

## Elimination of yielding asymmetry in extruded AZ80 alloy by ageing

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**Abstract:** Samples of AZ80 alloy were hot extruded at 380 °C and aged at temperatures of 170 °C and 310 °C respectively for different periods to compare the effect of precipitate structures on the tensile–compressive yielding asymmetry in magnesium alloy. Uniaxial tension and compression along the extruded direction were carried out at room temperature. It was found that the yielding asymmetry in the aged samples was not as significant as that in the as-extruded samples. This was because twinning occurred less readily in the aged samples. And it was also confirmed by the fact that the increment of the critical resolved shear stress (CRSS) for twinning was higher and the Schmid factor was lower in the aged samples in the presence of precipitate. Thus, it was concluded that the yielding asymmetry could be reduced and even eliminated by increasing the area fraction of the precipitate phase.

**Key words:** AZ80 Mg alloy; precipitation; ageing; yielding asymmetry; twinning

### 1 Introduction

Wrought magnesium alloys having a hexagonal close-packed (HCP) structure, exhibit significant tension-compression yielding asymmetry in contrast to metals having more symmetrical crystal lattices [1,2]. The ratio between the compressive yield stress (CYS) and the tensile yield stress (TYS) is used to describe the extent of the tensile–compressive yielding asymmetry in the alloy when it is strained along its principal direction of predeformation [1,3]. A number of studies have been carried out on the yielding asymmetry in wrought magnesium alloys [2,4–6]. This asymmetry has been attributed to the polar nature of the  $\{10\bar{1}2\}$  tensile twinning in the alloys [7,8]. This result has been confirmed by WANG et al [9] that the distinct difference in area fraction of twin along the extruded direction which results in this phenomenon, originates from the difference in the number fraction of twinned grains in AZ31 alloy.

Previous studies indicated that the occurrence of  $\{10\bar{1}2\}$  twinning depends on the texture, grain size and on the presence of a precipitation. The texture can often cause the compressive yield stress (CYS) to be only 0.4–0.7 times the tensile yield stress (TYS) along the

extrusion or rolling direction [1]. For example, this phenomenon tends to be different in AZ31 alloy when the angle between the applied stress and the extrusion axis varies which is caused by the variation of the angle between the grain orientation and the applied stress [10]. In addition to texture, grain size also influences the yielding asymmetry. YIN et al [11] and BOHLEN et al [12] found that the yielding asymmetry of magnesium alloy decreased with the decreasing in grain size. It was also found that the grain size of magnesium alloys can have a critical effect on twinning in the alloys [13]. There are a number of works on the effect of precipitation after ageing on the AZ80 alloy [14,15]. The presence of a precipitate was reported to modify the process of twinning in aged magnesium alloys. JAIN et al [16] and LV et al [17] reported that ageing could reduce the asymmetry when the magnesium alloy samples were aged to contain precipitation. However, in Ref. [16], this asymmetry was studied using a cast AZ80, which will mislead people of the definition of the tension-compression yielding asymmetry in wrought magnesium. Besides, LV et al [17] focused on the effect of ageing and solution on twinning during deformation. But they both did not give the effect of different morphology of precipitate and how deeply the density of precipitate affects the asymmetry.

Thus, the present study will focus on two aspects. One is to investigate the effect of the microstructures of precipitates and their orientation on twinning in aged specimens of the alloy AZ80. The other is to determine the relation between the density of the precipitates and the tensile–compressive yielding asymmetry in the aged alloy specimens.

## 2 Experimental

The material used in this study was a commercial AZ80 Mg alloy with the chemical composition (mass fraction, %): 8.3 Al, 0.6 Zn, 0.5 Mn, 0.002 Cu, Mg (balance). The as-cast ingot was solution treated at 410 °C for 12 h to dissolve the precipitated phase, and then extrusion was carried out at 380 °C with an extruded ratio 7 to obtain a homogeneous equiaxial microstructure. Following the determination of the age-hardening response, several conditions were chosen for additional testing: underageing, peak ageing and overageing, corresponding to ageing at 170 °C and 310 °C for 0–100 h and 0–24 h, respectively. The microstructures and morphologies of the samples were characterized by optical microscopy (OM) and scanning electron microscopy (SEM). The area fraction of precipitate was measured by counting images of precipitation and matrix using Photoshop. The microstructure of the as-extruded sample is shown in Fig. 1. It can be seen that owing to the high processing temperature, the specimen consisted of equiaxed recrystallized grains that had an average grain size of 31  $\mu\text{m}$ ; besides, twins were not found in the extruded sample. However, little precipitate exists at the grain boundaries which can be ignored compared to the fraction of precipitate in the aged samples.

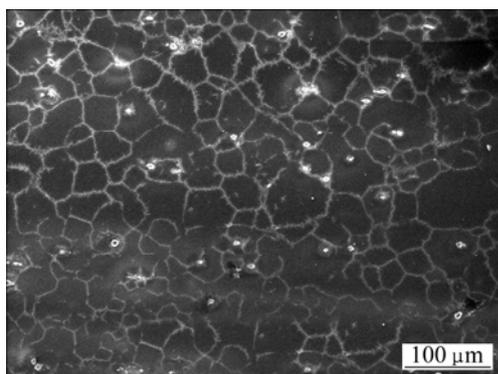


Fig. 1 SEM image of extruded AZ80 alloy

Tensile specimens with gauge dimensions of 2 mm×3 mm×10 mm, and compression specimens with gauge dimensions of 8 mm×8 mm×12 mm were cut along the extruded direction. Mechanical tests were conducted at room temperature with an initial strain rate of  $5 \times 10^{-2} \text{ s}^{-1}$  on a SANS-CMT5105 testing machine. Transmission electron microscopy (TEM) was used to

obtain the interaction between the precipitation and matrix. The foils were thinned by a twin-jet polisher and after that a JEM–200EX electron microscope operating at 200 kV was used to examine the foils. To characterize the texture of the samples in different conditions, (0002) pole figures from cross section of the extruded rods were measured using a X-ray diffractometer (model Philip X'pert PRD) with Co  $K_{\alpha}$  radiation.

## 3 Results and discussion

### 3.1 Variation of yield stresses and yielding asymmetry

The tension and compression yield stresses of AZ80 after ageing at 170 and 310 °C with different periods are shown in Fig. 2. It was noticed that tensile–compressive yielding asymmetry was very significant in the as-extruded AZ80 alloy, with the ratio of CYS/TYS 0.6. After ageing at 170 °C, the tension and compression yield stresses all increased compared with those of the as-extruded sample, during which the CYS/TYS increased to 0.93 at 6 h. After ageing for 24 h, the CYS exceeded the TYS, which means the tension–compression yielding asymmetry disappeared. For the samples aged at 310 °C, the yield stress increased at first and then decreased after 6 h ageing. And CYS/TYS increased from 0.92 at 2 h to 0.995 at 24 h.

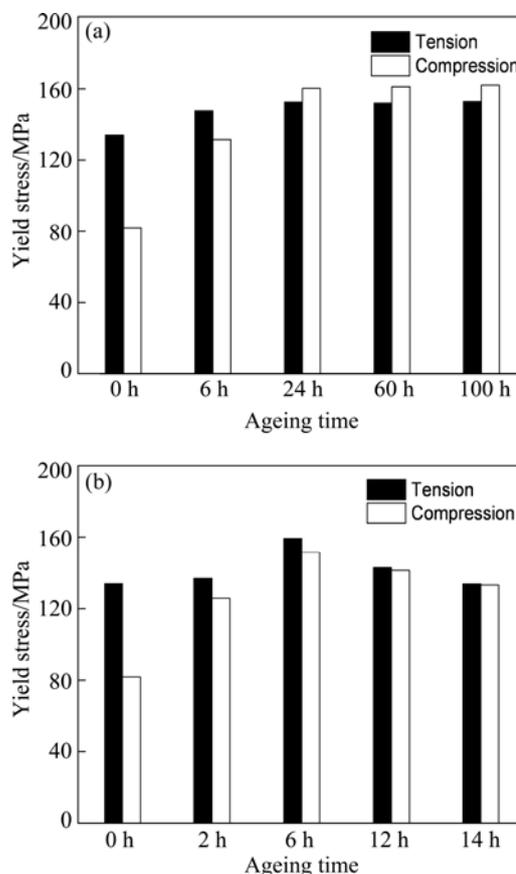


Fig. 2 Yield stress of as-extruded and aged AZ80 Mg alloy at 170 °C (a) and 310 °C (b)

### 3.2 Effect of microstructures on yield asymmetry

Since the as-extruded AZ80 alloy exhibited distinct yielding asymmetry in contrast to the aged samples, thus, to further elucidate the effect of twinning and precipitation, the microstructures of the as-extruded and aged samples after being compressed at 2% were investigated as shown in Fig. 3. It was found that twinning seldom occurred in the aged samples, while a significant amount of twinning was noticed in the compressive extruded sample. This means the difference in yielding asymmetry due to the appearance of twinning for the un-aged and aged samples, which is as the same as the results in the AZ31 alloys mentioned above.

The microstructures of the specimens aged at 170 °C for 6 h and 310 °C for 2 h are shown in Fig. 4 to further clarify the effect of precipitation in the yielding

asymmetry. Plate-like or granular-like  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> precipitated continuously in the alloy aged at 310 °C with the average grain size of 37  $\mu$ m; the plates were nucleated and grew homogeneously between the grains. Discontinuous precipitation occurred at 170 °C, with the formed precipitate having a cellular structure whose lamellas were parallel or perpendicular to the basal plane of the matrix, nucleated at the grain boundaries, and grew into grains. It has been concluded that twinning is prone to appear in coarse grains [18]; therefore, grain size is not the reason for the yielding asymmetry in this study.

TANG et al [19] indicated that the strengthening caused by discontinuous precipitation was stronger than that caused by continuous precipitation, which was opposite to the results in CELLOTTO et al's work [20]. However, from the yield stresses in Fig. 2, when being

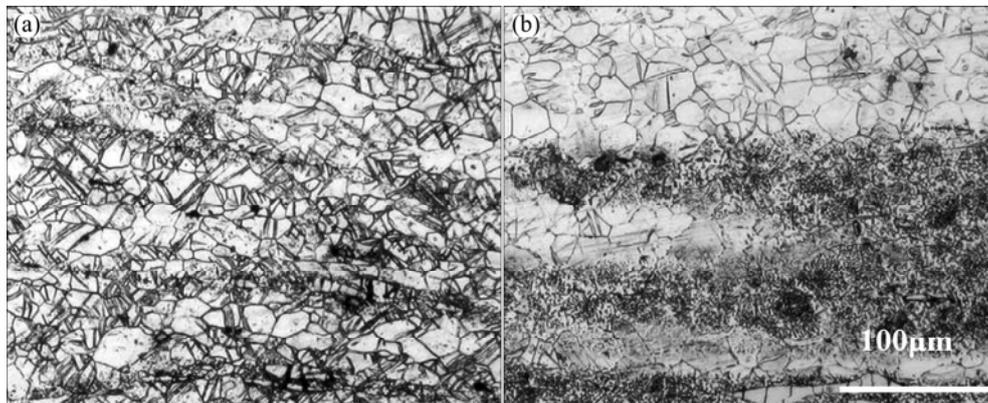


Fig. 3 SEM images of AZ80 alloy compressed to 2% after being as-extruded (a) and aged at 310 °C for 2 h (b)

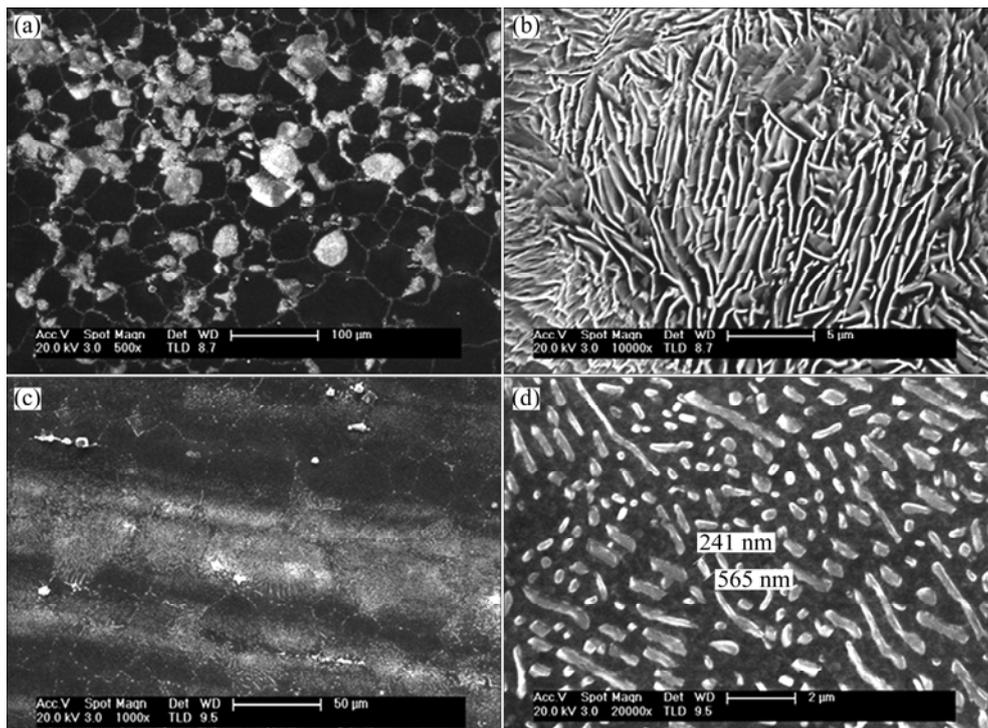


Fig. 4 SEM images of specimens aged at 170 °C for 2 h (a, b) and 310 °C for 6 h (c, d)

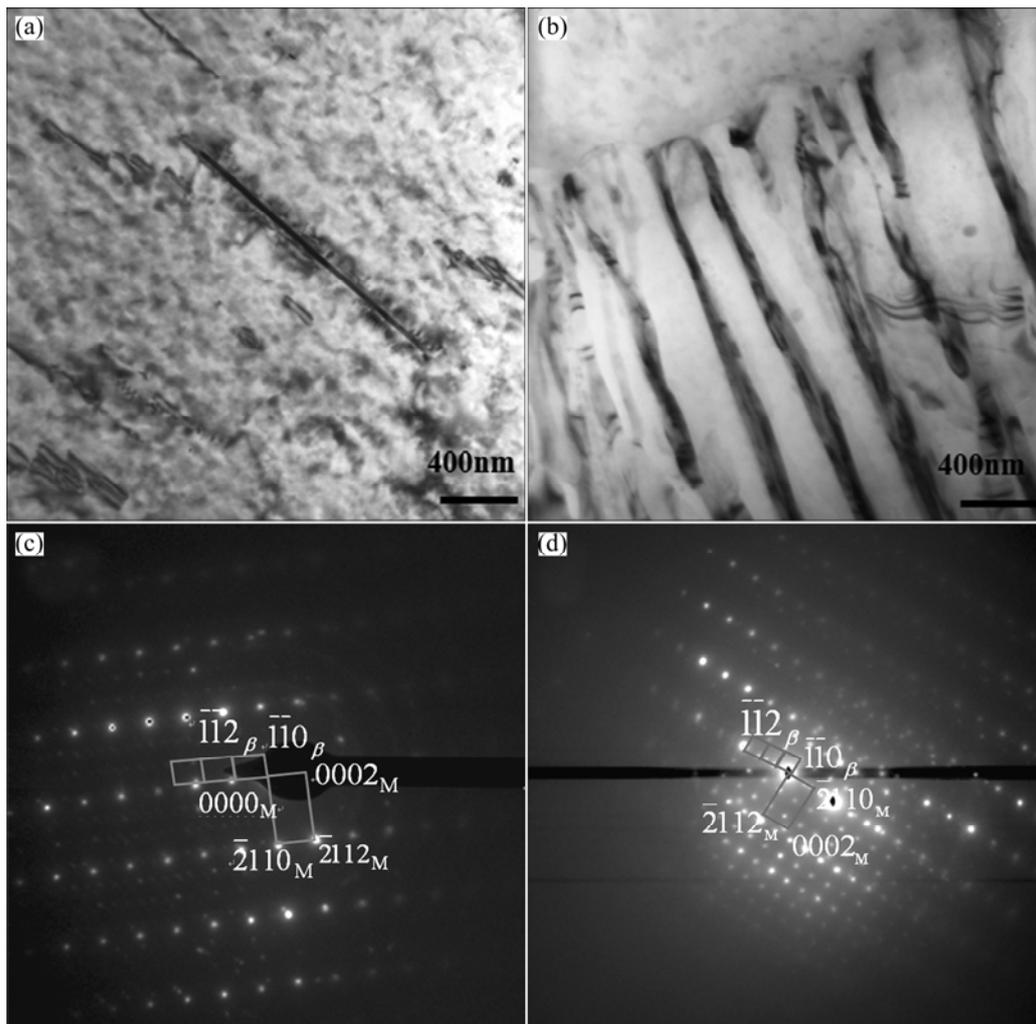
aged for 6 h, yield stress of the sample with a continuous precipitation was a little higher; while this result changed when aged for 24 h. This should owe to the fact that when being aged for 12 h, the samples were the peak and overaged with continuous precipitation.

The microstructures of discontinuous and continuous precipitates are shown in Figs. 5(a) and (b). In Figs. 5(c) and (d), interaction between the precipitate and matrix was determined using the reflection spots by TEM. It was shown that the orientation relationship between the precipitation and the magnesium matrix is  $(0002)_M // (110)_\beta$  and  $[01\bar{1}0]_M // [1\bar{1}2]_\beta$ , which is consistent with the other literatures [15]. The same result was shown between the discontinuous precipitation and the matrix. However, a tilt angle existed between the close-packed directions of the two phases, namely,  $2^\circ$ , suggesting that the angle between the matrix and the continuous precipitation and that between the matrix and the discontinuous precipitation were the same. This result indicated that the interaction between the two

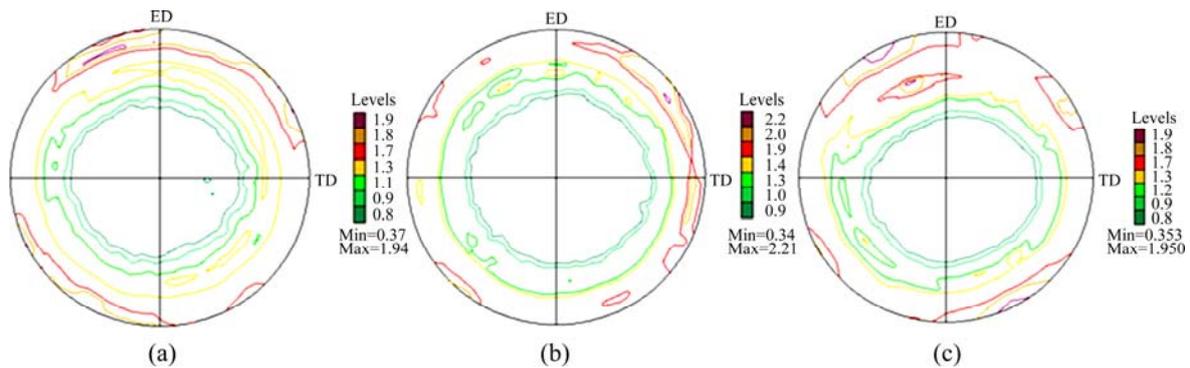
phases was not responsible for the yielding asymmetry exhibited differently in the aged samples.

### 3.3 Effect of grain orientations on yielding asymmetry

The orientation of the grains affects the occurrence of twinning as well. Thus, in order to evaluate if the differences between the as-extruded and aged samples could be attributed to differences in their textures, the (0001) pole figures corresponding to the two conditions were determined as shown in Fig. 6. The results are the same as those in other literatures [21]. It can be seen from the pole figures of the three samples that the intensity of the basal pole was distributed in a circular manner around the extrusion rods. The farther the basal pole was from the center, the greater the intensity was, indicating an obvious ring-like extruded texture with most of the basal planes oriented nearly parallel to the extrusion direction. By referring to Ref. [22] about the analysis of HCP metals, the texture components with the maximum pole densities for the three compared samples



**Fig. 5** TEM images of discontinuous precipitate (a) and continuous precipitate (b), diffraction spot for continuous precipitation (c) and discontinuous precipitation (d) (M is denoted as matrix)



**Fig. 6** (0001) pole figures of samples in different states: (a) As-extruded; (b) Aged at 170 °C for 6 h; (c) Aged at 310 °C for 2 h

are  $(\bar{1}430)[10\bar{1}1]$ ,  $(02\bar{2}1)[1\bar{1}01]$  and  $(\bar{1}430)[10\bar{1}2]$  respectively. The orientation factor for single deformation mode such as basal slip or twinning is given by:

$$\mu = \cos \varphi \cos \lambda \quad (1)$$

where  $\varphi$  is the angle between the loading axis and the normal of slip plane;  $\lambda$  is the angle between the loading axis and the slip direction.

From the texture in Figs. 6(a) and (c), when the stress axis is parallel to the extruded direction, the orientation factor for basal slipping in the extruded and aged at 310 °C for 2 h samples are 0.17 and 0.36 respectively, while the values for twinning are 0.33 and 0.25 comparatively.

The activation for twinning and slipping is also depending on the critical resolved shear stress (CRSS). According to Orowan mechanism, CRSS for different deformation mechanisms will be strengthened by the presence of precipitation. NIE [23] worked on the increment of CRSS in Mg–5Zn alloy and found that the CRSS for basal slipping and twinning in aged sample are 20 and 80 MPa compared to 18 MPa for both of them in the extruded samples. STANFORD et al [24] investigated the dominance of certain deformation modes in strongly textured AZ91 alloy to estimate the CRSS for basal slipping, prismatic slipping and  $\{10\bar{1}2\}$  twinning. The results showed that the basal slipping mode showed only a small hardening increment with ageing of around 5 MPa; however, the twinning showed increased hardening with increased ageing time. This means that the stress to activate twinning is higher in the samples with more precipitation. Taking CRSS and orientation factor into consideration, the activation of twinning is suppressed by precipitation.

In addition, for comparing the as-extruded samples with the aged ones, it was found that the grains of the aged material were larger in size and were dispersed and thus more conducive to twinning. On this basis, it could be concluded that this phenomenon was caused by the

existence of precipitate. JAIN et al [16] suggested that the effect of the precipitate is even more marked in a highly textured material. The results of the current study suggested that the precipitation of  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> affected the nucleation of twins. Thus, the interaction between twinning and the precipitate formed has been proposed to primarily arise from the difficulty experienced by the migrating twin boundaries in propagating through a highly dense precipitate [25].

### 3.4 Effect of precipitates fraction on yielding asymmetry

To clarify the effect of precipitation, the microstructures of samples aged at 170 °C for 100 h and at 310 °C for 6 h are shown in Fig. 7. It can be seen that when the samples were overaged, the grains were full of precipitates which prevent the activation of twinning.

The yielding asymmetry should be reduced by the fraction of precipitates after the effect of morphology and interaction between the phases was excluded. Figure 8 shows the CYS/TYS ratios and the area fraction of the precipitates in the samples aged at different temperatures for different periods. The trends of the samples aged at 170 and 310 °C were similar. That is, both the CYS/TYS ratio and the area fraction of the precipitates both increased with an increase in the ageing time. This meant that the tensile–compressive yielding asymmetry could be attenuated when the fraction of precipitates became higher.

When the samples were peak aged or overaged, the tensile–compressive yielding asymmetry did not change with continuous ageing. Two reasons are responsible for this issue. First, precipitates nucleated from the grain boundaries as twinning does, which will struggle energy to make the activation of twinning much harder. Second, dislocation on basal plane was the main deformation mode along the three close-packed directions, and  $\beta$  phase parallel to  $(0001)_M$  will hinder the migration of dislocation during deformation.

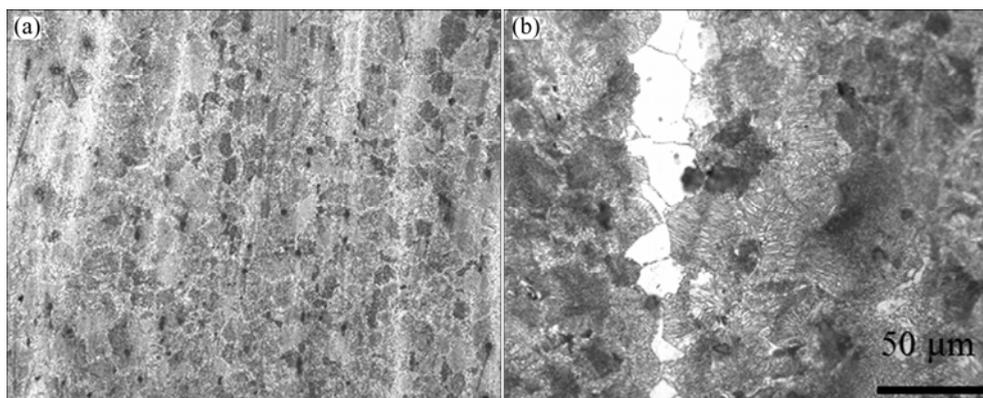


Fig. 7 SEM images of alloys aged at 310 °C for 24 h (a) and 170 °C for 100 h (b)

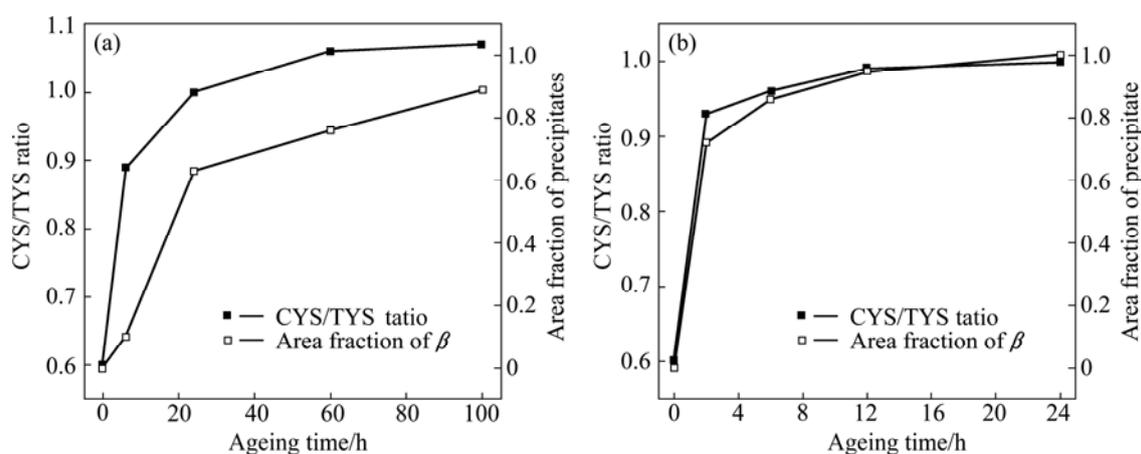


Fig. 8 CYS/TYS ratio and area fraction of precipitates when specimens were aged at 170 °C (a) and 310 °C (b)

## 4 Conclusions

1) The tensile-compressive yielding asymmetry was significant in the as-extruded samples but not so prominent in the aged samples. This was due to the occurrence of precipitation. The strengthening of the CRSS for twinning was higher than that for the basal slips; conversely, the orientation factor for twinning was lower in the aged samples than that in the as-extruded samples. This was the reason that twinning was less likely to occur in the as-extruded sample.

2) After ageing at 170 °C, a discontinuous precipitate phase was formed, with lamella grew from the grain boundaries to the interior of the matrix. Ageing at 310 °C made the formation of a continuous precipitate phase that was plate-like and granular-like distributed between the whole grains. On the basis of observations of the diffraction spots of the investigated samples, it was found that the orientation of the continuous precipitate with respect to the matrix was the same as that of the discontinuous precipitate with the matrix.

3) The tensile-compressive yielding asymmetry could be attenuated by increasing the ageing time when the fraction of precipitates increased. When the samples

were peak aged or overaged, the tensile-compressive yielding asymmetry did not change with continuous ageing.

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## 时效消除 AZ80 镁合金的屈服不对称性

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**摘 要:** AZ80 镁合金在 380 °C 挤压之后, 分别在 170 °C 和 310 °C 下进行不同时间的时效, 以比较析出相的形貌和结构对镁合金屈服不对称性的影响。对沿挤压方向切取的样品在室温下进行单轴拉伸及压缩实验。结果发现, 时效样品的拉压屈服不对称显然比未时效挤压样品的弱很多, 这是因为时效样品中的孪生发生远少于挤压样品中的。此现象可以通过时效样品中孪生的临界剪切力增量比基面滑移的增量大, 而其取向因子却减小使得其发生更困难来证实。镁合金中的拉压屈服不对称性可以通过增加析出相分数来减弱甚至消除。实验结果显示, 当合金内析出相分数达到饱和后, 拉压不对称性消失。

**关键词:** AZ80 镁合金; 析出相; 时效; 屈服不对称; 孪生

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