Experimental investigation of the viscoplastic behaviours and microstructure evolutions of AZ31B and Elektron 717 Mg-alloys

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HIGHLIGHTS

• Viscoplastic behaviours of AZ31B and Elektron717 alloys under hot stamping conditions were studied.
• The ductility of AZ31B and Elektron717 increased with strain rates and temperatures.
• Rare earth elements in Elektron717 suppressed the recrystallization and grain growth.
• Analyses of the grain size, texture and geometrically necessary dislocations in the hot deformation were performed.

GRAPHICAL ABSTRACT

ABSTRACT

An insight into the thermo-mechanical behaviours of AZ31B and Elektron 717 magnesium alloys under the hot stamping conditions was established. High-temperature tensile tests (i.e. 350–450 °C) at a strain rate of 0.1 to 5/s were conducted to examine the material viscoplastic behaviours. Additionally, microstructure characterizations were performed, using the electron backscatter diffraction (EBSD), on the deformed samples to capture the underlying deformation mechanisms. Dynamic recrystallization (DRX) and texture formation were observed during the deformation at high temperature in both alloys and are the primary factors that affect the viscoplastic behaviours. The yield stress of both alloys reduced with increasing temperatures and reducing strain rates. More importantly, the ductility of the samples increased with both the temperatures and the strain rates. The higher ductility at higher strain rates was primarily attributed to finer grains and the slightly weakened textures, enabling a more uniform deformation. A maximum ductility of ~2 was observed in AZ31B under 450 °C at 1/s while ~0.9 in Elektron 717 under the identical condition. The addition of rare earth elements in Elektron 717 may suppress the active DRX. The recrystallization type was identified as discontinuous DRX. The research findings deliver understandings on the viscoplastic behaviours and the deformation mechanisms of AZ31B and Elektron 717 under the hot stamping conditions and provide scientific guidance for feasibility study on applying hot stamping technique to Mg-alloy for forming complex geometry components.

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1. Introduction

Reducing the energy consumptions and strictly controlling the CO₂ emissions of the passenger vehicles are of great importance, which attracts a large number of academic and industrial researchers to develop lightweight materials with moderate strength and ductility. Magnesium alloys are one of the most promising lightweight materials, attracting many interests, due to its low density and high specific strength [1]. However, the application of magnesium alloys, particularly the magnesium alloys sheets, is far behind that of aluminium alloys and rarely used in the automotive industry. The main factors that limited their wide applications are the poor ductility and the strong textures [2,3]. Although some researchers successfully produced some vehicle panels by various forming techniques, such as superplastic forming [4], the costs of these technologies are generally high. Therefore, to aid developing better magnesium forming techniques with low costs and high efficiency, it is necessary to fundamentally understand the thermo-mechanical behaviours and, more importantly, to gain an insight into the underlying mechanisms of Mg alloys during deformations at various temperatures and strain rates, such that an advanced method can be achieved to improve the ductility and weaken the texture.

Research has been performed to study the thermo-mechanical behaviours of Mg alloys [5–10]. Both experimental and simulation work has been performed to capture the basic stress-strain relationship and investigate the corresponding deformation mechanism. Watanabe et al. [6] studied the deformation behaviour of a coarse AZ31 magnesium alloy sheet at elevated temperatures and found that the material exhibited high ductility of 196% at 648 K and 3 × 10⁻³ s⁻¹. They attributed its high ductility to the deformation mechanism of grain-controlled dislocation creep. Superplastic behaviours of AZ61 and AZ31 alloy sheets were evaluated in the temperature range of 573–693 K for rolling processes [7]. Wu et al. [8] investigated the super-plasticity of coarse-grained magnesium alloy, and elongation of 320% was obtained at 773 K and a strain rate of 1 × 10⁻³ s⁻¹. Jäger et al. [9] found that the ductility of AZ31B increased rapidly with the increased temperatures and attributed this to the dislocation glide and deformation twinning. H. Mirzae et al. [11–13] also did a comprehensive work on studying the viscoplastic behaviours of Mg alloys, e.g. AZ31, AZ61, AZ60, etc., focusing on achieving the constitutive relationships during hot deformations at a wide range of temperatures (~150–500 °C) and strain rates for thermal-mechanical applications. The material hardening behaviours during the hot deformation were quantitatively analysed by adapting the power-law relations to the experimental data, achieving the Zener-Hollomon parameters and providing valuable data for understanding the hot deformation behaviour of Mg-alloy. Tensile tests at quasi-static and high strain rates for AZ31B magnesium alloys were conducted to characterize the strain rate sensitivity, which would be applied in high rate deformation or high velocity forming processes. The temperature range was from room temperature to 250 °C [14]. There is also research on the mechanical behaviours of Mg rare earth alloys [15,16]. The influence of rare-earth elements resulted in weaker texture, refined grain size and second-phase particles, which would be responsible for its high strain hardening rate in Mg-10Gd-3Y-0.5Zr alloy during compression tests [15]. On the other hand, the thermo-mechanical modelling work, containing physical-based microstructural internal state variables, was also performed by a range of researchers, e.g. Lin et al. [17,18], Zheng, et al. [19], Li et al. [20] to develop unified constitutive equations to model the microstructure evolution under hot forming conditions. Wang et al. [21] demonstrated the significance of microstructure evolutions during the hot forming, successfully taking its effects into the material model, and effectively predicted the formability for the hot gas forming application. The microstructural evolution of metals such as grain size, dislocation density at different strain rates and temperatures could be predicted. The existing research findings are appropriate references for understanding the thermo-mechanical behaviours of some alloys for the warm forming application [6,22]. However, the thermo-mechanical behaviours of the Mg alloys at high temperatures and high strain rates, especially under hot stamping conditions [5,23,24], where a relatively high deformation temperature and high strain rate are required, are not well understood. More importantly, the corresponding microstructure changes and the deformation mechanisms are still unclear.

Dynamic recrystallization is one of the key phenomena dominating the hot deformation behaviour. During DRX, grain size and texture evolve due to the hot deformation, which concurrently removes defects like dislocations. In turn, the evolved grain size and texture also simultaneously determine the material hot flow stress-strain responses [21]. Research on understanding the dynamic recrystallization in Mg-Al-Zn magnesium alloys have been performed in the hot deformation process [11,25–27]. The dynamic recrystallization of AZ31 Mg alloys was preferably nucleated around contraction or double twin boundaries rather than the extension twin under uniaxial compression at 250 °C [26]. According to [28,29], the dynamically recrystallized fractions and the grain sizes increased with temperatures while decreased with strain rates. The dynamic recrystallization also determines the texture formation during the hot deformation, where strong basal textures are likely to be formed and inhibit further deformation, as suggested in [30,31]. The addition of rare earth (RE) elements was found to weaken the texture formation [32–34]. A solute drag pressure of segregated RE element Y on migrating boundaries was expected to suppress DRX requiring boundary migration using classical models [35]. Additionally, weakened texture and improved formability were also observed for Mg-alloy, containing RE [36] and Gd elements [37,38], from observations on the microstructure distributions [34] and hot compression stress-strain relations [38]. Hot extrusion was found to enhance the ductility of the Mg-alloy, containing Gd elements [37]. However, there are still lacking research to comprehensive investigate the recrystallization, texture formation, the effects of additions, etc., and more importantly, to correspond these microstructure distributions to the hot deformation behaviours of Mg alloys, especially for that containing RE additions.

This study fills the abovementioned research gap, focusing on the thermo-mechanical behaviours of two Mg alloys, i.e. AZ31B (conventional Mg-Al-Zn alloy) and Elektron 717 (containing RE elements), which are widely used for manufacturing automotive panels, under the hot stamping conditions. High temperature uniaxial tensile tests were performed at various temperatures and strain rates to achieve the stress-strain relations using a Gleeble 3800. Additionally, insights into the deformation mechanisms in these behaviours were achieved from the EBSD observations, which reveals the evolutions of the DRX, grain growth, texture and geometrically necessary dislocation densities.

2. Experimental method

2.1. Material and sample design

The material used in this research are AZ31B (Mg-Al-Zn alloys class) and Elektron 717 (ZE10A, Mg-Zr-rare earth alloys class) sheets with a thickness of 1.2 mm, provided by Magnesium Elektron. The chemical compositions are given in Table 1. AZ31B and Elektron 717 alloys are both wrought magnesium sheet alloys that are normally used in the warm forming process in automotive industries. The sample geometry of the as-received AZ31B and Elektron 717 is shown in Fig. 1. The samples were manufactured using the electro-

| Table 1 Chemical compositions of AZ31B and Elektron 717 (in wt%).
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<td>Zr</td>
<td>Mn</td>
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<tr>
<td>AZ31B</td>
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<td>3.03</td>
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<td>Elektron 717</td>
<td>&lt;0.001</td>
<td>1.18</td>
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discharge machine (EDM) with their principal axis parallel to the rolling direction.

2.2. High-temperature tensile tests

The tensile tests at high temperatures were conducted using the Gleeble 3800 thermo-mechanical simulator. The Gleeble 3800, equipped with a resistant heating and cooling system, is commonly used for studying thermo-mechanical behaviours of metals in different practical metal forming conditions. Before the high-temperature tensile tests, four pairs of thermocouples were welded to the specimen at 0, 2, 4, 6 mm from the centre of the samples, along the gauge length direction, to record the temperature distributions in the gauge length area. Based on the recorded temperature distributions, the effective gauge length of 6 mm was used for the following calculation of equivalent true strains.

In preparation for the tensile tests, a pair of thermocouples was welded to the centre of the specimen surface to measure the temperature and to provide feedback signals to the Gleeble3800, such that the designed heating rate and the target deformation temperature were accurately controlled. Additionally, a C-gauge was attached to the middle of the specimen to record the strain of the determined gauge length during the deformation. The thermocouples and the C-gauge are schematically shown in Fig. 1.

High-temperature tensile tests were performed at a strain rate of 0.1/s to 5/s under the temperature, ranging from 20 °C to 450 °C, and tensile tests at room temperature were performed as a reference. The selected strain rates and temperatures were expected to cover the most range of industrial Mg hot forming/stamping conditions [30,33]. It is also the fastest cooling method (i.e. ~300 °C/s) that can be used in Gleeble, thus was selected as the quenching method for the tests. Considering the high cooling rate of 5 °C/s, water quenching is one of the most efficient and common cooling methods to retain the deformed microstructure [30,33] for the subsequent microstructure observation. Hence, considering the testing conditions and the facility, water quenching is the best method to retain the deformed microstructure for the subsequent microstructure observation. Stress-strain curves during the high-temperature deformation were recorded, and the fractured samples were prepared for the subsequent EBSD observations.

2.3. Electron backscatter diffraction (EBSD)

EBSD characterization was conducted for as received and deformed AZ31B and Elektron 717 alloys samples at 20 °C, 1/s; 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s. For each condition, the EBSD scanning area was located at the position where a similar nominal true strain of ~0.3 (estimated by the width reduction) was found on the fractured samples. These samples were firstly ground gradually using SiC paper from 800 grit to 4000 grit. Then OPS suspension was used during polishing for at least 40 min to achieve a mirror surface finishing. Gatan Precision Echting Coating system (PECs) was then conducted to polish the free surface and remove possible contaminations. A 558 μm × 418 μm EBSD map with 1 μm step size at ×500 magnification was obtained using a Bruker e−FlashHR detector and Quantax Esprit 2.1 system. A 20 keV voltage was applied in a Zeiss Sigma SEM. The grain size and texture were recorded, and the fractured samples were prepared for the subsequent EBSD observations.

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calculated. Points with kernel average misorientation (KAM) value of <2° was recognised as recrystallized zones. The ratio between the recrystallized area with the overall EBSD map area was recognised as recrystallized volume fraction.

3. Experimental results

3.1. Initial microstructures of AZ31B and Elektron 717 alloys

The IPF maps of the as-received AZ31B and Elektron 717 alloys are shown in Fig. 3(a) and (d), respectively. The grain size distributions for the two alloys were calculated by area fraction, excluding boundary grains, and are presented in Fig. 3(b) and (e). In general, the average grain size (i.e. 12.5 μm) of AZ31B is slightly smaller than that (i.e. 17.6 μm) of Elektron 717. The texture of the as-received AZ31B, as shown in Fig. 3(c), concentrates in the centre of the {0001} pole figures and is much stronger than that of as-received Elektron 717, as shown in Fig. 3(f), whose texture distributes along the transverse direction (TD) in the {0001} pole figures.

3.2. Thermo-mechanical behaviours at different strain rates and temperatures

Fig. 4 shows the tensile curves of AZ31B and Elektron 717 alloys at different strain rates and temperatures. As shown in Fig. 4(a), the ultimate tensile and yield stress of both AZ31B and Elektron 717 decreases with increasing temperatures at the same strain rate of 1/s. The yield stress of Elektron 717 (i.e. around 270 MPa) is higher than that of AZ31B (i.e. around 225 MPa) at 20 °C, but the ultimate tensile stress is much lower. Both the ductility of Elektron 717 and AZ31B go up with increased temperatures. The AZ31B Mg alloys exhibited a maximum ductility of about 2 at 450 °C, 1/s, which is almost 5 times of that at 20 °C, 1/s. Higher ductility for AZ31B compared to Elektron 717 is observed in all cases. The ductility of >0.6 at the elevated temperatures (i.e. ≥350 °C) for both alloys is expected to be sufficient for most of the forming applications where the geometry of the component is not too complex.

Fig. 4(b) shows the stress–strain curves of AZ31B and Elektron 717 under various strain rates at 350 °C. As expected, both the yield and ultimate tensile stress rise up with the increase of strain rates due to the strain rate hardening effects [23]. In terms of AZ31B, the ductility

Fig. 3. The as-received microstructures of (a–c) AZ31B and (d–f) Elektron 717. Note that (a, d) are the EBSD IPF maps. (b, e) are the analysed grain size distributions from the corresponding EBSD IPF maps in (a) and (d), respectively. (c, f) are the corresponding (0001) pole figures.
increased monotonically, from 1.5 to 1.9, within the studied strain rate range, from 0.1/s to 5/s, due to the facilitated recrystallization, which will be described in the next section. Considering Elektron 717, a slight increase in the ductility, from 0.7 to 0.9, is also observed with higher strain rate.

The corresponding strain hardening rate curves of AZ31B and Elektron 717 are shown in Fig. 5. The hardening rate sensitivities of AZ31B to both the temperatures and the strain rates are higher than that of Elektron 717. Comparing the hardening rate curves in Fig. 5 (a) with (c), the hardening rate of both AZ31B and Elektron 717 decreases with the increasing temperature. A much higher hardening rate, up to 800 MPa/unit strain was observed in AZ31B at 20 °C, while the hardening rate was only up to 600 MPa/unit strain in Elektron 717. The strain hardening rate sensitivity of AZ31 to the temperature is higher than that of Elektron 717. Fig. 5(b, d) presents the hardening rates of AZ31B and Elektron 717 at 0.1/s to 5/s. With the strain rate increasing, the hardening rate of both material increases. However, the hardening rate of Elektron 717 is less sensitive to strain rates, as observed in Fig. 5(d), where no apparent differences were observed for the hot deformed Elektron 717 at various strain rates, especially when the true strain level increased to 0.2.

3.3. Grain size distributions at different strain rates and temperatures

Fig. 6(a–c) shows the EBSD IPF maps of deformed AZ31B samples at 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s. The quantitative analysis of the grain sizes at the corresponding conditions is also given in Fig. 7. Comparing the grain size distributions in Figs. 6(a) and 3(a), it can be observed that the grain size increased after the hot deformation at 350 °C, 1/s. Additionally, the grain sizes slightly increased with the increase in temperatures from 350 °C to 450 °C, comparing the grain size distributions in Fig. 6(a) and (b), where the average values slightly increased by ~1 μm. This is attributed to the faster grain growth at higher temperatures. Considering the uniformity of the grains, it can be observed that a
bimodal grain structure, consisting of coarse and fine grains, presents in the sample deformed at 350 °C, 1/s, while more homogeneous grains present in the sample deformed at 450 °C, 1/s, in Fig. 6(b). The homogeneous grain structures are beneficial to the uniform deformation of the material, thus higher ductility was achieved for 450 °C, 1/s in Fig. 4(a).

Considering the strain rates effects on the material microstructure, smaller (i.e. ~14 μm) and more homogeneous grains were observed in the sample deformed at 350 °C, 5/s (Fig. 6(c)) than that (i.e. ~18 μm) at 350 °C, 1/s (Fig. 6(a)). The homogeneous microstructures with smaller grain sizes in samples at 350 °C, 5/s may be one of the main factors that enhanced the material ductility, as observed in Fig. 4(b).

The EBSD IPF maps of deformed Elektron 717 samples at 20 °C, 1/s; 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s are shown in Fig. 6(d–f). The grain size of Elektron 717 samples at 350 °C, 1/s, 450 °C, 1/s and 350 °C, 5/s becomes smaller than that of initial Elektron 717 sample (in Fig. 3(d)), indicating the occurrence of recrystallizations. The grain size reduction of the Elektron 717 sample at high temperatures indicates that the rare earth element in Elektron 717 may suppress the grain growth, and the grain growth restriction is related to grain boundary pinning effects caused by grain segregation of rare earth alloys [40].

Fig. 7 gives a quantitative analysis of the average grain size in AZ31B and Elektron 717 under identical hot deformation conditions. As described, for AZ31B, average grain size increased, from ~12.5 μm (initial grain size) to ~18 μm, after the material deformed at 350 °C, 1/s. The increase in the grain size of AZ31B samples deformed at high temperatures may be attributed to its fast grain growth. Tan et al. [41] also found that the grain refinement of AZ31 during DRX was insignificant at high temperature due to the rapid grain growth, and showed a maximum value at 250 °C. With the temperature increasing to 450 °C, the average grain size slightly increased to 19 μm. The grain becomes smaller in AZ31B at higher strain rates, where the grain size significantly decreased from ~18 μm to ~14 μm when the strain rate increased from 1/s to 5/s.

Comparing to the initial grain size of Elektron 717 samples, the grain size of Elektron 717 samples deformed at high temperature all decreased, due to the occurrence of DRX. The average grain size of Elektron 717 at 350 °C decreased to ~12.5 μm from an initial grain size of 17.6 μm. Considering the temperature effects, a slight increase in the average grain size from ~12.5 μm to ~13.5 μm was also observed with higher deformation temperatures from 350 °C to 450 °C. Considering the strain rates effects, a similar trend to that of AZ31B was observed, where the grain slightly decreased at higher strain rate. The results are consistent with reports from Fatemi-Varzaneh et al. [42], in which the size of the dynamically recrystallized grains increased by increasing temperature and reduced by increasing strain rate.

Comparing the average grain sizes of Elektron 717 and AZ31, it can be observed that the average grain size of Elektron 717 is always much smaller than that of AZ31B under identical conditions, except the initial states. The grain growth delayed in Elektron 717 due to the addition of rare earth elements, resulting in grain boundary pinning effects caused by solute segregation or particles [40].

3.4. Textures at different strain rates and temperatures

The texture is also related to the thermo-mechanical behaviours of Mg alloys. Fig. 8(a–c) shows the texture of deformed AZ31B samples at 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s. The (0001) pole figures show that the typical as-received basal texture retained and reinforced in deformed AZ31B samples in all these conditions. Compared the texture formed at 350 °C, 1/s with 450 °C, 1/s, the stronger texture is formed.

Fig. 6. EBSD IPF maps at different strain rates and temperatures of (a–c) AZ31B at 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s and of (d–f) Elektron 717 at 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s.

Fig. 7. Average grain sizes of AZ31B and Elektron 717 at initial, 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s conditions.
at higher temperatures. No significant change was observed at different strain rate.

Fig. 8(d–f) plots the (0001) pole figures of deformed Elektron 717 samples at 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s. These figures show that the texture is much weaker than that of deformed AZ31B samples at the same condition, and the texture mostly distributes along TD direction. The strongest texture of Elektron 717 was also observed at 450 °C, 1/s in Fig. 8(e). Rare differences were observed comparing different strain rates, as shown in Fig. 8(d, f), indicating that texture is less insensitive to strain rate than temperature. According to these pole figures, the anisotropy of Elektron 717 is expected to be much weaker than that of AZ31B. Therefore, Elektron 717 is expected to be a more promising Mg alloy for forming under multiaxial stress state where the anisotropic properties is a large issue.

Fig. 9 plots the maximum value of texture in AZ31B and Elektron 717 samples at the initial state and deformed conditions, namely initial, 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s. Considering the maximum texture of AZ31B, it became larger after the deformation, where the value increased slightly from ~12 (i.e. initial value) to ~14 after deformation at 350 °C. Additionally, the texture became stronger at a higher deformation temperature, where the maximum value increased from ~14 to ~16. The stronger texture at a higher temperature may due to its higher recrystallized fractions. The newly recrystallized grains tend to grow in a similar direction, as given in Fig. 12, thus leading to an increased maximum texture [43]. At higher temperatures, the contribution of the grain boundary sliding mechanisms may also increase [44]. Hence, the grains may rotate or slide in a more consistent direction during the higher temperature deformation and thus stronger textures.

Higher strain rates reduced the maximum texture, comparing the texture value (i.e. ~14) at 350 °C, 1/s in AZ31B with that (i.e. ~13) at 350 °C, 5/s. Similar trends of the texture change at high strain rate was also observed in a thermo-mechanical plane-strain rolling process of Mg-Zn-Zr alloy [45]. The weaker texture of AZ31B alloys deformed at 5/s, 350 °C was speculated to result from the more uniform grains (Figs. 6, 7 and 9). These slightly weakened textures may contribute to the improved ductility in the sample deformed at high strain rate.

The trend of the maximum textures of Elektron 717 under various conditions is similar to that of AZ31B, while the texture values are much lower than that of AZ31B in all conditions. Rare earth elements were observed to segregate to the grain boundaries [46] due to the large atomic size misfit between the magnesium and rare earth elements [35]. Thus, the texture weakening behaviours in Elektron 717 may be attributed to the boundary pinning effects caused by solute segregation or particles [40].

Fig. 8. (0001) pole figures at different strain rates and temperatures of (a–d) AZ31B at 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s and (e–f) Elektron 717 at 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s.

Fig. 9. Textures of AZ31B and Elektron 717 at initial, 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s conditions.
3.5. Dynamic recrystallization at different strain rates and temperatures

In thermo-mechanical tests of Mg alloys, dynamic recrystallization plays an important role in their mechanical behaviours and has an influence on the texture change. Therefore, the recrystallized volume fraction was calculated and presented in Fig. 10. The recrystallized volume fraction of AZ31B is much higher than that of Elektron 717 in all conditions, indicating that the DRX was suppressed in Elektron 717 [35,47,48]. For AZ31B, the recrystallized fraction of deformed samples at 450 °C, 1/s is much higher than that at 350 °C, 1/s, indicating that the deformation at higher temperature contributes to a larger recrystallized fraction. However, with the strain rate increasing from 1/s to 5/s, the recrystallized fraction of AZ31B is almost unchanged, although the annealing time at a strain rate of 1/s is much longer than that at 5/s. In high strain rate deformation, the DRX of magnesium alloys is generally improved [45,49], and the grain growth is suppressed [40]. More homogeneous microstructure with fine grains is formed at high strain rate, which would contribute to the higher ductility in AZ31B sample deformed at 350 °C, 5/s, compared to the sample deformed 350 °C, 1/s (Fig. 4(b)).

In order to analyse the behaviour of the recrystallized grains in AZ31B and Elektron 717, the recrystallized grains were detected with the recrystallized fraction component of HKL CHANNEL 5 software. The grains with average angle exceeding 2° is classified as deformed grains, and some grains whose sub-grains were below 2° but misorientation between subgrains was above 2° were identified as sub-structured grains, the remaining grains were recrystallized grains [50]. (0001), (10T0) pole figures of parent grains in AZ31B and Elektron 717 at 450 °C, 1/s are shown in Fig. 11(a, b). Typical basal fibre texture is formed in AZ31B [51], and Elektron 717 shows a rare earth texture with weak intensity. The texture in Elektron 717 is mainly distributed along the transverse direction (TD) in (0001) pole figure, and two parts of the texture are located in the two poles along rolling direction (RD) in (10T0) pole figure. The recrystallized grains in AZ31B and Elektron 717 exhibit a similar texture distribution to the parent grains. Nevertheless, the texture intensity in recrystallized grains is much weaker.

To qualitatively examine the recrystallization process and identify the relationship of orientation between the parent grains and the recrystallized grains, typical areas were selected and analysed in Fig. 12. The recrystallized grains in AZ31B, e.g. A1, A2, A3 are located around the parent grain (PA) and shows similar orientation to the parent grain (PA), in which their c-axis are parallel to the normal direction as shown in Fig. 12(a). In Fig. 12(c), the recrystallized grain A1, A2, A3 and parent grain PA are all located in the centre of the [0001] pole figures, resulting in basal texture. Whereas in Elektron 717, the recrystallized grains B1, B2, and B3 are all close to the parent grains PB1, PB2, PB3 were highlighted in Fig. 12(d) which exhibit similar orientation to the corresponding parent grains. As shown in Fig. 12(f), the position of recrystallized grains is near their parent grains in (0001) pole figure, and these grains are distributed along the TD direction, forming weak RE texture instead of strong basal texture [52]. The DRX in these two alloys during the thermal-mechanical tests forms similar texture distributions, which are due to the similar orientation formation between parent grains and recrystallized grains. The DRX generally included three types according to various temperature ranges. For example, in ZK60 Mg alloys, DRX was associated with twinning and dislocation.

![Fig. 10. Recrystallized volume fraction of AZ31B and Elektron 717 at 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s.](image1)

![Fig. 11. (0001), (10T0) pole figures of (a) all grains in AZ31B, (b) all grains in Elektron 717, (c) recrystallized grains in AZ31B, (d) recrystallized grains in Elektron 717 at 450 °C, 1/s.](image2)
slips at low temperatures (below 473 K), continuous DRX (CDRX) was observed at intermediate temperature range (473 K–523 K), and discontinuous DRX (DDRX) dominated at high temperatures (573 K–723 K) [53]. Discontinuous DRX and continuous DRX were both observed in hot compression of AZ31 Mg alloys [29]. The DRX mechanisms in these thermo-mechanical tests are speculated to be discontinuous DRX (DDRX), in which the new grains are formed by bulging of low-angle grain boundaries (LAGBs) and divided from the original grains [28,54,55], similar crystal orientations are retained in the parent and recrystallized grains.

3.6. GND at different strain rates and temperatures

In general, geometrically necessary dislocations (GNDs) dominate work-hardening process in inhomogeneous deformation of polycrystals, because GNDs are generated rapidly and exceed statistically stored dislocations (SSDs) in some cases [56,57]. In order to analyse the dislocation change during these thermo-mechanical tests, GNDs were obtained from local orientation measurements on planar surfaces using MTEX [58]. According to [58], five components of the dislocation tensor and one difference between two other components can be determined from the local orientation measurements, and these six components can be applied to extract the geometrically necessary dislocation content by linear optimization. For clarity, the GND calculated here were used to study its distribution, and more importantly to achieve the quantitative values under different conditions. The EBSD maps have been proved to quantitatively represent the total dislocation density in polycrystalline, compared with transmission electron microscope (TEM) [59], transmission Kikuchi diffraction (TKD) [60] and surface pitting [61] methods. Additionally, the recent studies also showed that the GND density calculated from EBSD is consistent with the crystal plasticity finite element modelling [62,63]. Therefore, considering achieving both the quantitative values and the distributions, the EBSD recovered GND maps are valuable for exploring the deformation mechanism and corresponding to the micro response of interest (i.e. stress-strain relationship).

Fig. 13 shows the 558 μm × 418 μm EBSD derived GND density maps at 350 °C, 1/s; 450 °C, 1/s; 350 °C, 5/s. The GND densities of deformed AZ31B and Elektron 717 samples are much higher than that of the initial AZ31B and Elektron 717 samples. In these deformed samples, the GND density of recrystallized grains is much lower than that of unrecrystallized grains, because the new recrystallized grains nucleate and grow at the expense of regions full of dislocations. The GND densities of deformed AZ31B and Elektron 717 samples at room temperature are much higher than those at a higher temperature in which recovery and dynamic recrystallization occur. In addition, the GND density is much higher along the grain boundary than that in the centre of the grain. Similar results were reported in the deformed copper [56]. Comparing the GND density in here are more sites with high GND density in Elektron 717 samples at high temperature (ig. 13(d–f)), compared to the corresponding AZ31B samples (Fig. 13(a–c)). The magnified figure in Fig. 13(b, e) shows this difference more clearly. For clarity, the step size for the manified figure is 0.4 μm to achieve detailed GND distributions.

In Fig. 14, the GND density of samples at 20 °C, 1/s is much higher than that at high temperatures, and the GND density decreases with the temperature rising at the same strain rate. Recrystallization and annealing during these thermo-mechanical processes contribute to the decrease of GND density [28]. The flow stress and strain hardening rate in these two Mg alloys, especially AZ31B alloys, decrease with temperature increasing, but their ductility increases with temperature increasing, because of more active recovery and dynamical recrystallization at a higher temperature which results in lower dislocation density. In addition,
compared the GND density of samples deformed at 350 °C 1/s with the samples deformed at 350 °C 5/s, the GND density is higher at the higher strain rate 5/s, which contributes to higher hardening, especially in AZ31B alloys (Fig. 5(b)). The GND variation of Elektron 717 is similar to that of AZ31B, indicating that a similar trend for dislocation variation in the thermo-mechanical process is expected to occur in these Mg alloys. The GND of AZ31B is lower than that of Elektron 717 at high temperatures, which may be attributed to less active DRX in Elektron 717 than that in AZ31B.

4. Discussion

As shown in Fig. 4(a), the ductility of AZ31B increases while the yield stress decreases with the increase in temperatures, especially from the room temperature to 350 °C. The magnesium crystals generally possess two independent easy slip systems, both including the slips of dislocations with \( \frac{a}{N} \) type Burgers vectors within the (0001) basal plane, which fails to satisfy the Taylor criterion requiring five independent easy slip systems [64]. Therefore, the ductility of magnesium at room temperature.
temperature is poor. At high temperatures, the non-basal slip systems were activated, due to lower critical resolved shear stress (CRSS) for these non-basal slips at elevated temperature [64,65], and increased the ductility of AZ31B. In addition, DRX happened at high temperature and further enhanced the ductility of magnesium alloys, which also allowed a decrease in the hardening [18,64]. The ductility of Elektron 717 also shows a sharp increase from room temperature to high temperatures, and its yield stress decreases. However, the strain hardening rate of Elektron 717 is almost independent of temperature. This may be related to the low recrystallized fraction of Elektron 717 at high temperatures, because the addition of RE in Elektron 717 can result in inhibition of DRX [35,47,48]. That may also be the reason why the ductility of Elektron 717 is much lower than that of AZ31B, because more active DRX in AZ31B improves its ductility effectively. The suppressed DRX in Elektron 717 may be attributed to the significant solute drag pressure of rare earth elements on migrating boundaries. As concluded in [13,35,46], segregations of rare earth elements at grain boundaries were observed, and would be related to the decrease in grain sizes of the Mg-RE alloy. This is due to the large atomic size misfit between rare earth elements with magnesium atoms, producing a significant solute drag pressure on migrating boundaries and thus suppressing DRX [35].

For observed anomalous strain rate effects on the ductility, where the ductility of AZ31B behaves higher at higher strain rates, it mainly resulted from its finer grains and lower texture at a higher strain rate. At higher strain rate, it may provide more nucleation sites like twins for dynamic recrystallization, resulting in a homogenous and fine microstructure with weak texture [45]. These AZ31B and Elektron 717 samples behave higher stress at a higher strain rate, mainly due to their higher GND density in Fig. 14. Therefore, it is possible to achieve much higher flow stress and ductility at a higher strain rate.

Regarding the hardening rate change at various temperatures and strain rates, the hardening rate of AZ31B magnesium alloys decreases with temperature increasing, as shown in Fig. 5. This strain softening is generally attributed to the DRX phenomena [42,66]. The recrystallized fraction of AZ31B samples increases with temperature increasing, so the effects of strain softening become stronger at higher temperatures. For the strain rate effects on the strain hardening behaviours of AZ31B, it shows much stronger hardening at higher strain rate, which is mainly attributed to the higher GND density. In addition, as shown in Fig. 14 (b), the mean GND density is higher in the AZ31B sample deformed at higher strain rate of 5/s, resulting in higher hardening. However, unlike AZ31B, the strain hardening rate of Elektron 717 shows few changes at various temperatures and strain rates, probably due to lower recrystallized volume fraction in Elektron 717 samples, resulting in less softening effects.

For clarity, the microstructure observations were performed on samples after both the hot deformation and the water quenching. Due to the high strain rates, the hot deformation time is similar to the quenching time during which the both DRX and static recrystallization may occur. It is difficult to distinguish the DRX with static recrystallization. In this work, we focused on the effects of deformation temperatures and strain rates on microstructures, where the thermo-mechanical conditions were strictly controlled. Considering the similar cooling time for samples under all conditions, the observed microstructures are expected to clearly represent the effects of hot deformation conditions. On the other hand, due to the required high strain rates, it is difficult to use any in-situ facilities to capture the microstructure evolutions, where deformation occurred within 2 s at a strain rate of 1/s. Gleeble equipped with water quenching is the best possible way to study the thermo-mechanical behaviours at high temperatures, and especially at high strain rates, and retain the microstructures. A similar process was conducted in hot compression tests for a ZK60 Mg alloy at 300 °C and strain rate of 1, 15 and 50 s⁻¹ with water quenching, where dynamic recrystallizations were observed [45]. Though it is difficult to distinguish DRX and static recrystallization, considering the recrystallized grains in Fig. 12, the recrystallized grains in both AZ31B and Elektron 717 alloys exhibit similar orientation to the corresponding parent grains. It is speculated that this recrystallization mode may be discontinuous dynamic recrystallization [28,54,55]. The occurrence of static recrystallization generally needs enough temperature and time. Hence, considering the fast cooling speed during water quenching, as given in Fig. S1, the occurrence of static recrystallizations may be limited.

It may be worth mentioning that, for industrial applications, the product geometries are complex and different from the test specimens, where multiaxial stress state will be applied during forming. Thus, the formability of Mg-alloy under multiaxial conditions is also one of the most concerned issues in the industry. Though the ductility of Elektron 717 in the rolling direction is smaller than that of AZ31B in the rolling direction under uniaxial conditions, as observed in this study and also in [52,67,68], the textures of Elektron 717 developed during hot deformation were much weaker than that of AZ31B. Hence, Elektron 717 is expecting to behave better under multiaxial conditions. As suggested by Boba [69], the formability of ZEK100 Mg alloys (similar to Elektron 717) below 200 °C was better than that of AZ31B from limited dome height test results. This study focused on revealing the deformation mechanism from the macro- and micro- relationship perspective, where the uniaxial tests are more appropriate to capture the hot deformation nature. Considering the industrial interest, future work on formability study under multiaxial conditions at the studied temperatures and strain rates is of value.

5. Conclusions

The thermo-mechanical behaviours of AZ31B and Elektron 717 Mg alloys were studied using a Gleeble 3800 material simulator, and the microstructures of AZ31B and Elektron 717 Mg alloys at different temperatures and strain rates was characterized using EBSD. The following conclusions are drawn:

1. The yield and tensile stress of AZ31B and Elektron 717 Mg alloy reduced with the increase in temperature while increased with the increase in strain rates. However, the ductility of AZ31B and Elektron 717 increased with both the temperatures and the strain rates. The AZ31B Mg alloys showed a maximum ductility of ~2 at 450 °C, 1/s, while ~0.9 for Elektron 717.

2. The increased ductility at higher strain rates was attributed to the smaller and more homogeneous grain formation due to the DRX, and the weakened texture formation. The increased strain-hardening rate was attributed to the higher accumulation of dislocations.

3. The increased ductility at higher temperatures was mainly attributed to the increased DRX volume fractions, despite the larger average grain size and stronger texture. While the reduced yield stress and strain hardening rates were due to the lower GND values at higher temperatures.

4. The addition of rare earth elements in Elektron 717 alloys may suppress the DRX and grain growth during hot deformation, resulting in finer grains, comparing to that of AZ31B. The strain hardening rate of Elektron 717 was less sensitive to temperature and strain rate, due to less softening effects of DRX at these conditions.

5. The DRX type was identified as the discontinuous DRX for both AZ31B and Elektron 717. The texture distributions of recrystallized grains in alloys were similar to that of their parent grains.

CRediT authorship contribution statement

Kai Zhang: Data curation, Formal analysis, Investigation, Software, Validation, Visualization, Writing – original draft, Writing – review & editing. Jing-Hua Zheng: Supervision, Investigation, Validation,
Visualization, Conceptualization, Writing - original draft, Review - viewing & editing. Zhoutao Shao: Data curation, Investigation, Writing - Review & editing. Catalin Pruncu: Investigation, Visualization, Writing - review & editing. Mark Turski: Investigation, Resources, Writing - review & editing. Carlos Guerini: Investigation, Resources, Writing - review & editing. Jun Jiang: Conceptualization, Supervision, Investigation, Validation, Visualization, Funding acquisition, Resources, Review - view & editing.

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Appendix A. Supplementary data

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References


