic recovery by detwinning, the increasing rate of overall anelastic recovery strain decreased accordingly. The exact changing behaviour of the anelastic recovery from detwinning or dislocation will be discussed in another paper.\(^{16}\)

The AE signals accompanied with the anelastic recovery process was described in details as mentioned above. Then, what is the relationship between AE signal and anelastic recovery strain? Figure 8 shows the relationship of overall AE counts and the anelastic recovery strain before the strain level of 1.0% by the red circles and the red line. A nonlinear fit to the experimental data was applied and a cubic relation was observed between these two parameters. This result can be explained by the cubic relation between the anelastic strain from detwinning and the detwinning volume, and the cumulative AE counts \( N \) is proportional to the energy released in detwinning which is proportional to the volume of the detwinning. According to this theory, we can obtain an equation showing the behaviour of \( N \) as a function of the recovery time on the basis of that the relation between AE

Fig. 7 Strain recovery behaviour after quickly unloading at strain of 4.26%.

Fig. 8 Relationship of overall AE counts and the anelastic recovery strain before the strain level of 1.0%.
and anelastic recovery strain is fixed as a function of recovery time,

\[ N \propto \varepsilon_t^3 \]  \hspace{1cm} (2)

and

\[ \varepsilon_t = \varepsilon_0 - \varepsilon_f \]  \hspace{1cm} (3)

where \( \varepsilon_0 \) is the initial strain immediately after the elastic recovery or the theoretical starting recovery point where the AE signal is started to release. \( \varepsilon_f \) is the strain at time \( t \). It has to be mentioned that, physical meaning of eq. (2) should express the direct relation between the anelastic recovery strain from detwinning and \( N \) even at higher strain level because the AE signals are only related to the detwinning process as mentioned above while the anelastic recovery includes both information of recovery from detwinning and dislocation annihilation.

Inserting eq. (1) into eq. (2) and eq. (3) based on the assumption of that the relation between \( \varepsilon_t \) and \( N \) does not vary with the increase of recovery time, and \( \varepsilon_t \) is proportional to the anelastic recovery strain from detwinning all the time.

\[ N = K \left( \frac{\varepsilon_0 + RT}{\alpha} \ln(t) - \frac{RT}{\alpha} \ln(2\tau) \right)^3 \]  \hspace{1cm} (4)

In present situation, \( T \) is room temperature, \( \alpha \) a constant,

\[ N = K(\varepsilon_0 + A \ln(t) - B)^3 \]  \hspace{1cm} (5)

Equation (5) is the general AE behaviour during anelastic recovery of pure magnesium. Given appropriate values of \( K, \varepsilon_0, \) the fitting result shows a very good agreement with the experimental result as shown in Fig. 9 for the recovery process at strain level of 4.26%. Then, we can deduce an equation from eq. (5) directly by applying differential processing by time \( t \),

\[ \frac{dN}{dt} = \frac{V(\varepsilon_0 + A \ln(t) - B)^2}{t} \]  \hspace{1cm} (6)

where \( V \) is equal to 3 K/\( A \) with a value of about 5.90 \( \times 10^4 \), other parameters are same to that in Fig. 10(a). Equation (6) shows the behaviour of AE count rate in the anelastic recovery process. The decreasing rate of AE count rate shows a decrease of detwinning rate with the increase of recovery time.

With the increase of recovery time, the interval time range between the two neighboring AE events or detwinning steps will increase accordingly. This is because that the internal stress decreases gradually with the increase of recovery time, and the detwinning process has to need longer incubation time or the time for storing enough energy to activate the detwinning. Supposed that all the AE signals have the same count per event with a value of \( F \), and then the incubation time \( I \) can be obtained from eq. (4).

\[ I = \frac{F}{\frac{dN}{dt}} = \frac{F}{V} \times \frac{(\varepsilon_0 + A \ln(t) - B)^2}{t} \]  \hspace{1cm} (7)

where \( I \) is the incubation time of the AE signals in recovery process, and \( F \) the average AE count number per event in the entire recovery process. In present situation \( F \) is about 4.55 obtained from the experimental data. A plotting of eq. (7) shows a good agreement with the experimental incubation time as shown in Fig. 10(b).

In present research, the anelastic recovery process at room temperature described by the AE count rate and the AE incubation time. It has to be noted that these two parameters are different to the traditional parameters of the materials properties such as the grain size, and yield stress etc because the AE count rate and AE incubation time can dynamically and directly express the internal evolution behavior of materials in recovery process.

3.4 Consistency between deformation and recovery

Twinning plays an important role in the deformation process of pure magnesium as described in our previous research.\(^{[10]}\) For a given grain orientation, \( \varepsilon_{tw} \) is the strain accommodated by twinning that is thought to be related to the characteristic shear of twinning system \( S \), Schmid factor \( m \), which is supposed to be decreased with increasing strain \( \varepsilon \), as well as the volume fraction of twinned grain \( \nu \)\(^{[17]}\) by

\[ \varepsilon_{tw} = m\varepsilon S = m\varepsilon M_{t} \]  \hspace{1cm} (8)

\[ m = M_{s} \varepsilon, \quad M = 1 - C\varepsilon \]  \hspace{1cm} (9)
It is found that the relation between cumulative AE counts and the twinning strain can be expressed by following equation in the initial stage of deformation process:

\[ N^{1/P} = k \varepsilon_{tw} \]  

where \( P \) is an exponent constant with a value of about 1.26 ± 0.02 in all cases, and \( k \) is varied according to experimental condition. The relation between \( N \) and \( \varepsilon_t \) in each recovery process before the applied strain level of 1.0% for both vertical and parallel samples is also plotted in Fig. 8.

It is interesting to find similar expressions between eqs. (10) and (2) in both deformation and anelastic recovery process. Equation (10) expresses the relation between AE and twinning behavior in deformation process, and eq. (2) shows the relation between AE and detwinning behavior. The two processes with contrary direction are reasonably thought to have similar equation. The relation between \( N \) and \( \varepsilon_{tw} \) cannot be expressed by eq. (10) any more with increasing strain level because the AE signals emitted from twinning nucleation are much more than that from the twinning growth and the twinning changes from twinning nucleation process to twinning growth with increasing strain level in deformation process. For the anelastic recovery process, at higher strain level, the decrease of \( N \) and the increase of \( \varepsilon_t \) show that fraction of anelastic recovery strain from detwinning decreases accordingly. The relation between \( N \) and \( \varepsilon_t \) does not follow eq. (2) any more at higher strain level. From present result in both deformation and anelastic recovery, a good consistence between deformation and recovery in the relation between AE and the corresponding twinning (detwinning) is established.

4. Conclusions

Anelastic recovery of pure magnesium in cyclic compression–quick unloading-recovery process was investigated in detail by AE measurements and the results obtained are as follows:

(1) By analyzing the RMS voltage of AE signals from both the background and the recovery process, it was observed that the recovery process of pure magnesium was accompanied with a gradual decrease in the strength of AE signals.

(2) The AE signals in recovery processes of different strain levels seems to be due to the same source of detwinning process because the same slope between voltage and logarithmic AE count of AE signals in different strain levels was found in the strong elastic waves related to detwinning process.

(3) The relations between twinning or detwinning and AE counts in both deformation and anelastic recovery process could be expressed by a general equation.

REFERENCES