On the roles of stress-triaxiality and strain-rate on the deformation behavior of AZ31 magnesium alloys

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ABSTRACT
The presence of complex states-of-stress and strain-rates directly influence the dominant deformation mechanisms operating in a given material under load. Mg alloys have shown limited ambient temperature formability due to the paucity of active slip-mechanisms, however, studies have focused on quasi-static strain-rates and/or simple loading conditions (primarily uniaxial or biaxial). For the first time, the influence of strain-rate and stress-triaxiality is utilized to unravel the active deformation mechanisms operating along the rolling, transverse- and normal-directions in wrought AZ31-alloy. It is discovered that the activation of various twin-mechanisms in the presence of multiaxial loading is governed by the energetics of the applied strain-rates.

IMPACT STATEMENT
It is shown for the first time that the higher deformation energy associated with dynamic strain-rates, coupled with high-triaxiality, promotes detwinning and texture evolution in HCP alloys with high c/a ratio.

Knowledge of the stress–strain response of alloys deformed at various strain-rates is important for the overall development of materials, both from the perspective of processing to an end-product. This is specifically true for magnesium (Mg) alloys, which have been identified as a viable light-weighting alternative to iron- and aluminum-based alloys [1] for structural applications. The adaptation of Mg and its alloys to these more advanced applications has been restricted due to limited room-temperature Plasticity, which is a consequence of Mg alloys’ limited ability to activate non-basal slip deformation mechanisms [2]. As a result, concurrent dislocation slip/glide is significantly restricted in Mg and its alloys and twinning plays a critical role in dictating the extent of their deformation and hence applicability for demanding applications.

Twinning is a fundamentally different process than slip as the twin activation energy is a function of several extrinsic and intrinsic phenomena such as the polar nature of stress, refer for further detail in [2,3]. In Mg alloys, the predominant room-temperature deformation twins activated are reported to be \{10\textbar 12\} twins. These extension twins account for the anisotropy exhibited during tension along certain crystallographic axes [4]. Further, when the c-axis is in compression, both \{10\textbar 11\} twin and basal slip have been found to dominate. The same regions, which undergo contraction twinning, are also capable of undergoing extension twinning. This phenomenon of multiple twinning mechanisms leads to a secondary–twinning mechanism during complex loading-states in which twin–twin interaction occur and plays a critical role in controlling the deformation response of Mg alloys [5,6].

The large body of work focusing on the deformability of AZ31 and other Mg alloys has been limited in that they predominantly report deformation mechanisms...
and mechanical behavior under mainly uniaxial (and occasional biaxial) loading conditions, carried out at various strain-rates. This is despite the fact that more often than not, in-service conditions may trigger a multiaxial state-of-stress [7]. For instance, structural applications, such as found in the automobile/aerospace industries, require components that will be subjected to a variety of strain-rates as well as states-of-stress. Therefore, understanding the behavior of Mg alloys under such conditions is of both scientific and technological significance. Work on the dynamic behavior has been performed showing that quasi-static and high strain-rate compression tests on rolled AZ31 reveal a strong dependence of yield strength, hardening and failure strain on the strain-rate when testing only in the normal direction (ND) [8]. Further, recent-studies have shown that at quasi-static strain-rates, for a low to moderate-triaxiality, the strain-to-failure is found to be greater in notched specimens than in initially smooth ones [7,9]. This increase in strain-to-failure has been attributed to the change in fracture mode [7,9]. However, a review of the extant literature reveals that no studies have been conducted to address the sensitivity of stress-triaxiality (defined as the ratio of hydrostatic pressure to the equivalent von Mises stress), in combination with strain-rates on twinning and detwinning behavior, and their crucial role in governing the plasticity of Mg alloys.

Hence, in the present study, we capture the deformation mechanisms of AZ31 through varying stress-state and strain-rates, followed by microstructural probes. The very high-rate data were obtained using a split-Hopkinson pressure bar, details of which can be found in [10]. Complex loading-states are achieved through changing the notch radius (Bridgman notch-geometries) within the gauge length of the cylindrical specimens (Figure 1(A), and Table 1 for dimensions) [11,12]. Initially, the specimens are heat treated for strain recovery purposes and are referred to as as-received specimens until further testing is performed, see supplementary document. The as-received microstructure after heat treatment consists of equiaxed grains with an average grain size of $15 \pm 2 \mu m$ with a strong basal texture (see Figure S1 in supplementary information) along the ND (averaged over $\sim 300$ grains).

The un-notched uniaxial tensile and compressive flow stress behavior tested along three different plate orientations subjected to both $10^{-3} \, s^{-1}$ and $\geq 10^3 \, s^{-1}$ strain-rates are plotted in Figure S2 in the supplementary document. These results compare well with literature. In order to understand the combined effect of stress-triaxiality and strain-rate, quasi-static ($10^{-3} \, s^{-1}$) and high strain-rate ($10^3 \, s^{-1}$) tensile tests were performed on notched cylindrical samples (Figure 1(A)). Figure 1(B) presents the load-displacement behavior for

Figure 1. (A) Schematic of the Bridgman notch cylindrical specimens along with the un-notched tension specimen. Mechanical responses for the different notch-geometries along (B) the RD; (C) the TD; and (D) the ND.
all the notch-geometries along the RD. The small-notch cylindrical specimen (green curve with ‘o’ marker in Figure 1(B)) tested at a strain-rate of (10³ s⁻¹) shows a distinct sigmoidal behavior when compared with the rest of the curves. The small-notch specimen has the highest-initial-triaxiality (1.68) of all the geometries (Table 1) which, when combined with dynamic strain-rates, results in a transition from near elastic-perfectly-plastic response to a sigmoidal response (the green curve with ‘o’ markers in Figure 1(B)). Changing the loading from quasi-static to high-rate loading of the small-notch sample clearly changes the deformation mechanisms, and results in a significant decrease in the displacement-to-failure. On the other hand, for the medium- and larger-notch geometries the difference is minimal.

In the case of the TD specimens (Figure 1(C)), stress-triaxiality coupled with strain-rate does not present any kind of anomaly. The displacement-to-failure for the medium-notch (triaxiality of 1.34), however, increases when the strain-rate is increased, which suggests a change in fracture mode, as shown in [7]. For all the specimens oriented along the ND (Figure 1(D)), a pronounced sigmoidal-shape is seen in the load-displacement response which has been shown to be indicative of twin dominated plasticity [13]. The sigmoidal mechanical response observed is true for all notch-geometries and at all strain-rates tested along the ND. Therefore, to investigate the anomalous behavior along the RD of the small-notch cylindrical specimen, we performed a very high-rate test (≈10⁴ s⁻¹, Figure 2(A)). When increasing the strain-rate, a significant transition in the deformation mechanism can be inferred as the sigmoidal-shape of the load-displacement curve becomes even more prominent. To further address this transition, post-deformation light microscopy, XRD and EBSD scans were obtained near the high-triaxiality-region for the small-notch specimens tested along the RD under different strain-rates. For the remaining discussion, we refer to microstructure characterization along the RD and any other microstructure characterization is referred to along with the plate direction along which it was studied.

The load-displacement curves as a function of strain-rates, post-deformed light microscopy and XRD results performed along the RD are shown in Figure 2. In the quasi-static strain-rate regime (Figure 2(A)), no sigmoidal load-displacement behavior is observed. This implies that the deformation is still controlled by full-dislocation motion at low-strain-rates even with such a high level of stress-triaxiality. However, as the strain-rate is increased, 5 × 10³ and 1.8 × 10⁴ s⁻¹, the load-displacement curves exhibit a distinct sigmoidal behavior, which is associated with the manifestation of twinning/detwinning. The energy absorbed (area under the load-displacement curve) during deformation is found to be 149.81 J, and 276.12 J for 10⁻³ s⁻¹, and 10⁴ s⁻¹ strain-rates, respectively. Note that along the RD, the notch-geometries and corresponding gauge-area listed as a function of strain-rate in Figure 2(A) were the same for all testing conditions. The behavior at dynamic strain-rates qualitatively represents the energy absorption characteristics during collisions.

The XRD was performed on the as-received as well as tested samples along the RD (Figure 2(B)). The XRD spectra of the as-received and post-deformed specimens at 10⁻³ s⁻¹ exhibit similar diffraction peaks (with measurements taken just below the fracture surface in the notch section). The relative peak intensities of the specimen tested at 5 × 10³ s⁻¹ for the (100), (002) and (101) fundamental reflections deviate from those obtained for the as-received and quasi-statically deformed specimens along the RD. This change in relative peak intensities is a qualitative indicator of texture evolution resulting from a fundamental change in the deformation mechanisms. Light microscopy micrographs taken from the sample along the RD (Figures 2(C–E)) combined with the mechanical behavior (Figure 2(A)) strongly indicate the likelihood of a transition from full-dislocation mediated plasticity to detwinning dominated. Table S2 presents the area twin density and average twin width calculated from approximately 7–10 regions which are similar to the micrographs in Figure 2(C–E). The as-received initial microstructure shows a large number of grains with pre-existing (1012) twins, however, the deformed microstructure of the sample tested quasi-statically along the RD shows presence of (1012), (1011) and (1013)/(1012) double twins [14] with an average 11.8 μm twin width (Table S2). Quantification of the twins in the high strain-rate deformed sample reveals that the twin density reduces from 4.9 twins μm⁻² to 1.44 twins μm⁻² in the deformed specimen (Figure 2(E)). The statistical twinning information from these light micrographs justify the likelihood of a transition to detwinning dominated plasticity at high strain-rates, as previously inferred from the

| Notch type               | Minimum gage diameter (mm) | Notch radius (mm) | Initial stress-triaxiality |
|-------------------------|---------------------------|------------------|--------------------------|
| Un-notched (uniaxial tension) | 3.25                      | NA               | 0.33                     |
| Large-notch             | 2.0                       | 1.2              | 0.94                     |
| Medium-notch            | 1.0                       | 0.6              | 1.34                     |
| Small-notch             | 0.67                      | 0.4              | 1.68                     |

Note: According to Bridgman’s criteria, the gage diameter to the notch radius was kept constant [11].

Table 1. Dimensions for different notch-geometries and the corresponding initial stress-triaxiality for each type of notch.
Figure 2. (A) Load-displacement curve for the small-notch cylindrical specimen along the RD at various strain-rates. (B) XRD patterns for the as-received and tested specimens along the RD. Light microscopy images of (C) the as-received sample along the RD; a small-notch cylindrical specimen tested at (D) $10^{-3}$ s$^{-1}$ and (E) $5 \times 10^{3}$ s$^{-1}$ along the RD. Note that the XRD pattern of tested sample (B) and the images (D) and (E) were taken just below the fracture surface in the notch sections.

load-displacement responses (Figure 2(A)) and the XRD peaks (Figure 2(B)).

Figure 3 provides the microstructural orientation information through EBSD analysis of the samples tested under various conditions along the RD. The EBSD maps (Figures 3(B–E)) were obtained from the area just below the fracture surface in the gauge-sections. The as-received microstructure (Figure 3(A)) along the RD shows a weak texture (note that the material has a strong basal texture along the ND). Upon characterization of the misorientation angles, the twins present were identified as $\{10\bar{1}2\}$ tensile twins, having misorientation angles of $\sim 86^\circ$ (outlined by black-lines in Figure 3). Under pure tensile loading (un-notched specimen) at $10^3$ s$^{-1}$ along the RD, (Figure 3(B)), $\{10\bar{1}1\}$ twins, having a misorientation angle $\sim 56^\circ$ (outlined by red-lines in Figure 3(B)), are formed along with the pre-existing $\{10\bar{1}2\}$ extension twins. $\{10\bar{1}3\}/\{10\bar{1}2\}$ twins, known as double twins [14] with misorientation angles $\sim 24^\circ$ (outlined by yellow-lines in Figure 3(B)), also form as a result of twin–twin interaction. Figure 3(C) represents the microstructure of the small-notch cylindrical specimen tested at $10^{-3}$ s$^{-1}$ along the RD. The resulting microstructure is like the one shown in Figure 3(B). At quasi-static strain-rates, a combination of tensile twins, compression twins and double twins are observed. Microstructures obtained from post-deformed small-notch samples along RD at higher strain-rates ($10^3$ and $10^4$ s$^{-1}$) are presented in Figures 3(D,E), respectively. No twins are observed in these deformed samples, indicating that a different combination of mechanisms are operative in these strain-rate regimes.

Next, basal (0001) pole figures (based on Figure 3) are plotted in Figure 4. The pole figure for the as-received material along the RD shows a somewhat weak texture. For pure tensile loading (un-notched, cylindrical specimen) at $10^3$ s$^{-1}$, no significant change is observed in the texture along the RD (Figure 4(B)). Figures 4(C,D) represent pole figures for small-notch cylindrical specimens tested under tension at $10^{-3}$, $5 \times 10^3$ and $1.8 \times 10^4$ s$^{-1}$, respectively, along the RD. Figures 4(C,D) suggest there is no major change in texture for $10^{-3}$ s$^{-1}$, and $5 \times 10^3$ s$^{-1}$ strain-rates. However, the sample tested at $1.8 \times 10^4$ s$^{-1}$ (Figure 4(E)) suggests a complete texture change from the as-received condition. Under the conditions of stress-triaxiality at $10^{-3}$ and $10^3$ s$^{-1}$, it can be seen that local complex stress-states do not bring about macroscopic texture change (Figures 4(C,D)) but do affect the deformation behavior at the macrostructure level (Figures 3(C,D)). Higher strain-rates ($10^4$ s$^{-1}$), however, result in a macroscopic texture realignment (Figure 4(E)) which, along with concurrent
Figure 3. EBSD maps along the RD of (A) as-received sample; (B) sample tested at $10^3 \text{ s}^{-1}$ in pure uniaxial tension; and small-notch cylindrical specimens tested at (C) $10^{-3} \text{ s}^{-1}$, (D) $5 \times 10^3 \text{ s}^{-1}$, and (E) $1.8 \times 10^4 \text{ s}^{-1}$ along the RD. These EBSD maps (B, C, D, and E) were obtained from the area just below the fracture surface in the gauge-sections (notch-root).

Figure 4. Basal pole figures (0001) along the RD of the (A) as-received sample; (B) the sample tested at $10^3 \text{ s}^{-1}$ in pure tension; and small-notch cylindrical sample tested at (C) $10^{-3} \text{ s}^{-1}$, (D) $5 \times 10^3 \text{ s}^{-1}$, and (E) $1.8 \times 10^4 \text{ s}^{-1}$ strain-rates along the RD.

microstructural evolution, affect the deformation behavior (see Figure 3(E)).

Figure 5(A) presents the initial microstructure along the RD which is subjected to quasi-static ($10^{-3} \text{ s}^{-1}$) and high strain-rate ($1.8 \times 10^4 \text{ s}^{-1}$) loading, respectively. During the initial stage of deformation, the microstructure is influenced by the induced stress-triaxiality irrespective of the strain-rate. After that two-possible deformation mechanisms dependent on strain-rate are discussed in the following sections.

In the case of quasi-static loading along the RD in small-notch specimen, at several GB (Figures 5(C,D)),
there will be interaction of the \{10\overline{1}1\} twins (formed as a result of induced stress-triaxiality, Figure 5(B)) and the parent \{10\overline{1}2\} twins leading to formation of twin-twin boundaries (TTBs) [15]. The newly formed TTBs play an important role during the deformation of the specimen wherein twin-twin interaction takes place via an impinging twin that transmits onto an existing twin. Thus, in our case, as discussed earlier, the impinging \{10\overline{1}1\} twin transmits onto the existing \{10\overline{1}2\} twin resulting in the formation of double twins having a \{10\overline{1}3\}/\{10\overline{1}2\} nature. Therefore, the resulting microstructure shows traces of three different types of deformation twins as discussed earlier. This mechanism is proposed to drive the deformation at quasi-static-rates where the load-displacement curve shows no signs of sigmoidal behavior (Figure 2(A)). Hence, we observe comparatively higher yield loads and prolonged elongation to failure. However, a potentially different mechanism drives the deformation at higher strain-rates.

The microstructure and texture evolution during high strain-rate deformation along the RD in small-notch specimen is discussed (Figures 5(C’,D’)). In the case of high strain-rate loading, the TTBs (in our case formed as a result of induced stress-triaxiality (Figure 5(B))) will dissociate into twin dislocations [15]. As the deformation proceeds further, the twin dislocations will glide onto the twin plane and facilitate the widening of the twins. Eventually the twins will disappear, leading to detwinning.

This can be further deduced from the load-displacement curves of the small-notch cylindrical specimens tested at different strain-rates (Figure 2(A)) along the RD. Also, detwinning in this case causes texture change from weak to a strong basal texture (Figure 5(D’)). Similar texture evolution is seen for the quasi-static compression along the RD wherein re-orientation of grains occur [16]. In the case of the notched geometries, the induced complex state of stress is present for both quasi-static as well as high strain-rates. However, in the case of higher strain-rate (\(1.8 \times 10^4 \text{ s}^{-1}\), the applied strain-energy is higher as compared to that at \(10^{-3} \text{ s}^{-1}\) strain-rate. Thus, this intensified, complex state of local stress leads to the dissociation and gliding of twin dislocations, resulting in detwinning and eventually a texture change.

The findings discussed above lay an important insight for quantifying the influence of deformation rate on stress-triaxiality. Previously detwinning in the literature has been strongly credited to pure atomic shuffling [17–19]. Proust et al. [17], have shown that detwinning occurs in the case of specimens subjected to strain path changes along the rolling direction (RD) or extruded direction (ED). With AZ31 having a strong tendency for texturing, twinning plays an important role when samples are pre-compressed parallel to the RD or ED leading to the activation of extension twins \{10\overline{1}2\} in the microstructure. On reversal of the strain paths, the twins formed during pre-straining tend to detwin as...
lower stresses are required for detwinning as compared to twin propagation. Similar works [18,19] have shown that detwinning via atomic shuffling occurs as a consequence of strain path changes.

Detwinning via atomic shearing, however, is a less-studied phenomenon in literature. Via a modeling approach, Yu et al. [15] have shown that under normal conditions of stress-states, when multiple twin variants interact, detwinning is an energetically unfavorable phenomenon as compared to twin–twin interaction in hexagonal close packed. Deformation in the presence of stress-triaxiality completely depends on the energetics of deformation. Hence, in summary, this work represents the first experimental-studies that show that during quasi-static loading, the energy during deformation is not sufficient to promote detwinning, whereas at high strain-rates, sufficient energy overcomes the energy barrier required for detwinning and favors detwinning over twin–twin interaction in high states of triaxiality. Detwinning also causes textural evolution from weak to a strong basal texture along the RD.

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