Original Article

Fracture behavior of twin induced ultra-fine grained ZK61 magnesium alloy under high strain rate compression

Abdul Malik\textsuperscript{a}, Wang Yangwei\textsuperscript{a,b,*}, Cheng Huanwu\textsuperscript{a,b}, Muhammad Abubaker Khan\textsuperscript{a}, Faisal Nazeer\textsuperscript{a}, An Rui\textsuperscript{a}, Bao Jiawei\textsuperscript{a}, Wang Mingjun\textsuperscript{c}

\textsuperscript{a} School of Material Science and Engineering, Beijing Institute of Technology, Beijing 100081, China
\textsuperscript{b} National Key Laboratory of Science and Technology on Materials under Shock and Impact, Beijing 100081, China
\textsuperscript{c} Fujian Kunfu Stock Co., Ltd, China

\textbf{A R T I C L E   I N F O}

Article history:
Received 22 January 2019
Accepted 10 June 2019
Available online 28 June 2019

Keywords:
UFG magnesium alloy
Compression
Extension twinning
Formation of ASB
Fracture mechanism

\textbf{A B S T R A C T}

In this study ultra-fine grained single pass extruded ZK61 magnesium alloy sheet is processed at temperature 350 °C with area reduction ratio 30. The complex dynamic mechanical behavior is studied experimentally with Split Hopkinson pressure bar over wide ranges of strain rates (1000–4000 s\(^{-1}\)) along the normal direction. Electron backscattered diffraction, scanning electron microscopy and optical microscopy analysis are employed to reveal the changes in texture and fracture analysis. Positive strain rate sensitivity is observed up to strain rate 3000 s\(^{-1}\) that is signature of basal slip and extension twinning although the grain size was very small. This finding is contradiction to experimental and theoretical predictions suggesting the elimination of twinning induced deformation in magnesium alloys in very fine small grains <3 μm. Flow stress and a fraction of extension twinning are apparently decreased as the applied strain rate increased from 3000 to 4000 s\(^{-1}\); that is attributed to adiabatic rise in temperature, early reorientation of crystal. Extensive grain reorientation causes the nucleation of extension twinning that changes fiber texture to strong double peak basal texture under high strain rate compression. Nucleation of principle crack was attributed to twin–dislocation interaction that provides dynamic recrystallized grains followed by the adiabatic shear band at the boundary of coarse grains. Contrary, secondary cracks are nucleated and propagated due to high local stresses at the triple junction of grain boundaries. Besides, scanning electron microscopy revealed ductile fracture under high strain rate compression.

© 2019 The Authors. Published by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

\section{Introduction}

Magnesium (Mg) being light weigh metal has gained great attention from past few decades. The low density 1.73 g/cm\(^3\), high specific strength, and high specific stiffness make it admirable structural and energy saving material in automotive and aerospace industry [1–4]. Mg alloys possess (0002) basal slip and \{10-10\} prismatic slip system due to hexagonal closed pack (HCP) crystal structure, since both slipping mode, contain \textless a\textgreater type Burgers vector so it is difficult to accommodate plastic deformation at high strain rate [5]. Thus for adequate deformation and to fulfill the Von misses criteria,
additionally (10-12)<10-11> twinning whether it is extension or contraction provide necessary deformation along the crystallographic of c-axis [6,7]. Twinning not only controls overall plastic deformation behavior but it also significantly affects the twin–twin or twin–dislocation interactions and failure mechanism through dynamic texture evaluation caused by crystallographic reorientation [8–10]. For activation of twinning critical resolved shear stresses (CRSS) is already reported by Zhang and Joshi [11] according to their calculation CRSS for basal slip and extension twinning are 0.5 MPa and 2–5 MPa, respectively [12]. Moreover, reported values for prismatic and pyramidal slip are (10–45 MPa) and (35–80 MPa) respectively [13,14]. Thus, it is quite easy to active (0002)<11-20> basal slip and (10-12)<10-11> extension twinning, whereas it is very difficult to active non-basal slips. Some, pyramidal slips were observed in Mg-alloys but under quasi-static loading [15–19].

Pure Mg [20,21] and many Mg alloys such as; AM80 [22], AZ31 [23,24], AZ31B [25], AZ61 & AZ91 [26], EW75M [27], AMX602 [28] and ZK60 [29,30] were studied under high strain rate compression. Most of them were characterized in as casted condition, but porosity put a big question mark on its mechanical properties. So, for requisite superior dynamic mechanical properties wrought alloys fabricated by casting followed by equal channel angular pressing (ECAP) [29], extrusion followed by hot and cold rolling [31–33] and single pass extrusion [23,34] were used to fabricate almost porosity free alloys. Thermomechanical processes are costly and time-consuming. Thus, a single pass of extrusion is far better comparatively because ZK61 contain high wt.% of Zinc (Zn) and Zirconium (Zr) which have the ability to refine the grain size during extrusion, additionally, it stabilizes the MgZn2 precipitates [35].

Dixit et al. [20] studied the dynamic mechanical behavior of an extruded pure Mg and observed extensive grain reorientation and texture hardening that is attributed to (10-12)<10-11> extension twinning. Li et al. [23] studied the extruded AZ31 alloy and observe that the fraction of (10-12)<10-11> extension twinning is high at a strain of 16%. Guo et al. [22] studied cast AM80 alloy and reported that (10-12)<10-11> extension twinning are significantly enhanced under high strain rate compression. Li et al. [30] fabricated an ultra-fine grained (UFG) extruded Mg alloy and reported that high strain hardening is due to the signature of (10-12)<10-11> extension twinning. Thus, (10-12)<10-11> extension twinning and basal slips (0002)<11-20> have predominant role in Mg alloys. UFG Mg alloys seeking special interest because of grain refinement through Zr which is an impressive approach to achieve superior mechanical properties. Nonetheless, grain refinement may also interfere with the combined effect of slip and twinning interaction in Mg alloys.

Up to date, limited investigations are performed on UFG Mg alloys [30], especially on UFG ZK61 Mg alloy under high strain rate compression. The limitation of the work is ascribed to less knowledge of their response under dynamic loading where sudden impact and its resistance are very essential. These factors restricted wider use of Mg alloys as a structural material in military and industrial applications. Previous investigations strongly suggested that the CRSS for twinning nucleation is dependent upon strain rate and grain size [36]. Additionally, Yang et al. [37] studied the microstructure and quasi static mechanical properties of hot-rolled AZ31B and alternate biaxial reverse corrugation (ABRC) processed UFG AZ31B Mg alloy. They improve the formability and reduce the anisotropy of yield strength, which is commonly observed in Mg alloys when they are subjected to tension and compression along extruded direction (ED). They also suggested that twinning should be suppressed at grain size <3.0 μm, whereas deformation was mediated by dislocation slips activity due to high stress concentration at grain boundaries in UFG Mg alloy.

Thus, a few questions originates (1) Does high strain rates favor twinning in ZK61 Mg alloy at very fine grain size 1.5 μm (2) What is the fracture mechanism and how does principal and secondary crack nucleated and propagated in ZK61 Mg alloy. In order to explain aforementioned problems and to reveal complex deformation behavior UFG extruded ZK61 Mg alloy, was characterized by electron backscattered diffraction (EBSD), scanning electron microscopy (SEM) and optical microscopy (OM) analysis.

2. Experimental procedure

2.1. Materials

In the current study investigated material ZK61 (Mg—6.2 wt. %Zn—0.6 wt. %Zr) was casted alloy purchased from “Fujian Kunfu Stock Co., Ltd, China”. The ingots of ZK61 alloy was solution treated at 350 °C for 15 h and subsequently extruded at 350 °C with area reduction ratio 30 and finally a sheet with a thickness 30 mm was fabricated. Later it was aged at 180 °C for 24 h (T5 heat treatment) and cooled down naturally at room temperature (in the whole paper, it will be considered as ZK61 Mg alloy). Fig. 1 shows a schematic illustration of the whole fabrication process.

2.2. High strain rate test

Cylindrical samples with a diameter of φ 8 mm × 8 mm for 1000 s⁻¹ and φ 5 mm × 5 mm for 2000–4000 s⁻¹ were cut along normal direction (ND) and subjected to uniaxial dynamic compression, performed with typical conventional Split Hopkinson’s pressure bar (SHPB) apparatus at room temperature. The details of the experimental technique is discussed in the literature [22]. Before testing samples were lubricated with graphite to reduce friction. For accuracy, three tests were conducted at each strain rate and their average is presented in Fig. 3(a).

2.3. Characterizations

For characterization of ZK61 Mg alloy, initial microstructure analysis was observed with EBSD analysis, while microstructural analysis of fracture specimens was observed with EBSD, OM and SEM. For metallography purpose, fractured specimens were mechanically ground and polished with colloidal silica before etching with acetic picric solution (5 mL acetic acid [CH₃COOH], 5 g picric acid [(HNO₃)₃C₆H₅OH], 10 mL distilled water, and 100 mL ethanol [95%]) as suggested by Guo et al. and Liu et al. [22,38]. The microstructure examination of the etched surface of specimens was observed by using
(ZEISS, AXIO) optical microscopy. For EBSD analysis specimens were prepared by grinding with 5000 SiC paper and subjected to mechanical polishing with a 0.05 mm colloidal silica suspension, and final electrochemical polishing by using the ACZTM commercial electrolyte for 90s at 33V. Hitachi S4800 SEM equipped with an HKL-EBSD system was used to perform EBSD and SEM analysis of initial and deformed samples at strain rate of 3000 and 4000 s\(^{-1}\). HKL Technology Chanel 5 software was used for data acquisition.

3. Results and discussions

3.1. Microstructure and texture analysis

The representative microstructure of ZK61 Mg alloy in ED-TD plane corresponding to crystal reference system investigated by EBSD is shown in Fig. 2(a). It is observed that the grains are very fine and equiaxed of size 1.5 \(\mu\)m. The misorientation distribution map with respect to the corresponding EBSD map is shown in Fig. 2(b). It is observed that majority of the grains are high angle grain boundaries (HAGB) 80% vol. (all other values are presented in Fig. 2(b)). Previous studies suggested that twinning induce deformation is very difficult to observe in a grain of size less than 3 \(\mu\)m, but in this work, a peak at 86.3° is observed that shows the presence of some \{10-12\}<10-11> extension twinning [37]. The intensity of \{0001\} basal texture and its planar homogeneity are very auspicious in finding dynamic mechanical properties of Mg alloys. Fig. 2(c) shows a pole figure analysis of extruded ZK61Mg alloy, where \{0001\} basal pole displays intense tilted extruded fiber texture with maximal intensity 14.5 rooted form hot extrusion. It is evident from pole figure analysis that c-axis of the HCP crystal is partially parallel to (ND) (tilted away from ND to the (ED)) and splitting ±17° toward transverse direction (TD) i.e., a-axis of basal planes are parallel to ED. This result is consistent with the literature [39].

Since most of the applications of Mg alloys components involves external loading subjected along the ND of the sheet. Therefore, in this study, we have applied compressive forces along ND. It is also observed that orientation of most of the grains (calculated from Chanel 5 software) has c-axis partially perpendicular to the compression direction as shown in schematic Fig. 1. This might be a favorable crystal orientation for twinning activity during dynamic compression.

3.2. Stress–strain behavior

High strain rate compression of ZK61 Mg alloy was performed along ND at room temperature. To ensure the repeatability three tests for each strain rate were conducted and scatterng of all average curves are plotted as illustrated in Fig. 3(a).

It is inferred from stress–strain curves of Fig. 3(a) that all curves show positive flow stress response up to 3000 s\(^{-1}\) with peak stress 404 MPa which is 84 MPa increase in comparison of compression at strain rate of 1000 s\(^{-1}\) (served as a reference), these findings are in good agreement with previous studies of Mg alloys [22]. Contrary, at the strain rate of 4000 s\(^{-1}\) the response was quite different, low flow stress 374MPa is observed which is 30 MPa less than that a strain rate of 3000 s\(^{-1}\) as shown in Fig. 3(a). This phenomenon may be attributed to thermal softening effect, low twinning fraction and variation in texture under high strain rate compression (further elaborated in Section 5).

The strain hardening rate (\(\frac{d\sigma}{d\varepsilon}\)) is defined as the rate of change of stress with respect to strain, and can be calculated with numerical differentiation of stress–strain data of compression curves. It is observed that the magnitude of strain hardening is continuously decreased with increasing strain rate (1000–4000 s\(^{-1}\)) as shown in Fig. 3(b). Strain rate sensitivity (SRS), rise in temperature (\(\Delta T\)) and absorption energy density (\(\Delta E\)) during dynamic compression is also calculated in order to further figure out the dynamic mechanical properties of ZK61 Mg alloy. SRS is an important parameter and can be calculated with the help of following equation.

\[
m = \frac{\ln(\sigma_2) - \ln(\sigma_1)}{\ln(\varepsilon_2) - \ln(\varepsilon_1)}
\]
where $\sigma_2$ and $\sigma_1$ are peak stresses and $\varepsilon_2$ and $\varepsilon_1$ are strain rates. Positive SRS $0.23$ MPa. s is observed up to a strain rate of 3000 s$^{-1}$ but at a strain rate of 4000 s$^{-1}$, it is negative $-0.24$ MPa.s under strain of 0.15. This calculation is also in good agreement with strain hardening rate and low flow stress at strain rate 4000 s$^{-1}$.

It is clear that a small increment in strain rate produces reasonable large stresses under dynamic loading, which induce $\Delta T$ in Mg alloys, so as per requirement flow stress should increase under high strain rate, contrary, it decreases due to the effect of thermal softening. The $(\Delta T)$ and $(\Delta E)$ can be calculated in the body of the sample with the following equations.

$$\Delta T = \beta \rho^{-1} C_0^{-1} \int_0^\varepsilon \sigma \varepsilon \, d\varepsilon$$  \hspace{1cm} (2)

$$T' = \Delta T + T_0$$ \hspace{1cm} (3)

$$\Delta E = \int \sigma \varepsilon \, d\varepsilon$$ \hspace{1cm} (4)

Fig. 2 – Microstructural analysis of ZK61 Mg alloy (a) EBSD map along ND (ED-TD plane) (b) misorientation angle distribution with respect to corresponding EBSD map (c) pole figure analysis of corresponding EBSD map.

Fig. 3 – True stress–strain curves of ZK61 Mg alloy under strain rate of 1000–4000 s$^{-1}$ performed with typical SHPB apparatus (b) strain hardening rate under all strain rates.
where $\Delta T$ rise in temperature, $T'$ changes in temperature after impact, $T_w$ is room temperature 300 K, $\beta$ is parameter of fraction of heat 0.9 (for ZK61 Mg alloy), $\rho$ is density 1.82 g/cm$^3$, $C_v$ is specific heat capacity $1.02 \times 10^{-3}$ J/kg K, $\sigma$ is applied stress and $\Delta\varepsilon$ changes in strain rate, and $\Delta E$ is absorption energy density. Almost 69°C rise temperatures and 68.3 MJ m$^{-3}$ absorption energy density is calculated at a strain rate 4000 s$^{-1}$. For the formation of adiabatic shear band (ASB), the temperature at the surface of the sample may be high in comparison of calculated theoretically in this study (discussed in Sections 4, 5). All other calculated values are depicted in Fig. 4(a).

It is obvious that Klosky compression bar does not accurately capture the elastic response; however, in this study, yield strength (YS) varies from 155 MPa to 255 MPa as shown in Fig. 4(b), which proposed that YS of ZK61 Mg alloy is sensitive to strain rate and increases with increasing strain rate.

### 3.3. Twinned induced deformation

In order to observe microstructure analysis after impact, fractured samples were analyzed using EBSD (for texture and twinning induced deformation) analysis. Fig. 5(a, c) and (b, d) shows EBSD maps and band contrast (BC) maps of the sample that was subjected to compression at strain rates 3000 and 4000 s$^{-1}$. A large fraction of $\{10\overline{1}2\}<10\overline{1}1>$ extension twinning is observed at strain rate 3000 s$^{-1}$, that are visible with red boundaries in BC map in Fig. 5(b). While the fraction of $\{10\overline{1}2\}<10\overline{1}1>$ extension twinning at 4000 s$^{-1}$ are very low in comparison to a strain rate of 3000 s$^{-1}$ as shown in Fig. 5(d). This suggests that $\{10\overline{1}2\}<10\overline{1}1>$ extension twinning has a major leading role for high flow stress in ZK61 Mg-alloy. Li et al. [30] studied a UFG ZK60 alloy, and reported that $\{10\overline{1}2\}<10\overline{1}1>$ extension twinning was dominant during the deformation process, but they did not provide any twinning evidence in their transmission electron microscopy (TEM) analysis, whereas based on texture evolution they concluded that $\{10\overline{1}2\}<10\overline{1}1>$ extension twinning had a dominant role for plastic deformation. In another study it was also reported that it is difficult for grain size $\leq 3 \mu m$ to undergo twinning induced deformation [37], but we have observed $\{10\overline{1}2\}<10\overline{1}1>$ extension twinning at grain size 1.5 $\mu m$ as evident in Fig. 5(a-d).

The twin induced deformation is also evidenced with the misorientation angle graphs that are presented in Fig. 6(a, b). In these factographs, we have observed strong peaks at 86.3° under strain rate 3000 and 4000 s$^{-1}$. These strong peaks are also associated with $\{10\overline{1}2\}<10\overline{1}1>$ extension twinning that induces plastic deformation in ZK61 Mg alloy.

### 3.4. Texture evaluation

Yield strength and flow stress are associated with $\{10\overline{1}2\}<10\overline{1}1>$ extension twinning, the former is due to low CRSS values 2–5 MPa, then lattice orientation of crystal and dislocation interaction with crystal orientation. Further, to access the possibility of twinning in deformed specimens, pole figures (PF) were analyzed to observe the textural changes under high strain rate 3000 and 4000 s$^{-1}$ compressions and presented in Fig. 7(a, b). It is observed that, initial fiber texture in Fig. 2(c) has changed and a transitional texture was developed. The (0001) basal plane showed strong double peak basal texture with maximal intensity 15.84 at 3000 s$^{-1}$ and 15.56 at 4000 s$^{-1}$. Fig. 7(a) shows that the strong basal texture, where (0002) basal plane is parallel and tilted toward ED while $\{10\overline{1}1\}$ shows random orientation. For 4000 s$^{-1}$ strong double peak basal texture is observed as shown in Fig. 7(b). This observation is well matched with the studies of Li et al., Dixit et al. and Asgari et al. [20,30,40], they also observed strong basal texture and double peak basal texture after impact. Moreover, Asgari et al. [40] attributed double peak basal texture with the $<c+a>$ slip activity. Conclusively, it is noticed that $\{10\overline{1}2\}<10\overline{1}1>$ extension twinning and (0002)$<11\overline{2}0>$ basal slip are responsible for high flow stresses in ZK61 Mg-alloy. In the context of all the aforementioned results, it is confirmed that these mechanisms are attributed to work hardening under dynamic loading.

![Fig. 4](image-url)
Fig. 5 – EBSD and BC maps of overall deformed ZK61 Mg alloy at strain rate (a, b) at 3000 s\(^{-1}\), (c, d) at 4000 s\(^{-1}\), red lines in BC maps are extension twins \{10-12\}<10-11>.

Fig. 6 – Misorientation angle graphs of ZK61 Mg alloy (a) at 3000 s\(^{-1}\), (b) at 4000 s\(^{-1}\) (strong peak at 86.3° is attributed to \{10-12\}<10-11> extension twinning).
Fig. 7 – Changes in the texture of ZK61 Mg alloy under high strain rate compression (a) at a strain rate of 3000 s⁻¹ (b) at a strain rate of 4000 s⁻¹.

Fig. 8 – OM analysis of fractured specimens of ZK61 Mg alloy (a) formation of ASB at a strain rate of 2000 s⁻¹ (b) big crack at the boundary of coarse grain and recrystallized grains at a strain rate of 3000 s⁻¹ (c) low magnification of the crack at a strain of 3000 s⁻¹ (d) magnified the view of the cracks at a strain rate of 3000 s⁻¹.
Dynamic compression under high strain rate is more uniform in comparison of quasi-static compression, we would like to indicate here that the samples failed along the planes of maximum shear stress 45° to the applied compressive force, such failure mode in Mg alloys has been already reported but poorly explained. So, in this section, a complete failure mode of ZK61 Mg alloy is presented.

Fig. 8(a) shows OM analysis of a sample that was subjected to compression at a strain rate of 2000 s⁻¹. We have observed many deformation bands that may be attributed to (0002)<11-20> basal slip and (10-12)<10-11> extension twinning. Brown arrows indicating recrystallized grains that may be representing the formation of the ASB. These recrystallized grains accumulated and formed a long snake-like morphology near the boundary of elongated grain and may have high local shear stresses σt. Further increase in strain rate may induce ASB and crack at the grain boundary of coarse grain and dynamic recrystallized (DRX) grains as shown in Fig. 8(b). Fig. 8(c, d) shows the ASB and cracks together that was subjected to compression at a strain rate of 3000 s⁻¹. It is observed that the crack is propagated at both sides of narrow ASB as shown in Fig. 8(d) which is a magnified view of Fig. 8(c), additionally many deformation bands were observed near the crack as marked with white arrows.

As, it is already reported that twin–twin interaction leads to crack formation in Mg alloys [22,27], similarly in this study we proposed that twin–twin interaction, twin–dislocation pile up at twin boundaries and stresses at triple junction grain boundaries causes a fracture in ZK61 Mg alloy. First twin–twin interaction and twin–dislocation interaction provides dynamically recrystallized grains as shown in Fig. 8(a). The grains having high local stressed σt diffuses one another due to large stress concentration and low grain boundary diffusion value of Mg alloys and make a white band consisting of very fine grains called ASB as shown in Fig. 8(d), additional stress lead to fracture due to the increase in local shear stresses.

Fig. 9 is a montage of the crack which is nucleated in ZK61 magnesium alloy at a strain rate of 2000 s⁻¹. The crack is developed at the boundary of dynamic recrystallized and coarse grains that propagated throughout the sample as shown in Fig. 9(c). Many secondary cracks were also observed (Fig. 9(a))
Fig. 10 – SEM micrographs of ZK61 Mg alloy (a) at a strain rate of 2000 s\(^{-1}\) (b) at a strain rate of 3000 s\(^{-1}\) (c, d) at a strain rate of 4000 s\(^{-1}\).

Fig. 11 – Schematic illustration of (a) crystal orientation under high strain rate compression, (b) pileup of dislocation at twinning boundary by reorienting the two crystals at 86.3\(^\circ\).

that may be attributed to voids and precipitates and the triple junction of grain boundaries. Tip of the cracks are also presented in Fig. 9(b, d), DRXed grains and deformation bands were also observed near the tip of crack.

SEM micrographs of ZK61 alloys under high strain rates are presented in Fig. 10. At strain rate of 2000 s\(^{-1}\) the non-uniform micro dimples and microcracks are shown in Fig. 10(a) and serve as a reference. The uniform micro dimples with a flat surface in a localized area are shown in Fig. 10(b, c) that were subjected to high strain rates 3000 and 4000 s\(^{-1}\). We have also observed localized melted zone at the fracture surface of a sample compressed at a strain rate 4000 s\(^{-1}\) as depicted by Fig. 10(c, d). The existence of micro-dimples and melting at the fracture surface of ZK61 Mg alloy suggested that the deformation becomes more ductile at strain rate 4000 s\(^{-1}\). This observation is in good accordance with the rise in temperature 69 °C and absorption energy density 68.3 MJ/m\(^3\) (Fig. 5) at strain rate 4000 s\(^{-1}\).

5. Discussion

Dislocation slip that varies with mechanical twinning is key deformation characteristic of Mg alloys under high strain rate compression at room temperature. These twins are generated due to local stresses \(\sigma_t\) and reorient the crystals from soft orientation to hard orientation by achieving their critical values. Initial fiber texture splitting toward TD by ±17° was transformed to the strong basal texture but still tilted toward ED at a strain rate 3000 s\(^{-1}\). While at strain rate 4000 s\(^{-1}\) crys-
tal reorientation was probably completed, c-axis of the grains are aligned parallel to ND i.e., basal planes reorient from partially perpendicular to parallel toward ED as shown in the schematic illustration in Fig. 11(a) and evident by {0001} PF in Fig. 7(b), this suggested that extension twinning at 86.3° have played their role to impede the dislocation glide and provided necessary flow stress that is intimately associated with the interaction of strain hardening, strain rate hardening, and thermal softening. Asgari et al. [40] suggested that a rise in temperature reduces the critical resolved shear stresses (CRSS) for non-basal slips. Therefore <c+a> dislocation will be dominant with an increase in strain, while they also reported double peak basal texture that was attributed to <c+a> dislocation activity. In the present work, we have observed double peak basal texture at strain rate 4000 s⁻¹, which is the signature of <c+a> dislocation.

Alloys that are plasticly deformed only by twinning are strain rate insensitive, while those dominated by twinning and slips are strain rate sensitive. The studied alloy is sensitive to strain rate. It is obvious that time for dislocation slip decreases at a high strain rate compression, which may increase the flow stress. But, for compression at a strain rate of 4000 s⁻¹ flow stress is low and may be attributed to the following important factors. (1) Rise in temperature (69 °C) during short interval of impact. (2) Low fraction of (10-12)<10-11> extension twinning (maximum grains have been reoriented due to the twinning (Fig. 5(c, d)) this suggesting very short time for accumulation of dislocation at twin boundary, and may annihilate the twin boundary very early. It may be a reason to cause crystal orientation more early from soft to hard and then soft orientation as illustrated in schematic Fig. 11(a).

(3) The stresses at grain boundaries are very high that may cause different slips activity in adjacent grains and replace with twinning at very high strain rate 4000 s⁻¹ (Fig. 5(c-d)). It can also be attributed to cracks (principal and secondary), once these cracks are formed, the maximum flow of kinetic energy (KE) will propagate through these cracks, thus showing a negative SRS ≈ 0.24. However, at a strain rate of 3000 s⁻¹, the (10-12)<10-11> extension twinning is significantly high in comparison of 4000 s⁻¹. The boundary of extension twinning impede the dislocation pileup and decreased dislocation mobility, thus increases the flow stress and shows positive SRS ≈ 0.23 as shown in the schematic illustration in Fig. 11(b).

It is obvious that almost 90% of the kinetic energy during impact is converted into plastic work, which induced thermal softening by increasing the heat in the specimen. It is reported that the increase in temperature in specimens of AM50 and AM80 Mg alloys was 72 °C (47 K), which is almost equal to the temperature theoretically calculated from Eq. (5) in ZK61 Mg alloy. Moreover, they observed a significant increase in temperature 137 °C on sample surface when ASB was fully formed [22,41]. Therefore, in this study, we can say that 130 °C rise in temperature may be induced at the fractured surface during the formation of ASB.

In Fig. 8 and 9 recrystallized grains, coarsen gains and deformation bands/twinning were found that shows the asymmetrical plastic deformation. Additionally, the fraction of deformation bands/twinning is higher than the fraction of recrystallized grains and coarse grain. This suggested that the crack propagation is due to twin–twin interaction and twin dislocation that resulted in DRXed grains [42]. As dislocation pileup increases at the grain boundaries and twin boundaries during deformation that produces DRXed grains which may further make an ASB by diffusion the grain boundaries, due to the low value of grain boundary diffusion and induction of adiabatic heat. Thus extra stress σd as a driving force may initiate a crack in these DRXed grains followed by ASB as shown in schematic Fig. 12(a) and evidenced in Fig. 8(a, d).

The secondary cracks are also observed in the montage of Fig. 9. These cracks are nucleated and propagated at a triple point junction of grain boundaries that lead to void in the sample. Fedorov et al. [43] reported that triple junction grain boundaries are favored for impeding and accumulating of grain boundary dislocation (GBD), the GBD pileup serves as a stress concentration; in this case, fracture is nucleated to release the stress concentration. Accordingly, in this study, the stress concentration near GBD pileup at triple junction of grain boundaries causes to produce voids. These nucleated voids followed by the voids that were already existed by alloying elements and precipitates to nucleate secondary cracks at the boundary of the triple junction of grain boundaries. Fig. 12(b) illustrates the formation and growth of the secondary crack.
6. Conclusion

Ultrasound grained extruded ZK61 Mg alloy of grain size 1.5 μm was fabricated and then subjected to dynamic compression under high strain rate 1000–4000 s⁻¹, following interesting outcomes are observed.

1 According to previous theoretical and experimental investigations, it seems very challenging to observe twinning induced deformation for grain size 3 μm, but in this study, we observed extension twinning at the grain size 1.5 μm. EBSD and BC map and misorientation angle graphs confirmed the twinning induced deformation. While PF analysis is evidence of grain reorientation that changes initial tilted fiber texture to strong double peak basal texture. This change in texture is also ascribed with (10-12)<10-11> extension twinning and <c+a> dislocations.

2 Positive strain rate sensitivity 0.23 MPa.s with peak stress 404 MPa is observed up to 3000 s⁻¹. This high increase in flow stress is associated to (10-12)<10-11> extension twinning and (0002)<11-20> basal slips. Additionally, low flow stress at 4000 s⁻¹ is attributed to adiabatic heat in the sample, early reorientation of crystals and low fraction of twinning.

3 The fracture was induced due to twin–twin, twin–dislocation interaction. Principal crack was initiated at the grain boundary of coarse grain and DRXed grains followed by ASB. Secondary cracks were nucleated and propagated due to voids at the triple junction of grains boundary. While SEM analysis concedes ductile fracture behavior due to uniform dimples at strain rate 3000 and 4000s⁻¹.

Conflicts of interest

The authors declare no conflict of interest.

Acknowledgments

This project is financially supported by the National Natural Science Foundation of China (Grant no. 51702015). Dr. Zheng from ZKKF (Beijing) Science & Technology Co., Ltd is acknowledged for EBSD tests and data analysis. We are thankful to Fujian Kunfu Stock Co., Ltd, China for providing ZK61 Mg Alloy.

REFERENCES

[25] Yu X, Li Y, Wei Q, Guo Y, Suo T, Zhao F. Microstructure and mechanical behavior of ECAP processed AZ31B over a wide


