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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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ABSTRACT

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 explor 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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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 Fonseca2013), 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 (Bertin 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.

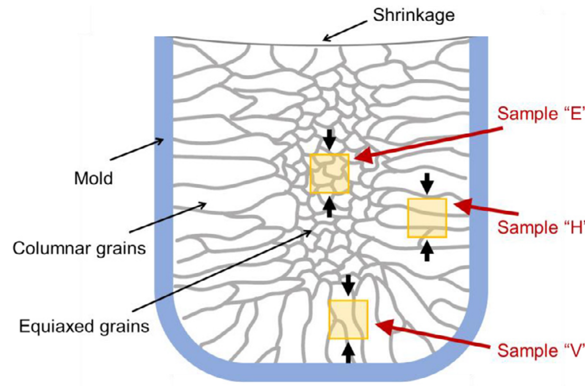

Fig. 1. Schematic representation of the locations in a single Mg ingot from which samples were extracted. Loading directions are marked with bold arrows. Chill zones on the mold interior walls are not illustrated.

Cubic samples ($10 \times 10 \times 10$ mm^3) 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