In-situ quantification of intra and intergranular deformation in pure magnesium using full-field measurements at low and high strain rates

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A R T I C L E   I N F O

Keywords:
Magnesium
Ductility
Grain boundary
Hopkinson bar
Digital image correlation

A B S T R A C T

Due to the presence of weak grain boundaries as well as significantly coarse grain structure with minimal deformability, grain boundary cracking is almost an inevitable source of failure when a cast magnesium-based alloy is deformed at low homologous temperatures. The main objective in this work is to quantitatively investigate the contribution of inter- and intragranular deformation to the macroscale deformability and ductility of nominally pure as-cast magnesium subjected to quasi-static and dynamic loading. The proposed mesoscale full-field measurement approach is first presented and verified through measurements carried out to investigate deformation and grain boundary cracking in cast magnesium subjected to quasi-static loading. The method is then extended into the analysis of mesoscale deformation and failure under dynamic loading conditions using an experimental setup consisting of a split Hopkinson pressure bar (SHPB) and a high-speed imaging system. In both cases, the effect of the initial grain configuration on the local deformation response of Mg is investigated. The results indicate that the contribution of grain boundary region deformation to the total deformation exerted on the Mg samples is significant and depends on the initial grain configuration. Also, the strain rate sensitivity of the material is found to be dominated by the material deformation in the vicinity of grain boundaries.

1. Introduction

The effects of grain boundaries on the properties of crystalline materials ranging from deformation resistance to electrical conductivity have been studied for decades. From the mechanical behavior perspective, the presence of large fractions of grain boundaries (equivalent to having finer grains) is generally associated with higher strength, higher fracture toughness and improved ductility (Dieter, 1986). However, certain metals and alloys may still show relatively low strength and brittle failure response even with fine grain structures. Low strength grain boundaries and the delayed activation of slip systems in these polycrystalline metals are documented as being the principal sources for such mechanical behaviors (Hughes et al., 2007). Magnesium and its alloys are examples for such relatively brittle metallic systems. Despite having a high specific strength which makes them appealing to automotive and aerospace applications, magnesium and its alloys are among a group of materials which show low ductility at room temperature. This behavior is mainly due to limited activation of deformation systems at low homologous temperatures, whereas the activation of non-basal slip systems at elevated temperatures can significantly increase the degree of intragranular deformation, enhancing the ductility (Mordike et al., 2001).

Grain refinement through severe plastic deformation followed by thermomechanical processing has been widely used to increase the ductility of Mg-based alloys at low working temperatures. (Arab et al., Arab and Akbarzadeh 2013; Li et al., 2009; Yamashita et al., 2001). Producing a uniform and fine equiaxed grain structure with dispersed thermally stable particles has been identified as a method for processing of Mg alloys with superior forming ability (Xie et al., 2017). A refined equiaxed microstructure enables grain boundary sliding, a major mechanism for superplastic deformation in Mg alloys, even at low homologous temperatures. In as-cast conditions, Mg alloys generally exhibit poor deformability due to both composition and grain structure heterogeneities. Forming ability issues exacerbate in the case of cast pure Mg, as no thermal stabilizing particle would be present to hinder grain growth during processing. Therefore, due to the presence of weak grain boundaries, as well as significantly coarse grains with minimal deformability, grain boundary cracking is almost an inevitable cause of failure when cast Mg is deformed at low homologous temperatures.

Efforts have been devoted to explore the characteristics of grain boundary cracking and understand intergranular fracture and nominally brittle behavior in various alloy systems. Metals with hexagonal close-packed (hcp) crystal structure have been given special attention due to their susceptibility to grain boundary cracking. Among studies

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https://doi.org/10.1016/j.mechmat.2018.07.007
Received 3 May 2018; Received in revised form 5 July 2018; Accepted 12 July 2018
Available online 27 July 2018
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that explore the origin of grain boundary cracking in hcp metals is the work of Hughes et al. (2007), in which a pseudo-three-dimensional model consisting of hexagonal arrays was used to model cleavage cracking in polycrystalline zinc. Although such modeling approaches can facilitate a more in-depth study of possible mechanisms governing deformation and failure in the examined materials systems, in-situ experimental measurements to verify modeling results are still very scarce. The lack of systematic experimental studies in this area may be due to challenges which arise with the implementation of high resolution in-situ measurement protocols that enable quantitative-based investigations, especially at grain scales.

Recent advances in multiscale full-field measurements, in particular the development of digital image correlation (DIC), have facilitated accurate quantification of mesoscale deformation in a variety of material systems, from metals to polymer and ceramic composites (Efstathiou et al., 2010; Koohbor et al., 2015; Raabe et al., 2003; Tracy et al., 2015; Zhao et al., 2008). Constant improvement of spatial resolution in imaging devices has also led to the enhancement of full-field deformation measurements at grain and sub-grain levels. Such advancements currently allow for studying grain-level in-situ strain localization and damage (Tasan et al., 2014), slip band formation (Di Gioacchino and Quinta da Fonseca 2013), micro-strain evolution in ultra-fine grained alloys (Zhang et al., 2014), in-situ phase transformation (Das et al., 2016), active slip system identification in polycrystalline metals (Chen et al., 2017; Di Gioacchino et al., 2015; Guery et al., 2016a), and parameter identification for crystal plasticity modeling and simulations (Berin et al., 2016; Guery et al., 2016b). Despite constant improvements in spatial resolution of in-situ measurements carried out under slow deformation rates, research works dealing with in-situ characterization of mesoscale deformation at high strain rates are still very scarce. In fact, to the best of our knowledge, there exist only a few studies that take into account in-situ dynamic deformation measurements in metallic systems (Bodelot et al., 2015). Recent trends in the literature have seen growing interest in the applications of full-field measurements and the in-situ characterization of local deformation and failure phenomena in a number of different materials systems subjected to dynamic loading conditions (Koohbor et al., 2017a; Ravindran et al., 2016a; Ravindran et al., 2016b). Advances in high-speed and ultra-high-speed cameras, in terms of both spatial and temporal resolutions, have paved the way for such studies (Pierron et al., 2011).

The main objective in this work is to present the latest findings in mesoscale full-field measurements conducted on nominally pure cast magnesium. The methodology introduced here provides quantitative information on the contributions of inter- and intragranular strains to the macroscale forming ability of the material. In the following, details regarding material preparation and the proposed experimental approach are provided. Discussions regarding the quasi-static and the dynamic testing protocols are provided. Next, results obtained through optical-based digital image correlation measurements carried out to investigate deformation and grain boundary cracking under quasi-static loading conditions are presented. The approach is then extended into the analysis of mesoscale deformation and failure in high strain rate conditions. Roles of initial grain configuration on the deformation response of cast Mg under different strain rates is compared and discussed in detail. Finally, the contribution of inter-granular strains to the overall deformation of samples with various grain orientations is quantified.

2. Experimental

2.1. Material and specimen geometry

The material examined in this work was nominally pure (99.9%) as-cast magnesium. The reason for choosing the as-cast condition was supported by the facts that (1) grain structure in a cast magnesium ingot is coarse enough to enable optical-based mesoscale full-field measurements with proper resolution, (2) the grain boundaries are mechanically weak, escalating the probability of grain boundary cracking and intergranular fracture, and (3) a single ingot of cast magnesium offers a wide variety of grain shapes and orientations. The latter point is discussed in more detail in the following sections.

Cubic samples (10 × 10 × 10 mm³) were cut from a single cast ingot using a band saw and machined using a milling apparatus. Samples for quasi-static loading were extracted from various locations of the ingot to provide a range of different grain shape/orientations. Fig. 1 schematically shows the locations from which quasi-static samples were extracted. It should be noted that an as-cast magnesium ingot contains a variety of local grain structures. Due to the nature of solidification processes in pure metals, columnar grains are formed on the inner mold wall and are elongated towards the center of the ingot. A central zone containing equiaxed grains is formed at the ingot center. Depending on the purpose, specimen location and loading directions can be selected such that a variety of different loading conditions and mechanical responses can be studied concerning the initial grain configuration. The samples were extracted such that the influence of grain orientation and loading direction would be reflected in the measurements. As such, for simplicity, samples were named as “H”, “V” and “E”, for horizontal, vertical and equiaxed grain configurations, respectively (see Fig. 1).

Common surface preparation practice, including stepwise grinding and polishing with an aqueous alumina powder mixture, was applied to each sample. The relatively soft mechanical nature of cast magnesium made it challenging to achieve a mirror surface finish. However, this was not considered to be a significant issue in the present work because the sample surface was intentionally speckled to enable image correlation. The primary intention with polishing was to reveal the grain boundaries without the obstruction of major scratches or defects on the surface. The locations of these boundaries were later used to distinguish between inter and intragranular deformation domains. Specimens were etched for 10 minutes in an etchant solution consisting of picric acid, acetic acid, water, and ethanol. The typical microstructure of a magnesium sample in this work is depicted in Fig. 2a. It should be stated that no notable impurity content was detected in the samples. The coarse millimeter-sized grain structure of the cast specimen is clearly shown in Fig. 2a.

One surface of each cubic sample was speckled for full-field DIC measurements. Speckle pattern production was achieved by first coating the sample surface with a thin layer of flat white paint. Immediately afterward, a thin coat of black carbon powder was sprayed on the partially-dried white paint to produce a high contrast and dense black and white pattern suitable for DIC measurements. The average powder particle size in this work was 10 μm, small enough to enable the selection of a small correlation window (subset size) that allows for
detection and study of highly localized deformation patterns developed in the vicinity of grain boundaries. Note that for our purposes, the carbon powder offers a much finer speckle configuration at higher magnifications than traditional black paint. Fig. 2b shows a typical pattern applied to samples in this work.

2.2. Experimental setup

Quasi-static experiments were carried out in a conventional compression testing machine with a 22 kN load-cell capacity. The measurement accuracy of the load-cell was ±10 N, equivalent to ±0.1 MPa margin of error in stress measurements. The specimen was inserted between hardened stainless steel plates and compressed at a constant rate of 1 mm/min which is equivalent to a nominal strain rate of $1.7 \times 10^{-3}/s$.

Images of the speckled surface during loading were acquired using a 5 MP CCD camera (Grasshopper GS3-U3-51S5M-C, FLIR) at a rate of 1 image per second and at a full-field resolution of $2448 \times 2048$ pixel$^2$. Imaging rate was synchronized with the load-cell sampling rate through a series of baseline measurements similar to the approach followed in Ref. (Koohbor et al., 2016). Strain noise and transmitted bars while illuminated by a Lumen 200 metal arc lamp. Lighting was kept only for several seconds to minimize heating effects. Strain gauges on both the incident and transmitted bars were used to detect the impact signal. These strain gauges were connected to a signal amplifier which outputted to an oscilloscope that captured the impact waveform. This impact signal also initiated the triggering of the camera.

Similar to our quasi-static experiments, images captured via high-speed camera were processed using a subset size of $13 \times 13$ pixel$^2$ and a step size of 1 pixel with a strain filter size of 5. These DIC parameters allow for studying localized deformed domains with sufficient resolution.

Measurement performance, i.e. displacement and strain noise floors, for both quasi-static and dynamic camera systems were determined through a series of baseline measurements similar to the approach described in Ref. (Koohbor et al., 2016). Strain noise floors for quasi-static and dynamic experiments were found to be 945 $\mu$ε and 1530 $\mu$ε, respectively. Due to negligible out-of-plane displacements as well as a sufficiently high depth of field in the utilized imaging system, the influence of defocusing which resulted from out-of-plane motion was minimal in all measurements.

Finally, one must note that adiabatic heating of the sample in dynamic deformation conditions may have some influence on the mobility of grain boundaries (Rittel et al., 2012). Since in-situ full-field temperature measurement was not possible at high strain rate conditions, temperature effects were not considered in the present study.

3. Results and discussion

3.1. Global quasi-static stress-strain response

As previously described, a single ingot of cast magnesium may contain different grain structures. This is a consequence of the solidification process, a brief discussion of which was presented earlier. The presence of different grain shapes and orientations was taken advantage of in this work to explain the possible deformation and failure mechanisms that occurred in coarse-grained cast magnesium. Quasi-static uniaxial compression was applied on specimens with different initial grain shapes and orientations. Quasi-static uniaxial compression was applied on specimens with different initial grain structures. Note that the global strain measurement in our quasi-static experiments was performed using the built-in ‘virtual extensometer’ tool of the utilized DIC software, which provides highly accurate strain measurements with sub-pixel resolution. This being far more precise than the conventional compressive strain measurement based on

![Image](image.png)
platens displacement, as confirmed in the literature (Ravindran et al., 2016b; Sutton et al., 2009).

In all cases, specimens show a nominally linear initial stress-strain response, with nonlinear deformation initiating at approximately 35 MPa. This region is designated as ‘Region 1’, in which the slight differences between the curves are expected to be due to the initial orientation and size of grains. It should be emphasized here that depending on the crystallographic orientation of individual grains in each sample, some grains may start to plastically deform before the others (Zhao et al., 2008); however, the global yielding occurs when all grains start to deform plastically.

After nominal yielding and upon entering Region 2, all curves converge and an orientation-independent response is observed. Note that it is anticipated that the samples with different grain structures show a different plastic deformation response. However, the deformation mechanisms for these samples might be such that a unified apparent global mechanical response is obtained for our three samples, at least after the initial yielding. This behavior is explained through microstructural deformation measurements, discussed in detail in the forthcoming sections.

Upon further deformation and entering Region 3, intergranular failure initiates in the form of cracks at grain boundaries and triple junctions, finally leading to failure. Fig. 4 shows a typical evolution of intergranular cracking and final failure in sample ‘E’. The strain map shown in Fig. 4e indicates that failure initiates from boundaries on which highest strain magnitudes are developed. Interestingly, there are also regions on the sample surface that show very small local strains. Formation of such low strain regions may be associated with locally developed basal textures which limit the deformability (Liu and Wang, 2016).

Fig. 4 shows that intergranular failure initiates from triple junctions. Failure occurs at global compressive strains of approximately 8%. Specimen failure is revealed through the formation of small visible cracks on the surface, as well as a drop in the global stress magnitude. Note that depending on the initial grain structure, failure stress and strains differ for specimens tested in this work. The lowest failure stress was obtained for sample ‘E’, i.e. the one with relatively small equiaxed grains (i.e. highest volume fraction of grain boundaries1). Since failure is governed by grain boundary cracking in this material, it is indeed expected that a specimen with high grain boundary fractions would fail at comparatively lower global stress and strain, as clearly observed in this work. Conversely, sample ‘H’, which is characterized by having columnar grains with shortest grain axis aligned with the loading direction, fails at comparably larger strains. This indicates that the failure in the examined samples is essentially controlled by the amount of local strain developed at the boundaries. The orientation of grains in sample ‘H’ limits the amount of shear strain developed at grain boundary regions, therefore pushing global failure strains to larger values. In contrast, for sample ‘V’, in which columnar grains are mostly aligned with the direction of compressive load, shear deformation on boundaries is expected to be higher and therefore, failure takes place at relatively lower strains.

Regardless of the orientation of the grains with respect to the loading direction, it was evidently shown that in quasi-static loading, failure always takes place by grain boundary fracture. This is indeed a well-understood phenomenon particularly for hcp metals and is related to the role of grain boundaries in transferring in-grain cleavage from one grain to another (Hughes et al., 2007). In hcp metals, owing to insufficient active slip systems at low homologous temperatures, cleavage may occur in grains over some well-defined planes. Cleavage planes in neighboring grains do not necessarily meet in a line in their common grain boundary. Therefore, the two cleavage planes within adjacent grains intercept at the grain boundary at some angle. Depending on the fracture resistance of the boundary, the mismatched strain resulting from the cleavage planes may be partially accommodated at the boundary. Previous studies have suggested that the deformation accommodation at grain boundaries significantly contributes to the overall failure strain of the material (Crocker et al., 2005; Hughes et al., 2007). However, the amount of such contributions has not been verified through in-situ experimental measurements. One particular objective of this work, as presented and discussed in detail in

Fig. 3. Quasi-static stress-strain curves obtained for three different samples shown on the right side of the plot.

1 Quantification of effective grain boundary fraction will be discussed in Section 3.4.
the following, is to quantify the contribution of such grain boundary accommodation in the overall deformation of the examined material.

As a final remark in the study of global stress-strain response, our specimens in this work were deliberately machined to dimensions such that the study of grain-scale deformations and grain interactions using an optical-based approach would be possible. One may argue that material with finer grains would have been readily used in high magnification optical (or electron) microscopes to conduct the same type of characterization. Although this would be a valid argument, our primary intention in this work was to implement a systematic approach that can be used to facilitate studies in quasi-static as well as dynamic loadings. In dynamic loading, as discussed earlier in the introduction, high magnification full-field measurements are limited to optical-based techniques. Our attempt in the present work was to implement a systematic approach that enables obtaining directly comparable results acquired from quasi-static and dynamic experiments with the purpose of explaining possible deformation and failure mechanisms that lead to low ductility in cast magnesium.

3.2. Global dynamic stress-strain response

Global dynamic stress-strain curves were obtained for the Mg specimens using a conventional SHPB apparatus. Fig. 5 shows dynamic stress-strain curves obtained from two independent tests. The curves represent the constitutive response of samples with an initial grain structure similar to samples 'H' and 'V' in Fig. 3. However, a conclusive comparison between the dynamic stress-strain curves and those previously reported in Fig. 3 may not be made here. This is because (1) the measurement accuracy in SHPB experiments is comparably lower than that of quasi-static experiments. This is because in quasi-static experiments, the applied load is directly measured by high accuracy load-cells, while in SHPB experiments the stress is estimated from signals measured by the strain gauges and based on one-dimensional wave equations. (2) Depending on the material examined, the transient stress state may expand over several microseconds at the early stages of deformation (Koohbor et al., 2016; Pierron et al., 2014). Such a behavior invalidates the stress measurements within the linearly elastic regime. Therefore, no conclusive remarks similar to those made for quasi-static experiments could be made here. Instead, a direct comparison between the stress-strain curves obtained in quasi-static and dynamic conditions has been made. Comparing quasi-static and dynamic constitutive curves, the following remarks are highlighted:

1 Considering the general shape of the curves obtained from dynamic experiments, a downward concave shape is exhibited. This shape is generally considered as an indication for higher work hardening rates, which in the particular case of Mg and its alloys is related to
the occurrence of higher amounts of twinning during deformation (Barnett et al., 2004; Li et al., 2009). Increased amounts of twinning lead to higher levels of work hardening, resulting in a noticeable concave shape in the stress-strain curve. Interestingly, the sample corresponding to Test 2 in Fig. 5 had a similar structure to sample ‘H’ in Fig. 3. For this sample, as most of the grains were oriented perpendicular to the loading direction, minimal shear strain was expected to develop on grain boundaries and the applied global deformation was mostly consumed in deforming the grains rather than shearing the boundaries.

2 Nominal failure strains achieved in dynamic loading are slightly higher than those in quasi-static experiments. This observation is consistent with previous ones (Li et al., 2009), and is explained through heightened twinning and dislocation activities in high strain rate compression of magnesium (Dixit et al., 2015; Li et al., 2012).

Note that the formation of twins could not be observed and studied in this work due to limitations in the spatial resolution of our full-field measurements. In particular, an in-situ study of twinning in dynamic loading requires substantially higher resolutions and could not be achieved using the utilized imaging system. The failed specimen characteristics in dynamic condition were similar to those observed in quasi-static experiments. The fracture, in the case of dynamic condition, was also seen to initiate from the boundaries and triple junctions in the material, leading to total failure.

Fig. 6 compares the constitutive response of the material under two loading conditions. Curves representing quasi-static and dynamic response were the averages of those previously shown in Figs. 3 and 5. The strain rate sensitivity parameter, \( m \), was determined from these curves using the following simple definition:

\[
m(\varepsilon) = \frac{\ln(\varepsilon/d)_{s}}{\ln(\varepsilon/d)_{k}}
\]

where subscripts, ‘d’ and ‘s’ represents dynamic and quasi-static, respectively. Strain rate sensitivity was determined as a function of strain and was shown to undergo an initial increase with strain. The initial increase in the strain rate hardening is consistent with the data previously reported by Dixit et al., 2015, and is due to a rapid increase of twins in the structure which promote the overall work-hardening of the material subjected to dynamic deformation. Strain rate sensitivity then reaches a maximum at a strain magnitude of about 2% and decreases afterward. The rate of decreasing \( m \) with strain accelerates at strains > 6%, within a region that corresponds to Region 3 previously shown in Fig. 3. The trend observed for the parameter \( m \) confirms that strain rate sensitivity of the material is indeed related to the micro-structural aspects. Specifically, as the contribution of intergranular deformation becomes more prominent at higher global strains, the material is expected to become less strain rate sensitive. This further indicates that the two competing mechanisms described earlier, i.e. intra and intergranular deformations might show different strain rate sensitivities (Lin et al., 2008; Song et al., 2009). Further elaborations and quantification in this regard are provided in the forthcoming sections where local strain maps are presented and discussed.

3.3. Local strain mapping and possible deformation mechanisms

The global stress–strain behavior of the samples indicated that there must be two significant mechanisms governing the overall deformation and failure of the cast magnesium samples. These two are intra and intergranular deformation, the former was shown to be dominant at lower global strains. Intergranular deformation, on the other hand, was described as the major deformation mechanism controlling deformation and failure at grain boundaries, becoming more critical at larger nominal strains and characterized as being less sensitive to the applied strain rate.

To provide more quantitative evidence on these competing mechanisms, focus is given here to the full-field measurement data. Fig. 7a shows typical local strain maps obtained for sample ‘H’ (see Figs. 1 and 3) under quasi-static loading conditions. Initial grain boundaries are overlaid on strain maps to give an insight on the location of high and low strain domains. In Fig. 7a, the columns represent contours maps at approximately 1, 2, 3 and 4% global strain. From these images, the strain localization between the grain boundaries is evidenced by the large contrasts along and across the grain boundary overlay. This is especially clear when analyzing the \( \varepsilon_{yy} \) and \( \varepsilon_{xx} \) maps. As the evolution of the strain progresses, the horizontal orientation of the grains and the initialization of grains sliding over each other is evident. Substantial variability in the normal components of local strain is accommodated by the formation of strong shear deformations in the material, as shown in Fig. 7a.

It is clearly observed that in-grain and grain boundary deformations show very different values than the global strains applied. The visibly wavy patterns are a result of the highly heterogeneous strain behavior. For instance, when examining the \( \varepsilon_{yy} \) component, the large bands of red indicate little localized deformation, in contrast to the purple and pink regions which represent high magnitude strain behavior. A similar response was consistently seen in all specimens in this work.

Locally developed strains can take significantly higher values than those applied globally. For instance, the \( \varepsilon_{yy} \) contour map shown in the upper right corner of Fig. 7a, corresponding to a 4% global strain exhibits areas that contain strains up to twice as high as those applied globally. It is interesting to note that the nominal failure strain of the material in both quasi-static and dynamic loadings is below 10%, while local strain maps indicate substantially higher in-grain strains. A reason for such phenomenon might be that in spite of the hexagonal closed packed crystal structure of pure Mg, relatively small (c/a) ratios = 1.622, give rise to the activation of slip modes other than merely the basal slip system. In particular, prismatic and pyramidal slip modes as well as [0112] < 011T> twinning systems, have also been identified to be active deformation modes in pure magnesium (Styczynski et al., 2004). These modes can all contribute to the relatively large in-grain strain development in the material, also proving that low global failure strain is essentially controlled by limited deformation and failure resistance of grain boundaries. Of course, this explanation may not be generalized to all Mg alloys, since alloying elements are established to be capable of altering the c/a ratio in magnesium (Minarik et al., 2016). With regard to observations made in the current work, there is smaller deformation activity occurring within the grains in comparison to the boundaries.
The high concentration of strain occurring preferentially at the boundaries in our experiments indicates that these particular grains’ orientations lend themselves to be more resistant to twinning activation (Wang et al., 2014). Regardless, strong strain gradients across grain boundaries lead to significant sliding of grains, the evidence for which is presented in Figs. 7b and 7c.

3.4. Quantification of in-grain and grain boundary deformation

Although strong deformation heterogeneities are evident in strain contours, full-field DIC measurements do not make a distinction between data obtained inside of grains and over the boundaries. Distinguishing in-grain deformation from that developed over the grain boundaries is essential in the present work since we intend to quantify the contribution of each to the total strain applied to the samples. Therefore, a simple approach is proposed where a mechanically relevant ‘effective grain boundary thickness (EGBT)’ parameter is introduced to distinguish between deformation of the grain boundary regions and deformation developed at grain interiors. The proposed idea of EGBT is schematically shown in Fig. 8. Note that the mechanically relevant effective boundary thickness introduced here is different from grain boundary width used for diffusion studies (Prokoshkina et al., 2013). The concept used in diffusion studies considers a very narrow region typically with dimensions of a few nanometers utilized to characterize mass transfer through grain boundaries. The notion of EGBT proposed here is significantly larger in size, several orders of magnitude larger than those used in diffusion studies. In addition, EGBT is not an inherent material property, rather it depends mainly on the dimensions of the area of interest in a DIC-based measurement, and is highly sensitive to image correlation parameters used in a DIC analysis. In particular, image correlation parameters that facilitate measuring more localized deformation patterns at higher spatial resolutions allow for selecting thinner EGBTs. For more detailed discussions regarding the influence of image correlation parameters in measuring mesoscale deformation at highly localized regions refer to (Koohbor et al., 2017b; Ravindran et al., 2017).

In order to quantify the EGBT in this work, local strain profiles across grain boundaries were considered. Fig. 9 shows a typical evolution of in-plane strain components across a short representative line ‘L’. Local strain data over the representative line segment ‘L’ were plotted and fitted with proper Gaussian-type curves, as shown in Fig. 9c. Gaussian curve fitting in this work was conducted in MATLAB®. Once the experimental data were fitted with the proper Gaussian function, the arithmetic mean and the standard deviation was identified, and the span of one standard deviation of the data was taken as the thickness of the effective grain boundary (δGB). Due to higher accuracy and better spatial resolution of quasi-static measurements in this work, EGBT measurements were carried out for quasi-static specimens and the obtained values were used for the case of dynamic loading conditions as well. The approach mentioned above was carried out several times for multiple line segments at various specimen locations. The average of all δGB measurements was obtained as 200 µm. This value was taken as the effective boundary thickness. The ratio of the surface area of effective grain boundaries to the sample surface, referred to as grain boundary fraction (ξGB), was calculated for each sample using the following expression:

$$\xi_{GB} = \frac{\delta_{GB} \times d_{GB}}{d^2}$$  

Fig. 7. (a) A typical contour maps are depicted, showing the evolution of in-plane strain components at different global strain magnitudes for sample ‘H’. Loading was applied in (-)y direction. Grain boundary outline is overlaid on contour maps. Images of initial and post-failure conditions of sample ‘H’ are shown in (b) and (c), respectively.

Fig. 8. Schematic representation of mechanically relevant effective grain boundary thickness (EGBT) and grain interior (GI) regions in this work. Effective boundary thickness is denoted by δGB.
where $d_{GB}$ is the total length of grain boundaries visible on the sample surface, and $d$ denotes the in-plane sample dimensions ($=10 \text{ mm}$). $\varepsilon_{GB}$ values for samples E, H, and V were determined as 68%, 42% and 48%, respectively.

Next, strain evolutions over grain boundaries and grain interiors are considered and analyzed in order to estimate the contribution of each mechanism to the total strain. Our methodology will be analogous to that proposed by Langdon (2006). In this approach, the total strain applied to the specimen, $\varepsilon_T$, may be expressed as:

$$\varepsilon_T = \varepsilon_{GI} + \varepsilon_{GB}$$

where $\varepsilon_{GB}$ is the strain associated with just the intragranular (in-grain) deformation, and $\varepsilon_{GB}$ is the strain developed over the entire network of grain boundaries. Notice that the latter, i.e. ‘grain boundary strain’ hereafter refers to the strain developed over narrow effective grain boundaries, the thickness of which was determined as $\delta_{GB} = 200 \mu m$. The contribution of grain boundary strains to the total strain, $\eta$, is then expressed as:

$$\eta = \frac{\varepsilon_{GB}}{\varepsilon_T}$$

whereas $(1 - \eta)$ indicates the contribution of in-grain strains to the total strain applied. Furthermore, due to the complex in-plane strain patterns, to enable a direct comparison of local and global strains, the definition of equivalent von Mises strain, $\varepsilon_{eq}$, is utilized to determine both in-grain and grain boundary strains. Equivalent strain in this regard is calculated as:

$$\varepsilon^{eq} = \left[ \frac{2}{3} (\varepsilon_{xx}^2 + \varepsilon_{yy}^2 + 2\varepsilon_{xy}^2) \right]^{1/2} (\text{‘GI’ or ‘GB’})$$

From a practical point of view, separation of in-grain and grain boundary regions within a whole-field area of interest are cumbersome. To accomplish this task with higher accuracy, we carried out two separate image correlation runs for each sample. As shown in Fig. 10, first, the whole speckled area was selected as the area of interest and all strain components were determined. Next, image correlation was performed again, this time with the initial area of interest only containing the grain interiors, from which the effective grains boundaries were separated from the beginning. Finally, the strain data for just the grain boundaries was calculated using Eq. (3). Digital image correlation for strictly the grain boundary regions was not possible due to the areas of interest being far too narrow. Image correlation parameters (subset, step and strain filter) were chosen to be the same for all DIC runs. Again, this approach is not rigorous and provides just a broad generalization about deformation activity occurring in the regions of the grain boundaries. Finally, all strain components were spatially averaged over the corresponding area of interest and further analyses were performed.

Fig. 11 shows the variation of the parameter $\eta$ with respect to total strain applied on our quasi-static samples. All samples show significant variations in the value of $\eta$ at strain levels lower than 0.02. This strain range is consistent with Region 1 discussed earlier in Fig. 3, and is associated with the initiation of plastic deformation in the material, the occurrence of which is highly heterogeneous due to the initial Schmidt factor of the grains. After plastic yielding, the $\eta$ parameter takes on values close to 55% for sample ‘H’. This indicates that the contribution of in-grain deformation and deformation of the grain boundary regions is almost the same in the deformation of the sample with grains mostly oriented perpendicular to the loading direction. In sample ‘E’, the $\eta$ factor displays higher values than those of sample ‘H’. This behavior can be related to the comparably larger fraction of grain boundaries in this sample, which can accommodate higher local deformations in the areas more closely associated with grain boundaries. The $\eta$ parameter for this sample takes on values close to 70%. Finally, in sample ‘V’, i.e. the one with grain boundaries mostly aligned with the direction of the far field load, the highest values of the $\eta$ parameter is measured. This observation is indeed consistent with our previous discussions and can be attributed to the occurrence of large local shear strains within the grain boundary network. For this sample, the contribution of grain boundaries to the overall deformation is estimated to be above 80%.

Interestingly, for all samples, a slight change takes place at total strain values close to 0.05. This strain value is approximately the
transition strain between Regions 2 and 3 in Fig. 3. Due to the unavailability of deformation data within the bulk of the samples, no further discussion can be provided on whether or not any internal damage in the form of grain boundary cracking occurred in the material. However, this can be a subject of interest for future computational studies or those facilitated by the use of X-ray tomography and volumetric image correlation (Langdon, 1970; Lenoir et al., 2007). Regardless, the result indicate that the role of grain boundaries in accommodating the deformation applied quasi-statically on pure magnesium is substantial. The contribution of grain boundaries in this regard was quantified to be at least 50%. This value is higher than that previously obtained through computational approaches (Hughes et al., 2007), and can be verified through more accurate crystal plasticity and micromechanics models in the future. Of course, one should bear in mind that the measurements in this work were limited to a single material with weak grain boundaries. However, a similar approach can be implemented to study other HCP metals with more regular grain structures and mechanically stronger grain boundaries.

Similar to the approach used to identify the parameter \( \eta \) with total strain for quasi-static samples. Symbols ‘H’, ‘V’ and ‘E’ indicate ‘Horizontal’, ‘Vertical’ and ‘Equiaxed’ grain orientations, respectively. See Fig. 1 for more details.

Fig. 10. Illustration of the approach utilized to separate in-grain deformation from grain boundary region deformation. Solid green regions in each image indicate the DIC area of interest. Showing from left to right: Full-field DIC area of interest, in-grain DIC area of interest, GI, and grain boundary area of interest, GB. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Fig. 11. Variation of the parameter \( \eta \) with total strain for quasi-static samples. Symbols ‘H’, ‘V’ and ‘E’ indicate ‘Horizontal’, ‘Vertical’ and ‘Equiaxed’ grain orientations, respectively. See Fig. 1 for more details.

Fig. 12. Variation of the parameter \( \eta \) with total strain for dynamic samples. Symbols ‘H’ and ‘V’ indicate ‘Horizontal’ and ‘Vertical’ grain orientations, respectively. See Fig. 1 for more details.
can be concluded that the deformation of the grain boundary regions plays a more significant role in the overall strain rate sensitivity of the material. At large strain magnitudes applied dynamically, the overall deformation of the material is controlled to a larger extent by the inter-grain deformation mechanisms rather than the shear-dominated grain boundary deformation mechanisms. On the other hand, a relatively higher strain rate sensitivity of the material observed at smaller total strains can be associated with a grain boundary deformation mechanism.

The concluding statements above may be of particular significance in modeling works that tend to study deformation and failure response of brittle polycrystalline systems. In this regard, appropriate models accounting for the strain rate dependence of grain boundary deformation seem to be essential to obtain more accurate and realistic material response. It must be emphasized here that crystal plasticity constitutive models incorporating strain rate sensitivity in intragranular deformations are well-established (Barlat et al., 2002; Salem et al., 2005). The observations in this work confirm that similar models are required for intergranular deformation studies, as well. In addition, the results found in this work can be used as guidelines in the development of grain boundary engineering (GBE) strategies in the processing of material with desirable mechanical properties (Cao et al., 2017; Randle, 2004; Wantanabe and Tsurekawa, 2004). The approach introduced in this work can be applied in studies carried out in the area of GBE, mainly shedding light on the mechanical characteristics of grain boundaries in a material subjected to high strain rate loading conditions.

Last but not least, experimental results such as those obtained in this work can complement multiscale modeling data obtained via various computational tools. For instance, recent molecular dynamics (MD) simulations suggest that 30° tilt and twist grain boundaries in pure Mg can be highly mobile as they correspond to energy minima (Liu and Wang, 2016). Results from similar modeling efforts combined experimental findings reported in this work can provide the roadmap for the processing of advanced engineered Mg alloys with superior strength and deformability that will be appealing to aerospace and automotive applications.

4. Conclusions

Mesoscale full-field deformation characteristics of nominally pure cast magnesium were investigated utilizing digital image correlation under both quasi-static and dynamic loading conditions. The quasi-static mechanical response of Mg as a function of initial grain orientation was studied along with the evolution of intergranular cracking under uniaxial compression. The contributions of intr-grain and inter-grain deformations to the total deformation of the samples were quantified. Initial grain configuration was confirmed to play a significant role in both the local and global deformation response of samples at quasi-static deformation conditions.

An approach facilitated by the use of a split Hopkinson bar apparatus and a high-speed camera was also proposed to capture local dynamic deformation behavior of the samples in-situ. The contributions of both inter and intragranular strain in dynamic deformation were also quantified, the former of which was found to be grain orientation-dependent, also playing a substantial role in the macroscale deformation. Strain rate sensitivity of samples was measured and found to be related to the amount of deformation occurring in the immediate area of grain boundaries. The key findings of the present can be summarized as:

(1) The initial grain orientation with respect to the loading direction can alter the ratio of grain boundary deformation to the total deformation.

(2) Irrespective of the grain orientation direction, the ratio of grain boundary deformation to the total deformation is higher in the quasi-static loading than dynamic loading condition.

Fig. 13. \( \eta \) parameter data used for direct comparing of quasi-static and dynamic results. The data shown in this figure is for a sample with grains aligned perpendicular to the loading direction, i.e. sample ‘H’.

Fig. 14. Ratio of \( \eta \) parameter for dynamic and quasi-static conditions versus total strain. Subscripts ‘dyn’ and ‘QS’ denote dynamic and quasi-static loading, respectively.
The results from this work can be used to provide insight into the dominant deformation mechanisms in magnesium, which in turn can be applied to the modeling and crafting of systems comprised of other low ductility polycrystalline metals.

Acknowledgments

This work was made possible following a technique developed through a program supported by the Air Force Office of Scientific Research (AFOSR) under Grant No. FA9550-14-1-0209 and FA9550-16-1-0623. The support of AFOSR is gratefully acknowledged.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.mechmat.2018.07.007.

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