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## Temperature Dependence of Low Cycle Fatigue Behavior in AZ31 Magnesium Alloy

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### Abstract

In situ neutron diffraction has been used to investigate the deformation behaviour of extruded magnesium at room temperature, -100°C and 150°C. Significant differences in the active twinning/slip systems, attributed to the strong crystallographic texture and temperature dependence, were observed. Twinning is the dominant deformation mode during the cyclic loading at room temperature and at cryogenic temperature. In contrast, at high temperature, additional slip planes were activated and reduction of twinning activities was observed and the deformation mode is slip-dominated.

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## 1. Introduction

Magnesium alloys are of growing importance for automobile applications, due to their excellent strength to weight properties and the pressure on manufacturers to develop lighter, more efficient vehicles. However, the formability and ductility of existing magnesium alloys are adversely affected by hexagonal close packed structure which means that there limited slip systems available and von Mises criterion of five available slip systems is not met at room temperature. Additionally, there is anisotropy on the single crystal level due to the hexagonal crystal structure. This limits the modes available for plastic deformation, and can give rise to significant intergranular stresses. The plastic strain is typically accommodated by a mix of dislocation slip and twinning deformation modes, giving rise to rapid texture changes at relatively small macroscopic strains. Whether slip or twinning deformation dominates depends upon the sense and direction of applied load in relation to the crystallographic texture. In extruded magnesium alloys, the texture is such that basal poles tend to lie in the transverse plane, i.e. perpendicular to the extrusion axis. In uniaxial compression parallel to the extrusion axis at room temperature, strain is mainly accommodated by twinning. However, due to the inherent directionality of twinning, this mechanism cannot accommodate the deformation introduced during tensile straining along the same axis, and thus under this loading condition slip deformation dominates. The active deformation mode is affected by temperature. For example, Chandrasekaran [1] performed experiments on extrusion of magnesium alloys at a range of temperatures including room temperature, 100°C, 150°C, 175°C and 200°C. They observed that twinning was the predominant mode of deformation at lower temperatures. However, the formability of Mg alloys can be improved at elevated temperatures (above 100°C), as additional slip planes are activated [2]. Jain and Agnew [3] also report that the critical resolved shear strength (CRSS) for activated basal, prismatic  $\langle a \rangle$  and pyramidal slip  $\langle c+a \rangle$  modes are reduced with increasing temperatures, whereas CRSS of tensile twinning is increased. The dominant deformation mode therefore changes at different temperatures.

For cyclic loading experiments on magnesium at room temperature, reversible twinning is prevalent. The deformation twins form during compression along the extrusion axis and completely disappear when subsequent tensile loading is applied. This influences both the macroscopic response and the generation of intergranular stresses. The present study builds upon previous research on the deformation behaviour of magnesium by investigating the dependence upon temperature of the twinning/slip balance. To our knowledge, this is the first report on neutron diffraction studies of cyclic loading tests at temperatures below ambient.

## 2. Experimental details

Extruded bar of Mg AZ31 was supplied by Magnesium Elektron Ltd. Fatigue test samples of diameter 8mm and gauge length 14mm were machined with their axes aligned parallel (this set of samples will be called //Mg in the content) and perpendicular to the extrusion axis (this set of samples will be called  $\perp$ Mg in the content). In situ cyclic loading tests were performed during neutron data acquisition at the ENGIN-X engineering diffractometer at the ISIS pulsed spallation neutron source, UK [4-5]. The tests were performed in strain control for room temperature and high temperature tests, using a clip gauge mounted on the extensometer. Position control was used for the low temperature test as an extensometer to work at that temperature was not available. The strain rate is  $1.0 \times 10^{-3}$ /s. Neutron diffraction spectra were acquired using a nominal scattering volume of  $4 \times 8 \times 4 \text{ mm}^3$  for data acquisition periods of 4 minutes per measurement point. Spectra were acquired at different load steps within a cycle and 12 measurement points were acquired in each cycle.

### 3.3. Results and Discussion

#### 3.1. Stress-strain hysteresis loop

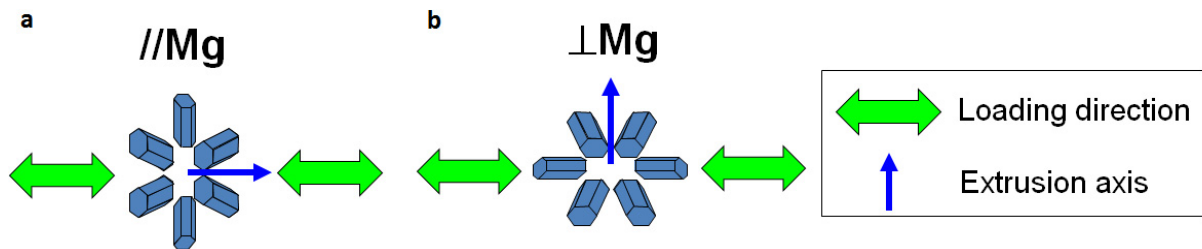


Fig. 1 Illustration of 00.2 grain in respect to the loading (bulky arrow) and extrusion axis (narrow arrow): basal poles are perpendicular to the extrusion axis, and the loading direction is (a) parallel and (b) perpendicular to the extrusion axis.

Extruded magnesium alloys have strong basal plane textures, where the c-axis is perpendicular to the direction of extrusion. Fig. 1 shows the applied loading directions with respect to the extrusion axis. Fig. 1a shows the //Mg samples were loaded in the direction parallel to the extrusion direction, which is perpendicular to the c-axis. Fig. 1b shows the  $\perp$ Mg samples were loaded in the direction perpendicular to the extrusion direction, and hence parallel to the c-axis.

Fig. 2 shows the macroscopic stress-strain responses for the initial loops at the room temperature tests for sample with loading direction parallel (//Mg) and perpendicular ( $\perp$ Mg) to the extrusion direction. Fig. 2a is the stress-strain curve for //Mg sample and Fig. 2b is for  $\perp$ Mg sample. In Fig. 2a, the initial yield stress in compression is about 90MPa, followed by a plateau with little work hardening. Upon unloading from compression, there is a far more pronounced degree of reverse yielding. This continues through zero stress, and eventually there is an inflection towards greater stress. This is consistent with other observations of the Bauschinger effect in magnesium alloys [6,7]. Beyond the inflection, the hardening rate rapidly increases due to the exhaustion of the detwinning mechanism. Unloading from tension again gives a near-linear response. However, upon entering the compressive regime once more, yielding begins earlier than in the initial loading and occurs more gradually, with a less sharp yield point and greater work hardening.

In Fig. 2b, initial yield in tension occurs at about 55MPa, and exhibits a shallow strain plateau. Strain recovery occurs almost immediately upon unloading and continues through zero to the maximum compressive strain, which is reached at the small load of -50MPa. Strain recovery is again significant before the compressive load is removed, so that the gradient upon passing through zero load is much lower than in the initial loading curve.

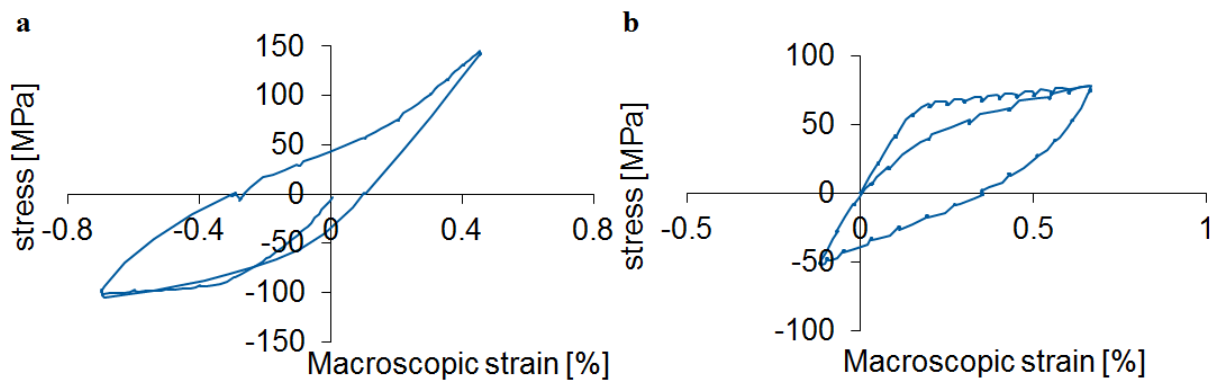


Fig. 2 Macroscopic stress-strain responses (a) //Mg AZ31 at room temperature (b)  $\perp$ Mg AZ31 at room temperature

The hysteresis loop of loading along the extrusion direction (Fig 2a) has a different shape from the test loading along the transverse direction (Fig 2b). As the  $c/a$  ratio of a hexagonal Mg lattice is less than  $\sqrt{3}$ , tension twins are activated easily by  $c$ -axis tension [8]. The dominant deformation mode is twinning on the  $\{10\bar{2}\}$  planes along the  $\langle 10\bar{1} \rangle$  direction and occurs during compressive loading parallel to the direction of extrusion, but not during tensile loading. This tensile twinning results in a  $86.3^\circ$  reorientation of the basal pole. It was also observed that detwinning occurs in the twinned areas on subsequent reversed loading with the tensile stress applied along the  $c$ -axis of the twinned materials. Due to the different deformation behaviours occurring during compressive and tensile deformation, extruded magnesium alloys display a strong asymmetry between compression and tension as shown in Fig. 2a. In contrast, the hysteresis loop is symmetric between the compression and tension cycle for  $\perp$ Mg sample as shown in Fig 2b, which is usually the result of the dislocation slip-dominated deformation in most materials.  $\perp$ Mg sample has a larger spread in the basal pole orientation in the plane parallel to the loading direction, which allows a more significant volume fraction of grains to deform by basal slip, rather than tensile twinning. For this test (Fig. 2b), twinning was not captured; whereas extensive twinning under the compressive loadings and detwinning during the subsequent reversed loading was observed for samples with the  $c$ -axis perpendicular to the loading direction (Fig. 2a). Furthermore the  $\perp$ Mg sample required much smaller maximum stresses to introduce the same level of plastic strain.

Fig. 3 shows the tests at  $-100^\circ\text{C}$  for  $//$ Mg and  $\perp$ Mg sample with the loading direction parallel and perpendicular to the extrusion direction respectively. Since a low temperature extensometer was not available, position control was used for the tests. The  $x$ -axis is the displacement between the sample grips, which includes the compliance of the machine and the sample. The hysteresis loop shapes are similar to the room temperature tests. The test for  $//$ Mg (Fig 3a) shows the characteristic hysteresis loops with concave region, which is attributed to the production and destruction of twins during cycling. The test for  $\perp$ Mg (Fig 3b) has a symmetric loop shape with much smaller maximum stresses required to introduce the same level of plastic strain comparing to  $//$ Mg (Fig 3a)

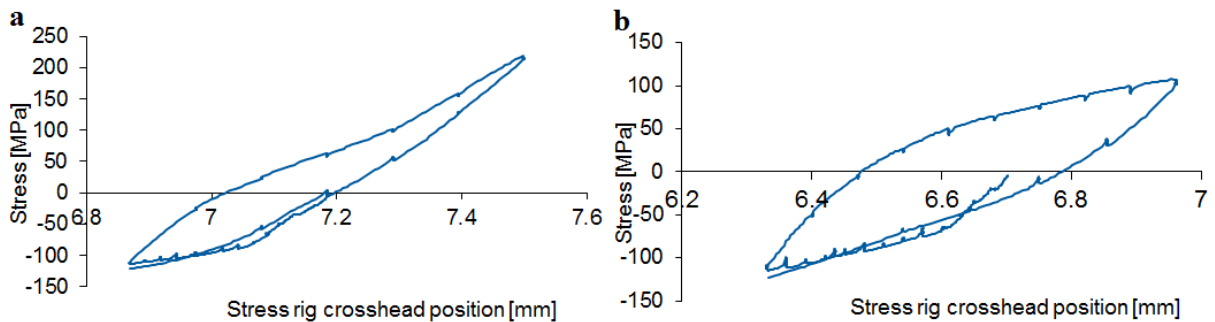


Fig. 3 Macroscopic stress-strain responses (a)  $//$ Mg AZ31 at  $-100^\circ\text{C}$  (b)  $\perp$ Mg AZ31 at  $-100^\circ\text{C}$

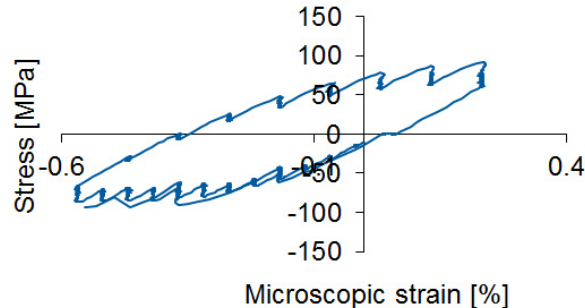


Fig. 4 Macroscopic stress-strain responses for  $//$ Mg AZ31 at  $150^\circ\text{C}$

Fig. 4 shows the test at  $150^\circ\text{C}$  for sample ( $//$ Mg) with loading direction parallel to the extrusion direction. The curve is composed of a series of plateaus due to stress relaxation during each data acquisition period. The initial yield stress in compression is about 50MPa, which is reduced as expected and followed with little work hardening.

The cyclic deformation behaviour has a symmetric shape loop, although this material has an asymmetric hysteresis loop when the test was performed at room and low temperature. In the test on //Mg sample at high temperature demonstrates that additional slip planes activated and reduction of twinning activities, the deformation mode is slip-dominated rather than twinning-dominated, which leads to greater similarities between tension and compression. The main reason is that the critical resolved shear strength (CRSS) for activated basal, prismatic  $\langle a \rangle$  and pyramidal slip  $\langle c+a \rangle$  modes are reduced with increasing temperatures, whereas CRSS of tensile twinning is increased. Further details of diffraction peak analysis to support this hypothesis are described in the next section.

### 3.2. Detection of twinning-detwining behavior

Fig. 5 shows initial neutron diffraction pattern of //Mg and  $\perp$ Mg. The diffraction spectra exhibit a typical Mg extrusion texture. In particular, the 00.2, 10.2 and 10.3 reflections are absent from the spectrum parallel to the extrusion axis, demonstrating a strong texture with the basal poles lying within or near to the transverse plane. Fig. 6 shows evolution of 00.2 intensities through the first cycle for //Mg during loading at  $-100^\circ\text{C}$  and at  $150^\circ\text{C}$ . For cyclic loading at  $-100^\circ\text{C}$ , the 00.2 peak emerges at  $-66\text{MPa}$  and keeps increasing with increasing load. Upon unloading, the 00.2 peak intensity decreases and then changes the rate of decreasing speed around  $50\text{MPa}$  and finally disappears at  $150\text{MPa}$ , where the detwining is exhausted and leaves slip as the only available deformation mechanism. This explains the existence of an inflection in the hysteresis loop (Fig. 3a). At  $150^\circ\text{C}$ , the 00.2 peak emerges around  $-70\text{MPa}$ , and the 00.2 peak intensity increases with increasing load. However after unloading 00.2 peak doesn't show decrease in intensity during unloading. When tensile stress is applied, the intensity starts decrease around  $30\text{MPa}$ . The maximum intensity of 00.2 peak at high temperature is much lower than the intensity observed at low temperature. It proves that twinning activity is reduced at higher temperature. The difference between the intensity evolution of 00.2 peak at different temperatures is also due to the different deformation modes that occur at different temperatures, which can be also explained by the lattice strain response during loading. There will be further discussion in the following section.

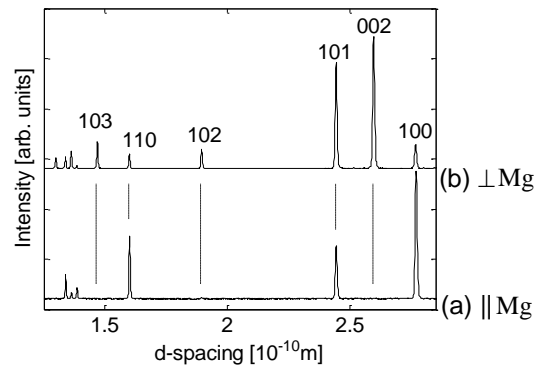


Fig. 5: Diffraction spectra of as-received material (a) parallel to extrusion axis //Mg; (b) transversely to extrusion axis  $\perp$ Mg.

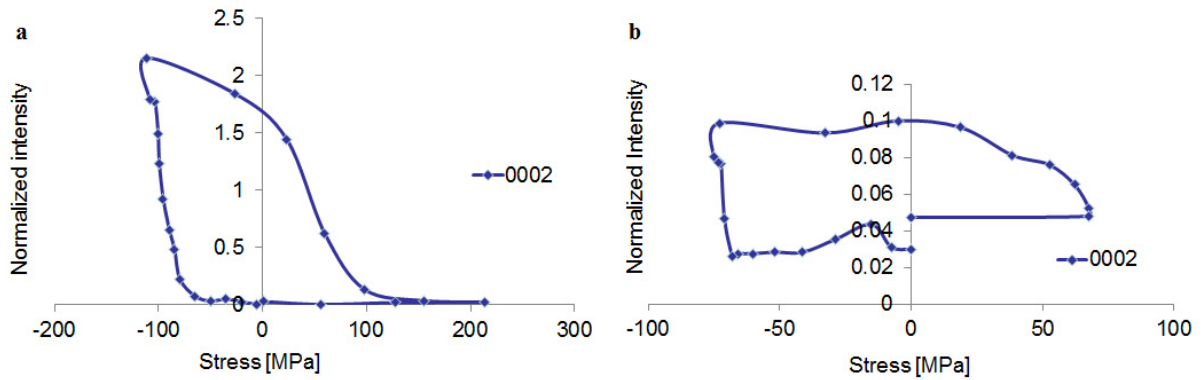


Fig. 6 (a) Evolution of 00.2 intensity through the first cycle for //Mg at -100°C and (b) Evolution of 00.2 intensity through the first cycle for //Mg at 150°C

### 3.3. Detection of activity of slip

Fig. 7 shows the elastic lattice strain as a function of applied stress for different temperature at initial loading. Due to the 00.2 diffraction peak absent in the initial axial diffraction pattern, the stress-free lattice spacing for 00.2 grain is calculated from the hcp-lattice parameters which were determined by Rietveld refinement of the full diffraction pattern acquired prior to loading. For the initial loading at high temperature, the 00.2 peak intensity is not enough for accurate strain analysis, so it is not included in Fig. 7b. Fig. 7a shows the lattice strain response starts deviate from linearity at the onset of plasticity. For the 10.0 lattice plane, it starts to deviate at -65MPa, which is the stress where the 00.2 intensity emerges. This is the evidence of the parent grain relaxing after twinning has taken place. Alternatively, the 00.2 grain shares greater portion of the applied load. As we mentioned earlier for high temperature, the 00.2 peak starts to emerge at about -70 MPa. However all the lattice strains deviate from linear elastic behavior much earlier than -70MPa, this shows that slip occurs before twinning. However, twinning occurs earlier than slip at the low temperature. Further evidence of detecting slip at the very early stage of the loading is shown by the peak broadening. Fig. 8 shows the peak width evolution as a function of applied stress for //Mg at high temperature. The favorable slip lattice planes, e.g. 10.0 (Prismatic  $\langle a \rangle$ ) and 10.1 (Pyramidal  $\langle a \rangle$ ) have the biggest changes in peak width, and this starts around -30MPa. They also showed the most extreme deviation from the elastic linear response (Fig. 7b). Both Fig. 7b and Fig. 8 have demonstrated that slip occurs before twinning at high temperature loading.

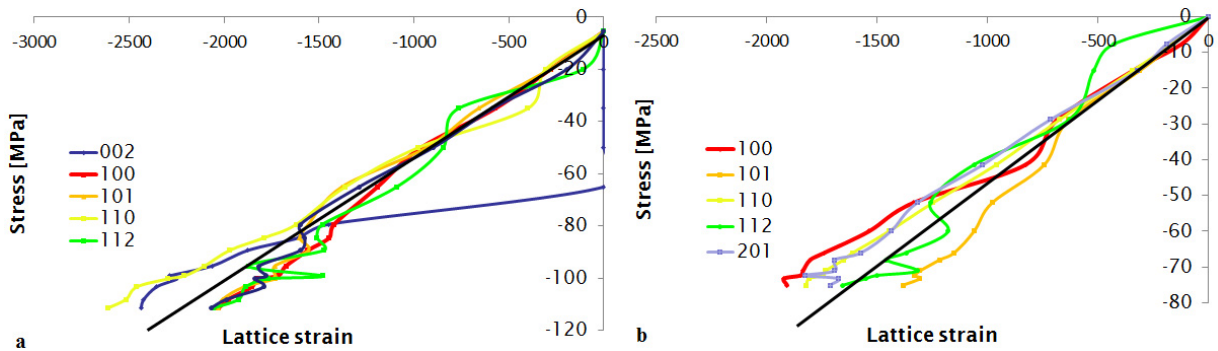


Fig.7 Axial elastic lattice strains as a function of applied stress a) loading at  $-100^{\circ}\text{C}$  b) loading at  $150^{\circ}\text{C}$

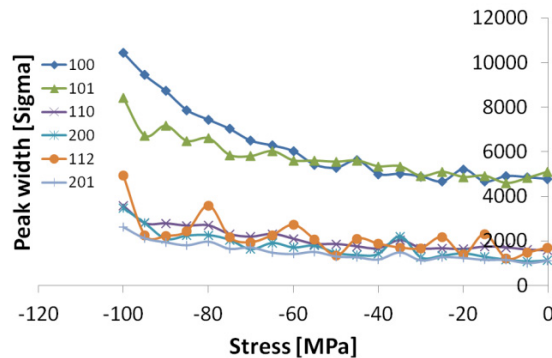


Fig. 8 Peak width evolution as a function of applied stress for //Mg at  $150^{\circ}\text{C}$

#### 4. Conclusion

In extruded magnesium alloys, the texture is such that basal poles tend to lie in the transverse plane, i.e. perpendicular to the extrusion axis. In uniaxial compression parallel to the extrusion axis at room temperature, strain is mainly accommodated by twinning. However, due to the inherent directionality of twinning, this mechanism cannot accommodate the deformation introduced during tensile straining along the same axis, and thus under this loading condition slip deformation dominates. The twins formed by plastic straining can be destroyed again when the sense of loading is reversed. In this paper, neutron diffraction is used to detect the deformation mode and measure the amount of twinning activity.

It has been shown that at room temperature and at cryogenic temperature, the hysteresis loop of loading along the extrusion direction has a different shape from the test loading along the transverse direction. Cyclic loading along the extrusion direction shows the characteristic hysteresis loops with concave region, which is attributed in the case to the production and destruction of twins during cycling. Cyclic loading perpendicular to the extrusion direction shows a symmetric loop shape.

However for the cyclic loading along the extrusion direction at  $150^{\circ}\text{C}$ , a symmetric loop shape is observed, although the material has an asymmetric hysteresis loop when tests are performed at room and low temperature. It is suggested that with additional slip planes activated and reduction of twinning activities, the deformation mode is slip-dominated rather than twinning-dominated, which leads to greater similarities between tension and compression. Although twinning is also observed at high temperature, the volume fraction of twins is less than at low temperature and it starts at higher stress level, which shows slower activation. For samples tested parallel to the extrusion axis, the successive production and destruction of mechanical twins was observed throughout the cyclic loading. Twinning is the dominant deformation mode during the cyclic loading at room temperature and at cryogenic temperature.

Whereas at high temperature, additional slip planes activated and reduction of twinning activities was observed, the deformation mode is slip-dominated rather than twinning-dominated.

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