



Short Communication

Enhancement of plasticity in Mg–3Al–1Zn alloy at cryogenic temperature



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ARTICLE INFO

Article history:

Received 6 June 2023

Accepted 16 July 2023

Available online 19 July 2023

Keywords:

Cryogenic deformation

Twin-twin interactions

Magnesium alloys

Plasticity

Electron backscatter diffraction (EBSD)

ABSTRACT

Compression tests were conducted at room and cryogenic temperatures to investigate the low-temperature plasticity in Mg–3Al–1Zn alloy. The strain to failure and fracture strength in Mg–3Al–1Zn alloy increase by 21.4% and 51.6% from room to cryogenic temperature. Using a *quasi-in-situ* EBSD method, it is found that $\{10\bar{1}2\}$ tension twins dominate at room temperature, while abundant $(10\bar{1}2)$ – $(01\bar{1}2)$ twin-twin interactions are observed at cryogenic temperature. Multiple slips, including pyramidal and basal slips, occur in twin-twin interactions, enhancing the strain to failure and flow stress at cryogenic temperature. The high dislocation density near twin-twin interactions boundaries would contribute to the high hardening rate and flow stress at cryogenic temperature. This work would provide a novel way to enhance plasticity in magnesium alloys and gain an in-depth understanding of twin-twin interactions.

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1. Introduction

Magnesium alloys are a promising lightweight material for applications in the automotive, aerospace and electronics industries, and they attract substantial interest from researchers and engineers [1–4]. The strain to failure in magnesium alloys is generally enhanced in elevated temperatures by activating more slip systems [5,6]. Exploiting its plasticity behaviour at low temperatures is of great interest, as deformation-induced twinning may play an increasingly important role in the plasticity to accommodate the imposed plastic deformation.

The most common twinning in magnesium alloys is $\{10\bar{1}2\}$ tension twins (TTWs) [3,7], and it is found that TTWs can

contribute to the increase of uniform elongation in tensile tests [8]. A grain generally twins on any one of six $\{10\bar{1}2\}$ planes to form TTWs. These TTWs can interact with each other to form twin-twin interactions, such as $(10\bar{1}2)$ – $(01\bar{1}2)$ interactions which show $60^\circ <01\bar{1}0>$ boundaries between the $\{10\bar{1}2\}$ tension twin variants [9,10]. More twins are expected to be activated at a lower temperature, leading to forming twin-twin interactions [9]. Twin-twin interactions can contribute to an enhanced strain hardening rate [8–11] because twin-twin interactions generally result in significant twinning-induced grain refinement [12]. However, the effects of twin-twin interactions on strain to failure still lack systematic research and reports. Cryogenic deformation is proposed to gain an in-

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<https://doi.org/10.1016/j.jmrt.2023.07.139>

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depth analysis of the effects of twin-twin interactions on strain to failure.

The mechanical behaviours of Mg alloy AZ80, AZ31 [11–13] and Mg-0.5Ca alloy [14] at cryogenic temperature were studied. It was found that the strain to failure decreased with decreasing temperature, and abundant TTWs were observed due to the limitation of dislocation slips. Pure Mg also shows enhanced tensile strength but reduced strain to failure at cryogenic temperature compared to room temperature; the reduced strain to failure was proposed to be related to the premature fracture [15]. However, at cryogenic conditions, some research found that the critical resolved shear stress (CRSS) of non-basal pyramidal slips in single-crystal magnesium and magnesium-lithium unexpectedly decreased with decreasing temperature from 293 K to 77 K [16,17], which could be attributed to the reduced pyramidal $\langle c+a \rangle$ dislocation transformations to immobile dislocations at cryogenic temperature [18]. Therefore, the twinning and dislocation activities and their effects on strain to failure at cryogenic temperature are still debatable.

Thus, this research aims to study the strain to failure and strength of an Mg alloy, AZ31, at cryogenic temperature and clarify the behaviours of twin-twin interactions and pyramidal dislocations. The *quasi-in-situ* electron backscatter diffraction (EBSD) method was applied to observe the microstructural evolution to better understand the relationship between microstructure evolution and plasticity at a local scale. The compression tests were conducted at room temperature (RT) and liquid nitrogen temperature (LNT, cryogenic temperature).

2. Methods

2.1. Material and experimental tests

The material used in this research is conventional rolled AZ31 alloy (Mg–3Al–1Zn alloy). All materials are in the O-temper. In this work, the cubic samples with dimensions of 7 mm × 7 mm × 7 mm were cut from the rolled plate by an electro-discharged machine (EDM). The compression tests at room and cryogenic temperature with a nominal strain rate of 1/s were conducted using an Instron 5584 machine. To achieve the cryogenic temperature at –196 °C, the samples were immersed in liquid nitrogen, and the assisted chamber was also applied to cool the samples. The compression direction (CD) of these samples was parallel to the rolling direction (RD), as the tension twins are easier to activate in this loading direction.

2.2. Electron backscatter diffraction (EBSD) measurements

Sample preparations for EBSD measurements were conducted for as-received samples using grinding with 4000 grit SiC paper for ~ 5 min and polishing with OPS suspension for ~15 min. These samples were placed in Hitachi 3400 SEM microscope with 25 keV voltage. A 540 µm × 406 µm EBSD map with a step size of 1 µm was scanned using a Bruker e-Flash, Quantax Esprit 2.1 system. A smaller step size of 0.5 µm was also applied to observe a more detailed microstructure. The

corner of the sample was set as a reference point for the scanned area. The same scanned area in the sample was relocated and scanned again after being imposed 0.06 compressive strain.

The EBSD data were analysed with the HKL CHANNEL 5 software. The twin boundaries were detected according to their specific misorientations around the orientation axes and highlighted with different colours. Then band contrast maps were overlaid with the corresponding twin boundaries. The slip trace analysis was conducted using MATLAB with MTEX code. The misorientation distribution map was plotted based on the grain and twin boundaries information. The kernel average misorientation (KAM) value was obtained by calculating the average misorientation between each pixel and its surrounding pixels, and the KAM value can qualitatively represent the dislocation density [19].

3. Experimental results

3.1. Mechanical behaviours of AZ31 at room and cryogenic temperature

Fig. 1(a) shows the true stress-strain curves under compression tests at 20 °C (RT) and –196 °C (LNT). It is worth noting that the strain to failure and fracture strength both increase with decreasing temperature. The strain to failure shows a 21.4% increase from 0.14 at RT to 0.17 at LNT, and the fracture strength shows a 51.6% increase from 335 MPa at RT to 508 MPa at LNT. The (0001) pole figures of these samples are plotted as insets in Fig. 1(a), showing a typical texture of rolled samples. When the CD is parallel to the RD, the CD is generally perpendicular to the c-axis of most grains, resulting in abundant twins. These two curves show the typical sigmoidal (S-shaped) compressive flow curve after a low-stress plateau [20]. The first part of the S-shaped curve is attributed to the initial twin formation at a low strain level. Subsequently, the newly formed twinned area is oriented in a plastically hard orientation and refines the initial grain size. These result in an increasing hardening rate [20].

In Fig. 1(b), according to the transition points of strain hardening, the strain hardening rate curves can be mainly divided into three stages, including (1) twinned stage, (2) hardening stage and (3) softening stage. In the twinned stage, the strain hardening rate of the LNT-deformed sample is much higher than that of the RT-deformed sample. The length of this section in the LNT-deformed sample is also longer; this result indicates that the twinning behaviour could be more active at LNT. After this section, most grains are twinned, and their c-axes change from perpendicular to the compression direction to parallel to the compression direction. Hence, the hardening rate of the LNT-deformed and RT-deformed samples during the hardening section is significantly high and almost increases linearly with increasing strain, similar to elastic deformation. In the final softening stage, the strain hardening rate decreases, which could result from activating the dislocation slip [21]. It is worth noting that the length of softening section is much longer at LNT than at RT, indicating that dislocation slips could be more active in this section at LNT.

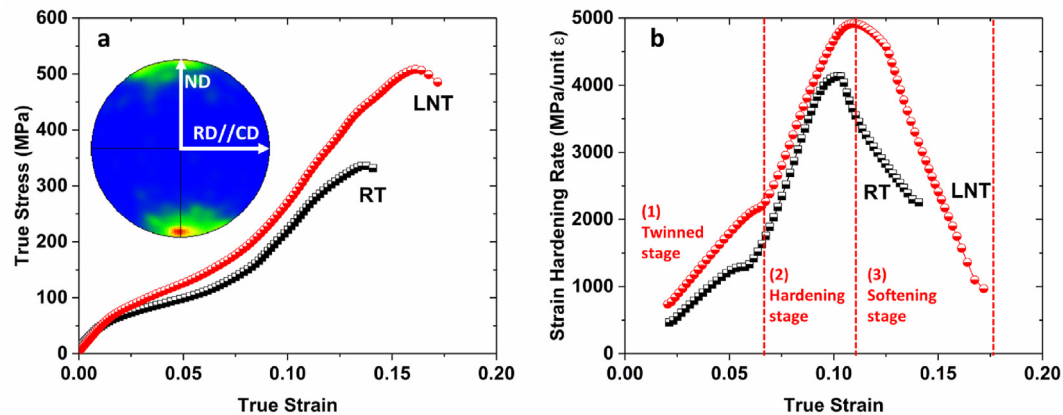


Fig. 1 – (a) True stress-strain curves results under compression tests at RT and LNT with CD parallel to RD, and (b) the calculated hardening rate variation as a function of true strain based on (a). The inset represents the texture using a (0001) pole figure.

3.2. Microstructural evolution of AZ31 samples at room and cryogenic temperature

To explain the enhanced mechanical properties and deformation mechanisms at cryogenic temperature, the microstructural evolution of these samples at RT and LNT were analysed using the *quasi-in-situ* EBSD method, i.e., the EBSD scanned areas are in the same position before and after deformation. As shown in Fig. 2(a–c), many $\{10\bar{1}2\}$ TTWs formed in the RT-deformed sample, while not only $\{10\bar{1}2\}$ TTWs but also abundant $(10\bar{1}2)\text{--}(01\bar{1}2)$ twin-twin interactions formed in the LNT-deformed sample, according to Fig. 2(e–g). Comparing the misorientation distribution maps shown in Fig. 2(d) and (h), the misorientation peak near 60° is steeper and higher in the LNT-deformed sample, indicating that twin-twin interactions are more active at LNT than RT.

Twin-twin interactions generally contribute to the high strain hardening rate, which would be the reason why the higher strain hardening rate was observed at LNT. In addition,

twin-twin interactions are generated by different twin variants with various crystal orientations, which could contribute to further dislocation slips and the enhancement of the strain to failure at LNT, as shown in Fig. 1.

3.3. Slip trace and twinning analysis

A thorough analysis of twinning and slip was conducted in the section. Fig. 3 plots the typical zone A (marked in Fig. 2(a)) in the RT-deformed sample, as its primary microstructure is TTWs. In Fig. 3(a) and (b), $\{10\bar{1}2\}$ TTWs are formed in the zone. Fig. 3(c) plots the KAM map of the deformed sample. The KAM value is low in the most area of TTWs, indicating that the dislocation density is low in TTWs. According to the SEM image in Fig. 3(d) and (e), basal slips are observed in the twinned area and cross the twin boundaries. These results indicate that basal slips are the primary slip type in the TTWs of the RT-deformed sample, while the dislocation density is low in most areas of these TTWs.

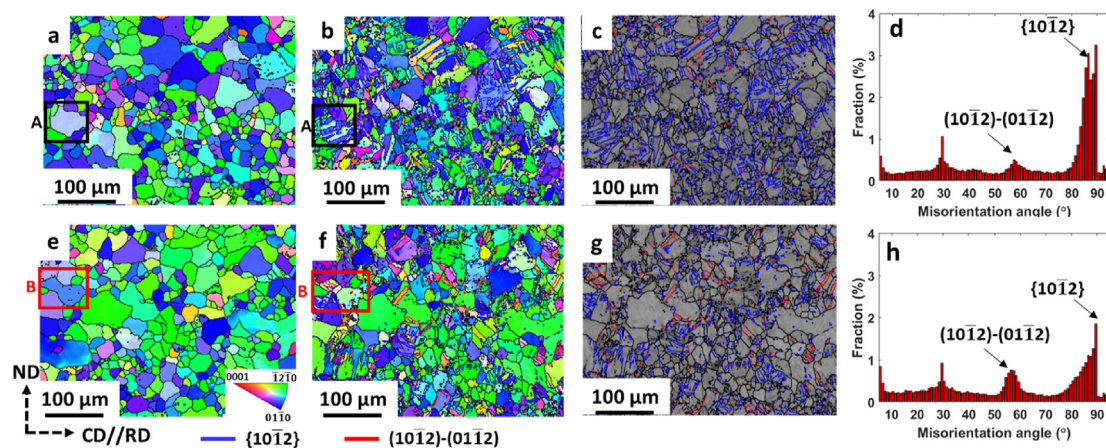


Fig. 2 – EBSD IPF images of (a, e) initial samples and (b, f) deformed samples to a strain of 0.06 with CD parallel to RD at RT and LNT, respectively, and corresponding (c, g) band contrast maps overlaid with twin boundaries, and (d, h) misorientation distribution maps, respectively.

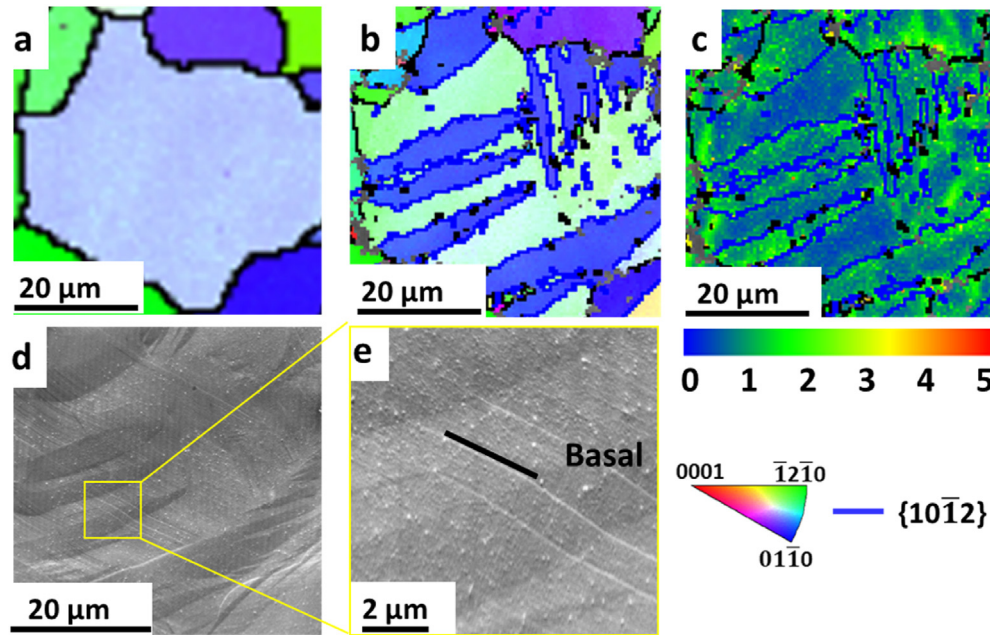


Fig. 3 – IPF images of Zone A (a) before deformation, and (b) after deformation to a strain of 0.06 at RT, and the corresponding (c) KAM map, (d) SEM image in the deformed sample, and the corresponding (e) magnified image, respectively.

The microstructural evolution of typical Zone B (marked in Fig. 2(e)) in the LNT-deformed sample was shown in Fig. 4. As shown in Fig. 4(a) and (b), abundant $(10\bar{1}2)-(01\bar{1}2)$ twin-twin interactions are observed in Zone B. According to the KAM map shown in Fig. 4(c), the KAM is high near the boundaries of twin-twin interactions, indicating that these boundaries would block the dislocation slips and result in high dislocation density. This would contribute to the high hardening rate at LNT. The SEM image in this zone and its corresponding magnified figure were plotted in Fig. 4(d) and (e). The active dislocation slips, namely multiple slips, including pyramidal and basal slips, are observed in the twin-twin interactions,

enhancing the strain to failure at LNT. Multiple slips generally have hardening effects, contributing to a higher hardening rate at LNT than at RT. Therefore, the higher hardening rate and strain to failure at LNT would be attributed to the active multiple slips with pyramidal slips in twin-twin interactions.

4. Discussion

The higher fracture strength and strain to failure at LNT rather than at RT is of great interest. This would be attributed to more twin-twin interactions at LNT than at RT, as

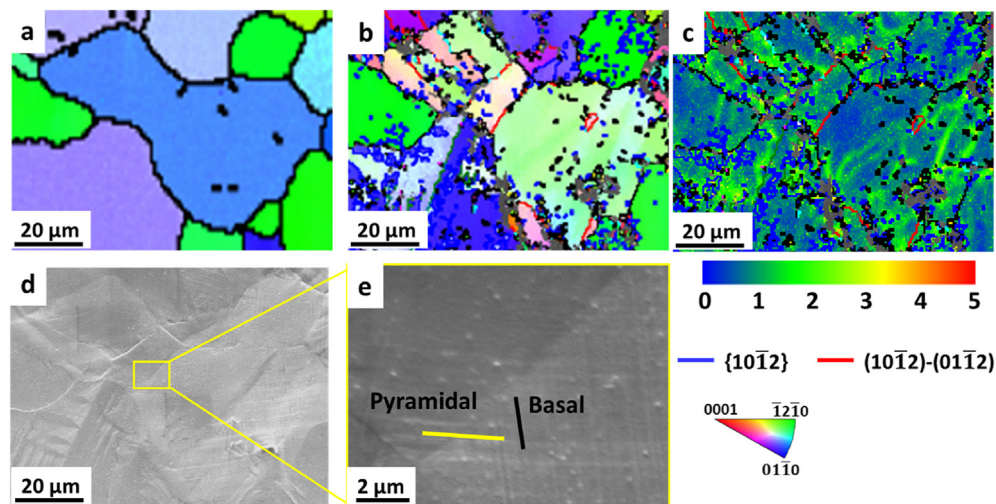


Fig. 4 – IPF images of Zone B (a) before deformation, and (b) after deformation to a strain of 0.06 at LNT, and the corresponding (c) KAM map, (d) SEM image in the deformed sample, and the corresponding (e) magnified image, respectively.

shown in Fig. 2. Twin-twin interactions generally result in a significant twinning-induced grain refinement [22] and contribute to improving strain hardening rate [23,24]. As shown in Fig. 4(c), the high dislocation density was observed around the twin-twin interaction boundaries, contributing to the high hardening rate and strength in the LNT-deformed sample. The twin-twin interactions in this sample would also locally increase the strength [25], increasing the global strength in the LNT-deformed sample. Therefore, the observed higher flow stress and strain hardening rate at LNT would result from increasing twin-twin interactions at the cryogenic condition.

The higher strain-to-failure value at LNT is because more dislocation slips were activated in twin-twin interaction regions. Twin-twin interactions were formed with twin variants, providing various crystal orientations for dislocation slips. As shown in Fig. 1(b), the more extended softening section was observed at LNT, indicating that more active dislocation slips would occur. In addition, multiple slips, including basal and pyramidal slips, were observed in the twinned area of the LNT-deformed sample (Fig. 4), and pyramidal slips generally contribute to a high strain to failure [26–28], so it is not unreasonable to observe the higher strain to failure of AZ31 alloy at LNT than that at RT.

Notably, the sites with high dislocation density and local stress near twin-twin interactions could be potential cracking sites [29,30], resulting in low ductility. However, the LNT-deformed sample shows both high strain to failure and strength. This would be attributed to its active dislocation slips, especially multiple slips, to relieve the stress around the boundaries of these twin-twin interactions at LNT. The result would be similar to the high strain to failure and strength induced by double twins, because the enhanced pyramidal $\langle c+a \rangle$ slip in coarse-grained Mg-4wt% Li would relax the localised stress generated by thin double twins [1].

The possible reasons for multiple slips and pyramidal slips observed in twin-twin interactions of the LNT-deformed sample are: (1) The various crystal orientations generated by twin-twin interactions would create more paths for dislocation slips. This is different from TTWs in the RT-deformed sample, as the single TTW variant generally results in one crystal orientation and the strong basal texture, which would suppress dislocation slips [31,32]. (2) The local stress is generally high near twin-twin interactions [25], which would contribute to the activation of pyramidal slips. (3) The anomalous temperature dependence of pyramidal slips would enhance the activity of pyramidal slips at cryogenic temperature. It is reported that the critical resolved shear stress of pyramidal slip in single crystals of Mg and Mg–Li decreases with decreasing temperature from room to cryogenic temperature [17,33]. This is because of the reduced dislocation transition from pyramidal slip to basal-dissociated immobile slip at the cryogenic temperature [18]. Therefore, it can be expected that pyramidal slips become more active in the LNT-deformed sample, resulting in multiple slips combined with basal and pyramidal slips, as shown in Fig. 4(e). The mechanisms for enhanced plasticity at low temperature could be applied in most Mg alloys rather than only Mg–Al–Zn alloy system. As

more active pyramidal slips also occur in Mg [33], Mg–Li alloys [17] and a Mg–rare earth alloy [34] at cryogenic temperature, which can contribute to their enhanced plasticity at low temperature. As the plasticity is also related to twinning behaviours in Mg alloys [14], their twinning activities should also be considered to achieve the enhanced plasticity at low temperature.

5. Conclusions

In summary, the mechanical behaviours and microstructural evolution, including twinning and dislocation slips, were investigated in Mg–3Al–1Zn alloy at room and cryogenic temperatures. The microstructure evolution before and after deformation have been revealed using *quasi-in-situ* EBSD and slip trace analysis. The following conclusions can be made.

- (1) The strain to failure and fracture strength of Mg–3Al–1Zn alloy with CD parallel to RD increased by 21.4% and 51.6% from RT to LNT, respectively. The hardening rate is higher at LNT than that at RT.
- (2) The main deformation microstructures at cryogenic temperature are twin-twin interactions and multiples slips, while tensions twins and basal slips dominate at RT. The significant improvement of strain to failure and strength would be attributed to abundant twin-twin interactions with active multiple slips at cryogenic temperature.
- (3) The KAM value, namely dislocation density, is high near boundaries of twin-twin interactions at cryogenic temperature, while the KAM value near tension twins at RT is low. This would contribute to the higher strength and hardening rate at cryogenic temperature.

Data availability

The raw/processed data required to reproduce these findings cannot be shared as the data also forms part of an ongoing study.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

We are grateful for the financial support from the President's PhD Scholarship of Imperial College London. The authors would like to thank the funding support by EPSRC under the Grant Agreement EP/R001715/1 on “LightForm: Embedding Materials Engineering in Manufacturing with Light Alloys”.

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