On the deformation twinning of MgAZ31B: a three-dimensional synchrotron x-ray diffraction experiment and crystal plasticity finite element model

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Abstract

Crystals with a hexagonal close-packed (HCP) structure are inherently anisotropic, and have a limited number of independent slip systems, which leads to strong deformation textures and reduced formability in polycrystalline products. Tension along the c-axis of the crystal ideally activates extension twinning as a deformation mode due to the lack of easy-slip systems. In this work, experiments were devised to study extension twinning in a polycrystalline Mg alloy AZ31B with a strong basal rolling texture by tensile deformation parallel to the plate normal. Three-dimensional synchrotron X-ray diffraction (3DXRD) was used to map the centre-of-mass positions, volumes, orientations, elastic strains, and stress tensors of over 1400 grains in-situ up to a true strain of 1.4%. More than 700 tensile twins were observed to form in the mapped volume under deformation. The measured center-of-mass positions and grain volumes are used to construct various 3D microstructures and model them with a Crystal Plasticity Finite Element (CPFE) code. It is observed that the average grain-resolved stress did not always select the highest ranked Schmid factor twin variant. In fact, the contribution of lower ranked variants was non-negligible. The CPFE simulation indicates that there is a small variation in the stress within each grain in the elastic regime, which increases drastically upon the onset of plasticity. One of the significant outcomes of this work is the new statistical information on the interaction between twin and parent grain. It is shown that, on average, there is a small difference between the stress normal to the twin habit plane in the parent and twin, but that this is not the case for the shear acting on the habit plane.

Keyword: 3DXRD, Twin-parent interaction, Crystal Plasticity Finite Element, Magnesium AZ31B

1. Introduction

The demand to implement light metals in aerospace and transportation industries has led to a substantial effort in understanding deformation mechanisms in high strength to density ratio candidates such as Mg and its alloys. Strength and ductility are two major factors in selecting an alloy for structural applications requiring crash-worthiness. Mg and its alloys typically have lower formability than competing materials like steel and aluminum alloys due to the limited number of independent easy slip systems at room temperature. Increasing formability requires a better understanding of deformation mechanisms over multiple length scales. Understanding the twinning deformation mechanism is challenging, because unlike slip, twinning is a sudden process that reorients a volume of a grain. It is widely accepted that the

Critical Resolved Shear Stress (CRSS) is the driving potential for activating dislocation sources on a known slip plane in the corresponding slip direction, however the drive for twinning is more ambiguous.

Early on, a “problem” with twin nucleation was recognized by Bell and Cahn (1953). They noticed that a wide range of values was being reported for the twinning stress in Cd crystals. Bell and Cahn conducted experiments on Zn (which, like Cd, has a c/a ratio greater than ideal) and found that samples which had been slightly bent and already contained small twin “wedges”, as well as samples on which they made a small indent during loading, twinned at lower stresses than undeformed and unindented samples. This suggested that the stress required for initiation of a twin is higher than the stress required to propagate and grow a twin. Still, intuitively, the principal of a CRSS for twin nucleation should hold. In fact, several studies have suggested that the orientation of the parent crystal defines which variant of a twin system can be the predominant or the most active through the Schmid relation (Brown et al., 2005; Gharghouri et al., 1999; Godet et al., 2006; Park et al., 2010). For example, Godet et al. (2006) used the Schmid factor to predict texture components upon deformation of a Mg alloy tube oriented for tensile twinning. Similarly, Barnett et al. (2014) have shown that the Schmid factor for twinning controls nucleation more than thickening in AZ31. However, several studies suggest that twin nucleation is not solely controlled by the Schmid factor. The presence of low Schmid factor twins and an absence of high Schmid factor twins have been reported for both primary (Beyerlein et al., 2010; Capolungo et al., 2009; Jonas et al., 2011; Knezevic et al., 2014a) and secondary twinning (Barnett et al., 2008). Jonas et al. (2011) suggest that twin variant selection may be influenced by the strain accommodation required by neighboring grains. When a twin is formed at a grain boundary, the neighboring grain must deform to accommodate the shape change; a push-off effect. The deformation in the neighboring grain will then create a back stress within both twin and parent grain (Clausen et al., 2008). Twin variant selection can then be affected by the amount and type of deformation necessary. For example, if prismatic glide is required, high internal stresses may develop and prevent the formation of that particular twin variant. A lower ranked variant that does not require a “hard” type of accommodation in the neighboring grain may form instead, or, the twinning may not occur at all.

In large-scale EBSD studies of Mg and Zr, it was observed that the majority of twins at least touch a grain boundary, and are believed to nucleate there (Beyerlein et al., 2010; Capolungo et al., 2009; Knezevic et al., 2012; McCabe et al., 2009). It was also observed that there does not appear to be a significant increase in probability of the formation of a twin in large grains versus small grains. In fact, Ghaderi and Barnett (2011) report that the number of twins formed per unit of nucleating boundary area tends to decrease with increasing grain size, because the stresses are lower. Yu et al. (2012) studied the effect of size on twinning by micropillar compression and tension of pure single crystal Mg. They observed that there was no twinning in micropillars with diameters of 2-10 μm in Mg. In this size regime pyramidal and $\langle c \rangle$ -type dislocations have been reported (Byer et al., 2010; Lilleodden, 2010), but no twins. The twinning did, however, occur in samples with a diameter of approximately 200 nm. The surface nucleation of defects was found to play an important role in twinning, alongside the level of stress within the crystal. In a polycrystal, the grain size effect is still confounded by the effects of neighboring grains, which are not yet fully understood.

Many fundamental questions about deformation twinning still remained un-answered. Various numerical techniques have been used to aid interpretation of experimental studies to answer some of these questions. At the micro-scale, crystal plasticity modelling has played a major role in understanding the fundamentals of twinning. A significant contribution to this area was given in the works by Van Houtte (1978), Tomé et al. (1991), Kalidindi (1998), and Staroselsky and Anand (2003) in which it was suggested that twinning can be modeled as a pseudo slip. The pseudo slip analogy has been used in various numerical analysis to explain twin nucleation (Beyerlein and Tomé, 2010; Niezgoda et al., 2014), twin-parent interaction (Barton et al., 2009; Clausen et al., 2008; Fernández et al., 2013; Juan et al., 2014), lattice strain evolution during twinning (Mareau and Daymond, 2010; Warwick et al., 2012), texture development (Coghe et al., 2012; Izadbakhsh et al., 2011; Jia et al., 2012; Knezevic et al., 2014c; Lévesque et al., 2010; Mareau and

Daymond, 2011; Mayama et al., 2011), different hardening processes (Clayton, 2009; El Kadiri and Oppedal, 2010; Knezevic et al., 2013), and twinning-detwinning (Kondo et al., 2014; Muránsky et al., 2009; Wang et al., 2012; Wang et al., 2013a; Wang et al., 2013b). However, using a pseudo slip scheme for modeling twinning does not capture the dynamic nature of twinning especially at the inception and nucleation stages (Abdolvand and Daymond, 2013b).

Studies of the interaction between twin and parent pairs have indicated that neighboring grains can play a significant role in stress development inside the twin or even in thickening of twins (Abdolvand and Daymond, 2013b; Aydiner et al., 2009; Bieler et al., 2014; Zhang et al., 2008). Hence, understanding deformation twinning requires a high population of twinned grains where each grain experiences a different arrangement of neighboring grains. Such a measurement can be done by 3DXRD, a novel method that allows fast and non-destructive measurement of the full stress tensor in a large population of grains (Oddershede et al., 2012; Oddershede et al., 2010; Poulsen, 2004). With 3DXRD, clusters of grains are illuminated simultaneously and the diffracted signals from these grains are measured with a detector while the sample is rotated about an axis perpendicular to the beam. Center-of-mass position, volume, crystallographic orientation, elastic strain, and stress within each grain are among the parameters that can be measured using 3DXRD. Diffraction Contrast Tomography (DCT) is another method that has been used to study deformation in large populations of grains. It has similar characteristic to 3DXRD, yet the sample has to be close to the detector, and consequently it is difficult to extract stress and strain from DCT with sufficient accuracy (Ludwig et al., 2009; Ludwig et al., 2008; Reischig et al., 2013).

In this work, the deformation mechanisms in Mg AZ31B are studied using a combination of 3DXRD and CPFE. Uniaxial tensile deformation is applied to an AZ31B polycrystal in a direction relative to the parent texture that triggers significant extension twinning. Codes are developed to determine parent and twin pairs and to study the effect of local stresses on the twin variant selection. The evolution of stress within each parent and twin is also studied. In order to provide complementary information and to investigate the heterogeneous distribution of stress within each grain, CPFE is applied to 3D grain microstructures generated directly from the 3DXRD measurements. The experimental set-up for the in-situ 3DXRD experiment is first described. The codes that are developed for analyzing data are subsequently explained. In the following section, the method that is used for constructing 3D mesh for finite element analysis is explained along with the user subroutine for modeling slip and twinning. Numerical and experimental results are provided at the end followed by a discussion section.

2. Experiment

2.1. Sample preparation

The material was received as a hot-rolled plate of Mg alloy AZ31B with a thickness of 9.52 mm and a typical rolled texture (c-axis direction concentrated parallel to ND) as shown in Fig. 1a. In this figure, RD and TD represent rolling and transverse directions, respectively. The average initial grain size was approximately 30 μm . From a measurement point of view, it is ideal to have no more than 100 grains in the cross section of the sample for 3DXRD, which requires a grain size of approximately 100 μm in a 1 mm x 1 mm cross-section. Hence, the material was heat treated for 20 hours at 500°C, in an Ar atmosphere to minimize oxidation. A micrograph of the subsequent rolling plane is shown in Fig. 1b. With little driving force for recrystallization and grain growth, the final average grains were fairly equiaxed with a grain size of approximately 50 μm , the largest grains being over 100 μm and the smallest approximately 30 μm .

Tensile ‘dogbone’ samples were fabricated by electric discharge machining (EDM) to a 1.3 mm x 1.3 mm cross section and 3.52 mm gauge length as shown in Fig. 1c. The tensile axis, z, is along the ND to directly induce tensile twinning. The samples were then chemically etched in nital solution (Mg bright

etch composed of 100 ml methanol and 20 ml HNO₃) in order to remove the EDM surface damage and reduce the cross-section of the gauge length to 1 mm x 1mm. The geometry allowed the samples to be “gripped” using a horizontal support without pre-deformation by squeezing. For the diffraction experiment, two black lines were painted on the sample to allow measurement of macroscopic strain by image correlation.

Fig. 1: (a) {1010} and {0002} pole figures showing the initial texture of the AZ31B material; (b) micrograph of the rolling plane after annealing for 20 hours at 500°C; (c) as machined AZ31B sample dimensions. All dimensions in mm and ND is the loading direction.

2.2. In Situ 3DXRD experiment

The 3DXRD experiment was performed at the high-energy X-ray scattering beamline 1-ID at the Advanced Photon Source (APS), Argonne National Laboratory (ANL). The sample was mounted in a stress rig, with the tensile direction vertical (along ND as shown in Fig. 2a). The X-ray energy was 51.996 keV (Tb K-edge, $\lambda=0.024\text{nm}$). A MAR165 detector, with an active area of $165 \times 165 \text{ mm}^2$ and a pixel size of $78.64 \times 78.64 \text{ }\mu\text{m}^2$, was placed at a sample to detector distance of 407 mm. The undeformed sample was preloaded to 5 N in order to ensure proper alignment. To probe all grains in the illuminated volume, and not just those that satisfy a single Bragg condition, the sample was rotated around the tensile axis (ND) and diffraction images were acquired in the angular range $[-150^\circ \text{ to } -30^\circ]$ and $[30^\circ \text{ to } 150^\circ]$ in steps of 0.5° . The accessible angular range is limited by the posts of the load frame. An exposure time of 0.5s per image and a beam size of $400 \text{ }\mu\text{m} \times 25 \text{ }\mu\text{m}$ (width x height) were used. This scan was repeated for twelve layers, as the sample was scanned along the z direction (ND). The stress rig was then driven in position control, while diffraction images were monitored, until new diffraction spots appeared on the inner rings of the diffraction pattern (compare Fig. 2b, and 2c) at 0.2% strain, which indicated the formation of twins. The specimen was then mapped, as when undeformed, while the crosshead displacement was held constant. Successive maps of the specimen were made after the sample was unloaded to 5 N, reloaded to 0.4% strain, and then to 1.4% strain. As the sample was deformed, the number of diffraction peaks increased due to the occurrence of twinning, and the diffraction peaks smeared out due to the increasing grain orientation spread and plasticity. To ensure that peaks could still be separated, as required for the 3DXRD reconstruction, the width of the beam was slit down from initially 400 microns for strains less than 0.4% to 300 microns for 0.4% strain and 200 microns afterward. Strain maps were collected at five different load steps. The whole 3DXRD experiment was carried out over a week. Measurement time for each step increased toward the last loading step with 9 hours for the preload step and 19 hours for the last loading step. The details regarding measurement steps are summarized in Table 1. In this report, results for the unloading step are not reported: (a) due to a limited beam-time, only part of the starting volume was scanned, (b) detwinning was not detected in the scanned volume, and (c) detwinning was not implemented in our CPFE code (see section 3.1).

a.

b.

c.

Fig. 2. (a) Experimental setup at APS. The sample is loaded along ND which is 33 in the figure. Sample portions of diffraction images for (b) the preload sample, (c) after 0.2% strain and the appearance of twins showing the spread of peaks. Images are negatives to better show peak spreading.

2.3. Data analysis and post-processing experimental data

The task of extracting information regarding the properties of the material from the stack of diffraction images was handled by the FABLE suite of programs [<http://sourceforge.net/p/fable/wiki/Home/>]. The data analysis can be separated into two stages: (I) identification of the grains, and (II) determination of their attributes.

Stage I of the analysis comprises four steps: (i) preprocessing, (ii) peak identification, (iii) transformation, and (iv) indexing. During pre-processing the raw diffraction images are processed, which consists of background subtraction using a mean background level. The peak searching process yields a filtered peaks file that contains a list of each reflection with the pixel and ω position and integrated intensity (using ImageD11, by J.P. Wright, <http://sourceforge.net/p/fable/wiki/Imaged11/>). In the transformation step, the diffraction peaks are converted to scattering vectors in reciprocal space. The indexing procedure then assigns the scattering vectors to grains by the GrainSpotter program (Schmidt, 2014).

Stage II of the analysis is performed using the FitAllB program (Oddershede et al., 2010). FitAllB serves a twofold purpose: (i) refinement of the global experimental parameters, and (ii) refinement of individual grain properties. The determination of grain-resolved physical parameters depends on the accurate calibration of global experimental parameters such as sample-to-detector distance, detector tilt, and beam center on detector position. In the current experiment these parameters, including reference lattice parameters of $a = 3.21613\text{\AA}$ and $c = 5.22337\text{\AA}$, were first refined from the preload data. Subsequently the

grain properties consisting of centre-of-mass positions, orientations and elastic strain tensors of each grain were refined for all load steps. In addition, the relative grain volumes were calculated based on the intensities of the assigned reflections. The estimated errors on all the refined parameters are summarized in Table 1, which shows the volume weighted mean of the grain resolved measurement errors on position, relative volume, orientation, and the three axial stress components in the sample system. Details on how the error bars are derived can be found elsewhere (Oddershede et al., 2010), while the sizes and trends will be discussed in section 4.2.

During peak-searching, indexing and refinement, the twins were treated as entirely separate and independent grains. These grains were then analyzed in order to identify the required orientation relationship, an 86° rotation c-axis for $\{10\bar{1}2\}$ primary twins. A twin is assigned to a parent when (i) the required orientation relationship is met, (ii) the distance between the centres of mass of the parent and twin is less than the sum of their radii, and (iii) twin and parent share the twin habit plane. It is possible for a grain to have a twin relation with more than one other grain. Due to the limited spatial resolution, two distinct twins with the same orientation in a single grain may be detected as one twin. In addition, very small grains/twins may be missed in the analysis since their scattered intensity will be too low.

Table 1. Details of the results and error bars of the 3DXRD analysis

Step	Macro-Strain %	Macro-Stress (MPa)	No. of Grains	Hold time (hours)	No. of Twins	Diffraction spots per grain	Average grains error bars					
							3-D position (μm)	Relative Volume %	Orientation ($^\circ$)	Stress Components (MPa)		
										ND (33)	RD (22)	TD (11)
Step-1	0.00	5	6345	9	86	83	23	23	0.098	13	24	17
Step-2	0.2	54	4989	11	238	81	28	40	0.093	16	29	21
Step-3	0.4	55	2833	20	437	72	42	57	0.097	24	43	31
Step-4	1.4	55	1411	19	711	73	66	67	0.110	39	70	52

3. Crystal plasticity modeling

3.1. Mathematical equations

A User MATerial (UMAT) subroutine developed by the authors is used for modeling plastic deformation caused by slip and twinning. A brief description of the code is given here, while a detailed explanation can be found elsewhere (Abdolvand and Daymond, 2012; Abdolvand et al., 2011). Generally, in the presence of both plastic slip and twinning, the plastic part of the velocity gradient can be written as (Kalidindi, 1998):

$$L^P = \left[1 - \sum_{\beta=1}^{N^{tw}} f^\beta \right] \sum_{\alpha=1}^{N^{pa}} S^\alpha \dot{\gamma}^\alpha + \sum_{\delta=1}^{N^{ch}} f^\delta S^\delta \dot{\gamma}^\delta + \sum_{\beta=1}^{N^{tw}} \dot{f}^\beta S^\beta \gamma_0 \quad (1)$$

where the first term on the right hand side of Eq. (1) represents plastic slip inside the parent grain, the second term represents plastic slip inside twins, and the last term represents the strain accommodated by the volume fraction of twins present. f^β is the twin volume fraction associated with shear on the twin system β , S^α is the current Schmid tensor of a system α , γ is the shear on the slip or twinning system, γ_0 is the characteristic twin shear which is 0.1289 for single crystal Mg (Hosford, 1993), and N represents the number of slip system available in parent (pa) and child (ch). N^{tw} is the total number of twin variants (six in this case) that can be potentially active in the parent. The updated Schmid tensor is a function of the updated normal to the slip/twin system (n^α) and the slip/twin direction (d^α):

$$S^\alpha = d^\alpha \otimes n^\alpha \quad (2)$$

The shear rates on each twin/slip system in Eq. (1) are calculated using a rate-dependent formulation proposed by Asaro and Needleman (1985):

$$\dot{\gamma}^\alpha = \dot{\gamma}_0 \left| \frac{\tau^\alpha}{g^\alpha} \right|^n \text{sign} \left(\frac{\tau^\alpha}{g^\alpha} \right) \quad (3)$$

where $\dot{\gamma}_0$ is a reference shear strain rate and n controls the rate dependency. τ^α and g^α are the resolved shear stress on the slip or twinning system α and strength of this system, respectively.

The twin volume fraction rate in Eq. (1) correlates with the shear rate on each twin system in the parent grain as (Kalidindi, 1998):

$$\begin{aligned} \dot{f}^\beta &= \frac{\dot{\gamma}^\beta}{\gamma_0}, f^\beta \geq 0, \sum_\beta f^\beta \leq 1, \text{if } \tau^\beta > 0 \\ \dot{f}^\beta &= 0 \text{ if } \tau^\beta \leq 0 \end{aligned} \quad (4)$$

The imposed restrictions in Eq. (4) indicate that the twin volume fraction cannot be negative, the sum of the twinned volume cannot exceed the parent grain volume itself, and the twinned regions are not allowed to detwin. τ^α is a function of imposed stress and Schmid tensor (Asaro, 1983):

$$\tau^\alpha = P^\alpha : \sigma \text{ where } \dot{\sigma} = \mathcal{L} : D^e \quad (5)$$

where P^α is the symmetry part of the Schmid tensor, σ is the Kirchhoff stress and the Jaumann rate of this stress is related to the elastic part of the rate of deformation (D^e) and the elastic tensor ($\mathcal{L}$).

The strength of each slip and twinning system (Eq. 3) is assumed to follow an extended Voce hardening relationship:

$$g^\alpha = g_0^\alpha + (g_1^\alpha + \theta_1^\alpha \Gamma) \left(1 - \exp \left(-\frac{\theta_0^\alpha \Gamma}{g_1^\alpha} \right) \right) \quad (6)$$

where g^α is the current CRSS, g_0^α is the initial CRSS, Γ is accumulated shear on all slip/twin systems, θ_0^α is initial hardening rate, and g_1^α and θ_1^α determine asymptotic characteristics of hardening. The ability of Voce hardening in modeling plastic deformation in Mg alloy is discussed elsewhere (Ma et al., 2012).

For modelling plastic deformation in Mg AZ31B, the prism $\{11\bar{2}0\}$ basal $\{11\bar{2}0\}$ and pyramidal $\{11\bar{2}2\}$ slip systems are used along with tensile twinning $\{10\bar{1}1\}$. Detwinning or other possible twinning modes are not included in the model at this stage. Various methods have been suggested for extracting CRSS or single crystal parameters (Herrera-Solaz et al., 2014; Sánchez-Martín et al., 2014). The material properties that are used for modelling in this paper are given in Table 2. These parameters are extracted by altering the parameters reported in Abdolvand and Daymond (2012) to capture the macroscopic stress-strain curve and texture components. The elastic constants used for Mg AZ31B are those from Simmons and Wang (1971) for single crystal Mg: $C_{11}=59.75$ GPa, $C_{33}=61.7$ GPa, $C_{12}=23.24$ GPa, $C_{13}=21.7$ GPa, and $C_{44}=16.39$ GPa.

Table 2. single crystal properties of Mg AZ31B

	n	$\dot{\gamma}_0$ s^{-1}	g_0^α GPa	g_1^α GPa	θ_0^α GPa	θ_1^α GPa
Prism	20	1.0×10^{-3}	0.06	0.01	0.24	0.08
Basal	20	1.0×10^{-3}	0.012	0.01	0.24	0.0
Pyramidal	20	1.0×10^{-3}	0.1	0.06	2.0	0.5
Tensile twin	20	1.0×10^{-2}	0.04	0.0	0.005	0.05

3.2. Constructing the 3D input file

Results from the 3DXRD experiment were used to generate an input file for the ABAQUS solver (2005). A code was developed to implement a weighted Voronoi tessellation, also known as Laguerre tessellation (Lyckegaard et al., 2011; Okabe et al., 1992), to generate 3D grain geometries based on the center-of-mass positions and volumes of the grains measured in the preload step (Step-1), i.e. the parent grains were used as the starting point. In general, for a given set of n sites $p_i = (s_i, w_i), i = 1:n$ where $s_i \in R^d$ is a seed point and $w_i \in R$ is a weight, a weighted Voronoi tessellation is a convex subdivision of d -dimensional space into cells C_i fulfilling (Lyckegaard et al., 2011):

$$C_i = \{X \in R^d | \|X - s_i\|^2 - w_i < \|X - s_j\|^2 - w_j, i \neq j\} \quad (7)$$

where w_i can be squared of the radius of each grain which is measured through the 3DXRD experiment. If the center-of-mass μ_a of a grain, say G_a , is known, it is possible to estimate the seed point (s_a) for the grain G_a using Eq. 8,

$$s_a = 2\mu_a - \mu_b \quad (8)$$

where μ_b is the center-of-mass position of the grain G_b that is constructed by setting its seed point position s_b equal to μ_a .

After strains had been applied to the sample, a smaller volume was probed in the 3DXRD experiment (See Table 1 and Fig. 5). Therefore, a representative cube of material containing ~1000 grains located in the middle of the probed volume was chosen for CPFEE modeling. It was possible to track most of these grains throughout the x-ray deformation experiment. This volume was then meshed with C3D8 elements, linear brick elements with 8 nodes and 8 integration points (IP) (Abaqus, 2005). The grain family for each of the elements was decided based on Eqs. 7-8 and the position of the element in the volume, i.e., once the volume was meshed, the position of each element in the volume was known. A grain was then assigned to each element based on the position of the element and how close the element is to a seed point. Each seed point represents only one grain, hence each element belongs to only one grain. With using this method, integration points of each element does not necessarily coincide with grain boundaries (Gonzalez et al., 2014; Knezevic et al., 2014b); not having integration point right at the grain boundary has minor effect on the *statistical* results that is reported here. This is discussed elsewhere (Abdolvand and Daymond, 2013a). A detailed description of the input models used for the simulations is given in Table 3 and shown in Fig. 3. Each model is identified with an M and a number that represent the step size used for meshing the volume. Hence, different mesh densities were assigned to each grain within the volume. The reason why the number of meshed grains for the same volume changes slightly in the models is that by using a smaller meshing step size, part of the grains that have their center of mass further outside of the investigated volume are also meshed. However, all of the interior grains retain the local environment throughout, i.e. the neighbors of each interior grain do not change from one model to another. This is confirmed by checking that all of the grains that have their center-of-mass position inside the selected volume are meshed.

These interior grains are used for FE convergence check as well as for comparison with the experimental results. Also, it is worth mentioning that the IP size in Table 3 represents the average distance between IPs in the models.

Table 3. Input files used for CPFE simulation

	Number of grains	Number of elements	Size of the cube μm^3	Mesh Step size μm	Number of IP per grain	IP size μm
M10	927	20^3	200^3	10	69	5
M5	1000	40^3	200^3	5	552	2.5
M4	1014	50^3	200^3	4	1079	2

It is assumed that the modeled cube is representative of the bulk, and hence periodic boundary conditions developed by Abdolvand et al. (2011) are applied to the free surfaces. The model is strained at a rate of $1.4 \times 10^{-4} \text{ s}^{-1}$. The effect of unloading and any de-twinning that may occur during unloading was neglected in the model, for the reasons stated earlier.

Fig. 3. The 3D microstructure constructed based on 3DXRD measurement in Step-1; models (a) M10, (b) M5, (c) M4. Random colors are assigned to the grains.

4. Results

The quantitative information provided from the measured center-of-mass positions, orientations, volumes, elastic strains, and stresses of each individual grain along with the simulated microstructure provides a powerful tool for investigating the relationships between twins and their parent grains and neighborhoods. In this work, the key features will be identified and explained. Even though it is mathematically easy to compare most of the experimental results with those from CPFE, it becomes difficult to clearly visualize the correlations. Therefore, to avoid busy figures and potential misinterpretations, not all of the results are overlaid.

In all of the results presented here ND, RD, and TD are labelled as 33, 22, and 11, respectively, with ND parallel to the loading direction.

4.1. Mechanical response

The mechanical response of the AZ31B alloy under uniaxial tension along ND is shown as a true stress-true strain curve in Fig. 4, with arrows indicating the stress and strain levels at which diffraction measurements were made. The deformation curve for the 3DXRD experiment is overlaid on the measured continuous deformation curve and both are compared with the results of the CPFE simulations. There is no difference between models M10, M5, and M4 in predicting the average stress-strain curve indicating that 8000 elements were sufficient to capture the average behavior of the aggregate. Overall, the 3DXRD results match the continuous loading curve quite well with a few differences. Firstly, the macroscopic yield point was slightly higher during the APS test (55 MPa compared to 50 MPa). There also appears to be no significant hardening after yielding in the APS test. During the 3DXRD measurement, the sample was held at a constant cross-head position for an extended period of time, ranging from 9 to 20 hours

while collecting diffraction images, during which stress relaxation occurs (5 MPa at Step-4). The continuous loading curve shows a classic response for a material that twins profusely: elastic loading until yield, followed by concave-up portion indicative of twinning and finally monotonic hardening until failure at approximately 325 MPa and 16% strain. The elastic modulus calculated from the change in axial stress and elastic strain yields 49 GPa, which is in good agreement with reported values of 45 GPa for a similarly oriented AZ31B polycrystal (Chun and Davies, 2011).

Fig. 4. Stress strain curve for AZ31B. The red curve shows results from the 3DXRD experiment at APS overlaid onto a continuous loading curve shown in blue. The numbers 1 to 4 indicate the strains (0, 0.2, 0.4 and 1.4%) at which diffraction measurements were made. Strain was measured using digital image correlation. Results of the CPFE simulations are presented in black. Fig 4b is magnified version of Fig. 4a for the strain range that 3DXRD experiment was carried out.

4.2. The mapped volume

The orientation maps are shown in Figs. 5a and 5b, where each sphere represents a grain as well as its volume. It should be noted that the sphere size does not represent the absolute size of the grain, but is used instead to give a sense of the mapped volume. The colouring shows orientation relative to ND.

Fig. 5: Representation of the mapped volume for (a) Step-1, and (b) Step-3. The size of each sphere represents the volume of each grain relative to the total mapped volume and the colour shows orientation relative to ND. The undeformed sample is at 5 N preload. z is the loading direction, x is the transverse direction, and y is the beam direction (through thickness). Values on each axis are given in μm .

As the sample was in an annealed state, it was possible to scan and resolve more than 6300 grains in the preload step. However, with increasing deformation, the number of resolved grains decreased due to smearing and overlapping of diffraction spots caused by the deformation induced intragranular orientation

spread. Also, due to the profuse density of twins, the size of the beam and the probed area were changed so that the scanned grains could still be resolved (see Fig. 5b). In total 1411 grains were resolved in Step-4, where half of these grains are twins that have formed since Step-1.

The results in Table 1 indicate that the errors on all refined parameters tend to increase with applied strain. This is to be expected as the individual grains become more deformed, and hence the center-of-mass position of the corresponding reflections, which are used to refine the grain resolved parameters, become less well defined. It should be noted that it is in fact strains that are directly refined from the experimental data, whereafter the stresses are calculated by means of the stiffness tensor given in section 3.1. It is observed that errors in the RD components ($\sigma_{22}, \epsilon_{22}$), which are parallel to the incoming beam when the posts of the load frame are at $\pm 90^\circ$ and hence not blocking the beam, are larger than for TD ($\sigma_{11}, \epsilon_{11}$) and significantly larger than on the ND components due to limited angular rotation about the loading axis. However, because of the strain to stress conversion, the large error bar on the ϵ_{22} strain component parallel to the incoming beam does not only affect the error bar on corresponding σ_{22} stress component, but in fact also the errors of the other axial stress components (by a factor of 1/3 since the off-diagonal C_{12} and C_{13} terms of the stiffness tensor are roughly 1/3 of the diagonal C_{11} and C_{33} terms). This effect along with the low flow stress of the material give rise to the relatively large error bars on all of the axial stress components. Oliver et al. (2004) have explained why the measured variations for cubic polycrystals are larger in the components perpendicular to the applied load. For the HCP crystal structure and in our experiment, these variations become higher as non-uniform stress is allowed. In section 4.4 the CPFE simulations show that these errors are associated with significant stress heterogeneity within each grain.

4.3. Statistical analysis of experimental results

A statistical analysis of the microstructure and Von-Mises stresses measured at different steps is illustrated in Fig 6. A cube located in the center of the probed volume in Step-1 and with $200 \times 200 \times 200 \mu\text{m}^3$ volume that contains more than 1000 grains for CPFE simulation is used for generating Figs. 6a to 6c. In Fig. 6a, the frequency (i.e. probability distribution) of the total misorientation between any two neighboring grains in the volume is shown. This figure indicates that more than 50% of neighboring grains have less than 15° total misorientation. The low angle of misorientation between neighboring grains normally results in an easier slip transfer between two grains provided that both grains are oriented for deforming by slip. Greater insight into the mechanism by which grains accommodate deformation can be made by comparing Figs. 6b and 6c. In Fig. 6b the frequency of misorientation between basal plane normal of any two neighboring grain is shown while in Fig. 6c, the frequency of the misorientation between basal plane normal and loading direction (θ) is shown. The basal plane misorientation of any two neighbouring grain follows a Gaussian distribution with a peak between 15 and 25 degrees. Fig. 6b shows that more than 61% of the grains have basal misorientations less than 25° . In Fig 6c, more than 80% of the grains have θ less than 30° , which requires twinning deformation to be the dominant mechanism.

Fig. 6: Statistical analysis of the 200 x 200 x 200 μm^3 probed volume. (a) Frequency of the total misorientation between any two neighbouring grains in Step-1; (b) Frequency of basal plane misorientation between any two neighbouring grains for Step-1; (c) Frequency of misorientation between basal plane normal and loading direction for Step-1; (d) Frequency of Von-Mises stress for loading steps; (e) Variation of Von-Mises stress as a function of misorientation between basal plane normal and loading direction; (f) Variation of Von-Mises stress as a function of basal plane misorientation between any two neighbouring grains. In Fig 6e and 6f the red line represent mean values.

A statistical analysis of stress developed in the probed volume is shown in Fig 6d to 6f. The frequency of Von-Mises stresses for the investigated grains is shown in Fig. 6d. As a result of the sharp and clean diffraction spots in Step-1 and being in the elastic zone, there is a sharp peak in the measured stress. As expected, the peak broadens with increasing plastic deformation. The stress frequency plot peaks at $\sigma = 20 \text{ MPa}$ in Step-1 and increases and stabilizes at $\sigma = 60 \text{ MPa}$ for the rest of the load steps. This is in agreement with the macroscopic stress strain curve as there is no significant hardening for strains less than 1.6%. Nevertheless, due to the increase in the dislocation density and rotation variations, the peak broadens. Also, while the measured macroscopic stress is 55 MPa, some of the grains in Step-4 develop stress values as high as 240 MPa ($\pm 80 \text{ MPa}$). The CPFE simulation, presented in section 4.4, will demonstrate a wide variation in stress between and within grains.

In Fig. 6e, the variation of Von-Mises stress as a function of θ for Step-2 is shown. Blue circles represent the measured average stress within each grain and the red line represents the average value for each level of θ . While the number of grains contributing to the average is negligible for θ larger than 60 degrees, the average stress varies narrowly around 80 MPa for $\theta < 60^\circ$. There is a significant variation of blue circles about the red line indicating the effect of local neighborhood on the stress developed in individual grains. These variations are shown in Fig. 6f as a function of misorientation between the basal plane normals of any two neighbouring grains. It is clear that the mean value for this figure does not really change with the basal misorientation. Considering the texture of the sample where a majority of the grains are oriented with basal plane normals nearly parallel to the tensile axis (Fig. 6c), a large basal plane misorientation is very likely to originate from a soft grain adjacent to a hard grain, and probably even surrounded by hard grains. Naturally, due to load sharing between hard and soft grains, the average value of stress should decrease at higher misorientation, yet, for this particular texture, it does not; since soft grains are mostly surrounded by hard grains they appear to be constrained to move with the hard ones.

4.4. Stress variation in parent grains

A comprehensive comparison between measured stresses within each parent grain and calculated stresses are given in Fig.7 and Fig. 8, where blue dots represent calculated stress at each IP, red crosses represent average stress for each grain calculated using the CPFE simulation, and black circles with error bars represent the measured values. In this paper, the area created by the blue dots of each grain is called the variation zone. Although the calculated macroscopic stress-strain curve for all of the CPFE input models are equal (see Fig. 4), the model M4 is used for generating the figures as more than 1000 IPs are assigned to each grain in this model and the convergence of the result for most of the grains is guaranteed. Generally, 350 IPs are required to capture stress variation within each grain; a comprehensive study of the required number of IPs per grain for capturing heterogeneous deformation within each grain is given in Diard et al. (2005).

The evolution of stress in the ND for all of the loading steps is illustrated in Figs. 7a to 7d. Due to low elastic anisotropy, there is almost no variation in the calculated stress for the “preload” step (Fig. 7a). That is why the blue dots in Fig. 7a are covered by the red crosses. However, the effect of plastic anisotropy in AZ31B is very pronounced, even at the onset of macroscopic plasticity (Fig. 7b); this can be clearly seen in the spread of the blue dots about the red crosses. Also, although the average stress within each grain in both CPFE and 3DXRD stay constant in the plastic zone (Fig. 7b to 7d), with more strain, the CPFE calculates wider variations about this average stress. Interestingly, this result is in agreement with the extracted error bars from 3DXRD, which are associated with peak smearing that results from stress and orientation variation across a grain. Nevertheless, for most of the grains, the measured stresses with their error bars fall into the CPFE variation zone.

As an example of stress variation in the RD and TD, calculated and measured values for these two directions for Step-3 are illustrated in Figs. 7e and 7f, respectively. As explained in section 4.2, uncertainty in the measured values for these directions are normally higher than the loading direction in common with Quinta da Fonseca et al.(2006), which is why larger error bars are associated with these calculations. Also, although the calculated and measured average stress within each grain is close to zero (especially in the RD), the CPFE simulation indicates that there are significant variations about the average values.

Fig. 7. Comparison between stresses measured in 3DXRD experiment and those calculated with CPFE simulations. Calculated and measured stress in normal (loading) direction for (a) Step-1 (unload), (b) Step-2 (onset of macroscopic plasticity), (c) Step-3 ($\epsilon_{\text{applied}} = 0.4\%$), and (d) Step-4 ($\epsilon_{\text{applied}} = 1.4\%$). Calculated and measured stress for Step-3 in (e) rolling direction and (f) transverse direction. In all of the figures, blue dot represents calculated stress at each integration point, red cross represents the average stress within each grain calculated in CPFE simulation, and black circles with error bars are the measured values.

As an example of shear stress variation, the calculated and measured values for all of the three shear stress components for Step-3 are illustrated in Figs. 8a to 8c. Similar to the perpendicular stresses,

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4 average stresses in both the CPFE calculation and 3DXRD measurement are close to zero. While the
5 calculated normal stresses in Step-3 vary between 5 MPa to 125 MPa, the shear components generally
6 vary within a narrower window of -30 to 30 MPa. Most of the stresses in this direction also fall into the
7 variation zone.
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9 The effect of misorientation between basal plane normal and loading direction (θ) on the stress
10 development in grains of the aggregate is illustrated in Figs. 8d to 8f. Not surprisingly, except for $\theta \geq$
11 50° , for which the population of the grains was not high enough to give a good statistical representation,
12 the measured and calculated normal stress decreases with the increase in θ (Fig. 8d). More importantly,
13 despite this trend, there is a wide variation in the calculated local stresses where even grains with $\theta \geq 50^\circ$
14 have some IPs that behave like a hard grain, i.e. stress levels at these “soft” grain IPs are as high as the
15 stress levels in IPs of a “hard” grain. These local hard spots in the soft grains are potential sites for twin
16 nucleation and growth (Abdolvand and Daymond, 2013a, b). A common trend between stress in both the
17 loading and perpendicular directions is that the stress variations in hard grains ($\theta < 15^\circ$) are much less
18 than that in soft grains. In the strongly textured material, soft grains are surrounded by hard grains, and
19 due to force equilibrium at grain boundaries, a wide variation in stress from one side of a soft grain to the
20 other could be observed.
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Fig. 8. Comparison between stresses measured in 3DXRD experiment and those calculated with CPFE simulations. Calculated and measured stress for Step-3 ($\epsilon_{\text{applied}} = 0.4\%$): (a) σ_{12} , (b) σ_{13} , and (c) σ_{23} . Stress variation in Step-4 as a function of misorientation between basal plane normal and loading direction for (d) normal direction, (e) rolling direction, and (f) transverse direction. In all of the figures, blue dot represents calculated stress at each integration point, red cross represents the average stress within each grain calculated in CPFE simulation, and black circles with error bars are the measured values.

4.5. Effect of twinning on texture

Tensile twins are identified by a sharp change in orientation of the crystal lattice, in Mg characterized by an approximately 86° rotation of the c-axis of the parent grain. When the grains of each orientation map are plotted on a $\{0002\}$ pole figure in Fig. 9a and 9b, the presence of twins is evident. In the undeformed microstructure (Fig. 9a), most of the basal poles are aligned near parallel to ND (out of plane, center of pole figure). With increasing deformation, $\{0002\}$ poles appear on the outer edges of the pole figure, towards RD and TD. In Fig. 9b the measured pole figure of Step-3 is shown. The pole figures shown are discretized such that each spot represents a grain, regardless of its size. So, the pole figures are representative of the *number fraction* of twins than the volume fraction. The corresponding measured volume fraction is shown in Fig. 9c. In agreement with Fig. 5a and 5c, a portion of hard grain volumes ($\theta < 20^\circ$) is transferred to newly born twins ($\theta > 75^\circ$). The twin volume fraction reported in Fig. 9d (black circles with error bars) was calculated based on the relative volume measurements from the 3DXRD experiment. Fig. 9a to 9c reveals that both the twin volume fraction and twin number fraction increase with strain. For this calculation, any twins present in the preload step (i.e., in the annealed sample) were excluded.

In Fig. 9d, the 3DXRD results for twin volume fraction are compared against those calculated by CPFE simulation and those reported by Chun and Davies (2011). In the work by Chun and Davies (2011), uniaxial load was applied along the normal direction of an AZ31B plate with the texture similar to Fig. 1a, and the evolution of the microstructure was studied by EBSD. Generally there is a good agreement between all of the data sets. In comparison to 3DXRD, at lower strains, CPFE under-predicts the twin volume fraction. However, for higher strains, the CPFE calculation is very close to the 3DXRD results and those reported by Chun and Davies (2011). The average twin volume fraction within each CPFE simulated grain as a function of θ is given in Fig. 9e. For $\theta > 40^\circ$, the average twin volume fraction is close to zero at Step-3; interestingly, hard grains with similar θ do not necessarily have the same twin volume fraction, reflecting the effect of local neighborhood and load sharing that significantly influences twin volume fraction.

Fig. 9. $\{0002\}$ pole figures for the strain steps (a) Step-1 (preload), and (b) Step-3 ($\epsilon_{\text{applied}} = 0.4\%$) composed of the mapped grains listed in Table. 1. with (c) illustrating the corresponding measured volume fraction. (d) Comparison between CPFE calculation and measured twin volume fraction. (e) CPFE calculation for twin volume fraction in each grain as a function of misorientation between normal to the basal plane of the grain and loading direction.

4.6. Schmid factor and twin variant selection

The number of twins measured for different steps is reported in Table 1. In this section, only the grains that fall into the cube-volume used for modeling will be studied (Table 4). For a direct comparison, not all of the twinned grains in CPFE are compared against the 3DXRD results, in fact, for each step, only those parent grains that were found in the experiment and existed in the CPFE model are counted. Also, the twins that existed at Step-1 are excluded from the reported number for the experiment. Generally, a good agreement is observed between the CPFE calculation and 3DXRD results.

Table 4. Comparison between CPFE and 3DXRD results for number of twinned grains

	Number of parent grains	Number of twinned grain in CPFE	Number of twinned grains in 3DXRD
Step-2	795	0	26
Step-3	668	98	96
Step-4	325	257	175

Historically the tendency of a grain to twin has been characterized by its Schmid factor, which requires knowledge of the local or global stresses. Since measurement of local stress requires application of advanced techniques, eg. High Resolution Electron BackScattered Diffraction (HREBSD) or 3DXRD, generally the macroscopic applied stress has been used to calculate the Schmid factor (Beyerlein et al., 2010; Capolungo et al., 2009); we term this the global Schmid factor. Correspondingly the Schmid factor calculated from the local (micro-) stress state can be termed a local Schmid factor. In Mg, the tensile twin system has six variants, each of which can have different Schmid factors. In this work, the Schmid factor of each variant is calculated using Eq. 9.

$$SF^{\beta} = \frac{P^{\beta} : \sigma}{\|\sigma\|} \quad (9)$$

where SF^{β} represents the Schmid factor of twin variant β , P^{β} is the symmetric part of the Schmid tensor (Eq. 2), and $\|\sigma\|$ is the norm of the stress tensor (highest value between absolutes of the principal stresses; whether global or local). The twin variant in the experiment was defined by the habit plane shared between the twin and parent grain, and the Schmid factor of this variant was computed using the applied stress (Global Schmid Factor (GSF)), or average stress measured within the parent grain, (Average Local Schmid Factor (ALSF)). The Schmid factors of all possible variants were computed, from which the rank of the variant that actually twinned can be determined. The contribution of each variant to the formation of twins is illustrated in Fig. 10. Variants in this figure are sorted from maximum to minimum, blue bars represent the Schmid factors calculated based on applied stress, and green bars represent those calculated based on the average stress measured for each twinned grains. In order to compare like-with-like in the CPFE calculation, first the average stress within each grain is calculated after which the rank of the predominant twin variant is determined. The predominant twin variant in each grain is the one that accommodates most of the shear due to twinning. In Fig. 10, results of CPFE calculations are presented in red.

Fig. 10. Number fraction of twins that form based on each twin variant. Schmid factors of each twin variant is calculated based on the macroscopic applied stress (blue bars), average measured local stress within parent grain (green bars), or average calculated stress by CPFE within grain (red bars). Variants in x-axis are ordered from maximum to minimum Schmid factor.

The trends shown in Fig. 10 indicate that not all of the twins form based on the predominant variant, although the contribution of this variant is the highest if the local average stress within each grain from experiment is used for the Schmid factor calculation (ALSF). Interestingly, based on the GSF calculation, the third variant has the highest contribution followed by the second and the fifth variants. The fourth variant has the minimum contribution in the 3DXRD results if the ALSF is used as the criterion for variant selection, while the third variant has the smallest population for the CPFE. A comprehensive discussion on the effect of local stresses and stress heterogeneity within grain on the variant selection and Schmid factor is given in section 5.3.

4.7. Stress development in twin and parent pairs

In this section, the measured stresses for individual parent and twin pairs are studied. For simplicity, the CPFE results are not plotted as they have already been discussed in Figs. 7 and 8, or will be discussed in section 5.3.

The internal stresses in the 15 selected parent grains and their 20 twins were calculated from the measured stress tensor and are plotted in Figs. 11a. The parent grains selected have their c-axes within 30° of the tensile axis and appeared in more than one strain step. A schematic diagram of the stress components to be discussed is shown in Fig. 11i, where σ_{cp} is the stress along the parent c-axis direction projected through the twin, σ_n is the stress along the twin plane normal, and τ_{rs} is the resolved shear stress on the twinning plane and in the twinning direction. In the stress-strain plots to follow, the twins are plotted as (gray) circles and the parents as (black) circles. A line shows the mean value and error bars have been omitted for simplicity. It is also important to note that not all parents and not all twins are present at every step, thus, the population of grains used to calculate the mean at each step changes. The average response will be discussed here, but it is not necessarily indicative of the grain level response in a particular grain.

From Figs. 11a to 11d, the average normal stress in the 15 grains and their twins generally follows the macroscopic loading curve and there is only a slight difference between the stress in the twins and the parents. This is also true for σ_{cp} , the stress along the parent c-axis (Fig. 11b). Again, the value is close to the axial stress, which can be expected since the parent c-axis misorientation from the loading direction is not large (Fig. 6c). The twin plane normal stress, σ_n , in the parents is relatively constant at 23-30 MPa throughout the deformation. The average normal stress in the twins is quite close to those of the parent (Fig. 11c). The resolved shear stress on the twinning plane, τ_{rs} , is on average 22 MPa higher in the parents than in the twins (across all three strain steps). While the plots in Figs. 11a to 11d show some

general trends, they do not make clear which twin belongs to which parent. Hence, an examination of some individual twin and parent pairs is needed.

Fig. 11. Definition of the internal stress components indicated in the Middle: σ_{cp} , the stress along the parent c-axis direction projected through the twin, σ_n the stress along the twin plane normal, and τ_{rs} , the resolved shear stress on the twinning plane in the twinning direction. Grain-level stress in 15 individual parents (black circles) and twins (gray circles): (a) σ_{33} , (b) σ_{cp} , (c) σ_n , and (d) τ_{rs} . Grain-resolved stress in a parent and the corresponding twins: (e) σ_{33} , (f) σ_{cp} , (g) σ_n , and (h) τ_{rs} .

Figs. 11e to 11h show the stress evolution in a parent grain with two corresponding twins- a variant 6 twin and a variant 1 twin. The stresses along the normal and parent c-axis directions are generally as expected relative to the mean behavior in Fig. 11a. The evolution of the twin plane normal stress is shown in Fig. 11g. Two parent twin planes are shown, since twinning is observed to occur on two different planes. The normal stress on the variant 6 twin plane is approximately 20 MPa larger than on the variant 1

twin plane until 1.4% strain where the difference becomes smaller. The variant 6 twin is detected with a normal stress smaller than in the parent. The variant 1 twin is detected at a normal stress of 20 MPa, which is very close to the parent normal stress. The latter case agrees with the general trends observed in Fig. 11c, where the normal stress in twins are, in average, close to that of parents. The resolved shear stress on the two twin planes is similar throughout the deformation, varying by a maximum of 5 MPa at 0.2% strain. The variant 6 twin is detected at 0.4% strain when the resolved shear stress is 19 MPa in the parent and -10 MPa in the twin. With continued deformation the resolved shear stress in the twin increase to -7.5 MPa and in the parent decreases to 3 MPa. The variant 1 twin forms when the resolved shear stress in the parent is 7 MPa and forms with a negative resolved shear stress of -1 MPa. These results agree with the average results in Fig. 11d where the resolved shear stress in the twin is found to be lower than in the parent.

5. Discussion

5.1. Methodology and 3DXRD experiment

Details of the sample used for this work and the experimental technique were given in sections 2.1 and 2.2. The specimen used in the 3DXRD experiment had a cross-sectional area of 1mm x 1mm, small enough to enclose a number of grains to give a spotty, indexable diffraction pattern, but large enough to represent bulk material deformation. The high surface-to-bulk ratio means that care should be taken in preparation of the sample surface. Small samples often require manufacturing by EDM, which can lead to an increase of impurities that affect the flow stress of the material. In experiments on Zr plate, Kaschner et al. (2010) found that oxygen and carbon were present along the gauge length of EDM machined samples (1 x 1 x 3mm gauge) to a depth of at least 30 μ m, which appeared to inhibit twinning. However, oxygen and carbon are not believed to have such an effect in magnesium. Regardless, in the present work, the samples were mechanically polished and chemically etched to remove 150 μ m of surface in order to eliminate any effect from the EDM surface finish.

One of the main limitations of the 3DXRD technique is the limited strain at which specimens can be successfully mapped. With increasing plastic deformation, the azimuthal broadening of the diffraction spots increases, which can lead to two problems. Firstly, the diffraction spots may overlap and become difficult to deconvolute. Secondly, the diffraction spot is spread over a large area of the detector so the signal to noise ratio decreases. This means that the spot may disappear and go undetected after thresholding, or, the determined center of mass position of the spot may be inaccurate. This causes the percentage of illuminated volume that is actually indexed to decrease. The spot overlap can be reduced by decreasing the beam size and illuminating a smaller volume so that fewer diffraction spots appear on the diffraction image. Additionally, various thresholding levels can be tried in order to detect both high and low intensity peaks as well as to help with spot overlap. Taking this limitation into account, another difficulty is the softness of the material studied. At low strains the stresses in AZ31B are very low and are in the range of the error bars calculated in this method (see Table 1). Nevertheless, the trends captured for AZ31B here are similar to another study by the authors on Zircaloy-2 (Abdolvand et al., 2014a, b), a much harder material with a smoother grain size distribution.

5.2. General mechanical response and twinning

As explained in the previous sections, most of the results found in this paper are for the very early stages of plastic deformation. From the onset of plasticity up to 1.5% strain (Fig. 4), there is almost no hardening in the material, which is associated with formation of twins (Clausen et al., 2008). This is followed by a significant hardening. Although the beam size and the probed volume changed at each measurement step, the number of twinned grains increased rapidly (Fig. 9). This observation is predicted by the present CPFE simulation, and matches previous observations by EBSD (Chun and Davies, 2011). While the CPFE prediction for evolution of twin volume fraction is very close to the 3DXRD and EBSD results

(Fig. 9d), the CPFE overestimates the number of twinned grains for Step-4 (Table 4.). The overestimation is due to the deterministic assumption for nucleating a twin, where if the twin volume fraction reaches a critical value at an IP, reorientation, i.e. twin nucleation, takes place (Abdolvand and Daymond, 2013a). Previous CPFE modeling of EBSD measurement has indicated that a twin normally nucleates at a triple point where a high stress concentration exists as a result of stress incompatibility, regardless of being in a plastically hard or soft grain (Abdolvand and Daymond, 2013b). In a 3D map of grains, the frequency of triple points in the meshed volume is higher than in a 2D map, which results in overestimation of the number of twinned grains in the CPFE. In the real material there is a high probability of forming twins in such grains, but they might not be resolved in the 3DXRD experiment. The results of the CPFE simulations may be improved by using an irregular mesh where a finer mesh can be used at the grain boundaries or where Gauss points can be placed at the grain boundaries (Gonzalez et al., 2014). This will provide a more realistic representation of geometries of the grain boundaries.

5.3. Schmid factor and twin variant selection

In Section 4.6 it was shown that depending on whether the applied or average local stresses are used to calculate the Schmid factor, different trends in twin variant selection would be concluded. To shed more light on this, a subset of 15 grains was chosen to study stress development in the parent and twins (Fig. 11). The characteristics of these grains are summarized in Table 5. In this table, the c-axis represents the misorientation between the c-axis of the crystal with the tensile axis, SF represents Schmid Factor, and SF_1 represent the maximum SF of the same grain. There are a number of lower ranked variants, however, in all but three cases the SF/SF_1 ratio is greater than 0.9 and the Schmid factor is at least 0.43. Since the grains are highly oriented for twinning, even variants 5 and 6 can have Schmid factors of over 0.95 SF_1 . Of interest are grains 436, 749, and 2438, which contain multiple twin variants of various ranks. The local Schmid factors were calculated using the stress state in the step where the twin was first identified. Statistical studies of twins with EBSD (Capolungo et al., 2009) and with CPFE (Abdolvand and Daymond, 2013a) have shown that when a grain has its c-axis closely aligned with the straining direction, there is a high competition between all variants for forming a twin. In these cases, local stresses play a significant role in defining which variant undergoes twinning. Examining the current data set more closely, the variation of SF_1 for all of the modeled grains found in 3DXRD at Step-4 are plotted in Fig. 12. The Schmid Factors are calculated using the local stresses (LSF) at each IP, and are represented by blue dots. The red crosses represent ALSF from the CPFE (i.e. the LSF averaged over all the IPs in the grain), and the black circles represent ALSF from the 3DXRD measurements. The LSF varies significantly within each grain. In most of the cases, the ALSF from the 3DXRD data falls into the LSF variation zone. With the experimental set up used in this study, it is impossible to define where in a grain a twin nucleates, and consequently it is not feasible to determine which IP correlates with the inception area. Hence it cannot be determined if the twin incepted at the hard spot area in a grain, i.e. the area with maximum SF_1 .

Table 5.: Individual grains chosen to study the effects of local stress on the calculation of the twin Schmid factor

Parent no.	c-axis mis (°)	Twin no.	SF	SF/SF ₁	SF rank	ALSF	ALSF rank
5	10	6347	0.478	0.98	4	0.484	3
11	13	6348	0.439	0.927	6	0.298	2
163	11	6379	0.460	0.953	5	0.497	4
164	15	6557	0.420	0.898	6	0.187	4
		6952	0.461		2	0.443	4
242	16	6605	0.411	0.893	6	0.378	4
436	13	6441	0.475	1.000	1	0.446	3
		6695	0.433	0.912	6	0.489	4
		6825	0.472	0.994	2	0.082	4
749	23	6752	0.328	0.776	6	0.462	6
		7275	0.421	1.00	1	0.457	6
851	21	6367	0.430	0.998	2	0.226	1
895	16	6369	0.442	0.957	5	0.466	3
1002	15	6386	0.465	0.991	2	0.486	4
1148	12	6402	0.479	0.998	2	0.487	2
1215	11	6405	0.477	0.986	3	0.478	4
1227	15	6409	0.444	0.951	5	0.467	2
1497	7	6431	0.491	0.996	3	0.478	3
2438	8	6486	0.490	1.000	1	0.434	1
		6777	0.487	0.993	3	0.416	3

While local or global stresses can change the value of SF₁, the orientation change within each grain resulting from elastic and plastic deformation can also change the value of SF₁. In Fig. 12b, SF₁ for each IP is calculated using the global stress (GSF) and the average value for each grain is indicated with red crosses. Since the same stress is used for all of the IPs, any variation in SF₁ is a sign of orientation variation. Fig. 12b indicates that, firstly, the GSFs in most of the grains are close to 0.5 and secondly, there is a negligible variation in GSF. This result indicates that local stresses make the dominant contribution to the Schmid factor for determining twinning, and not the global stresses. This is sensible considering the local nature of twin inception.

Fig. 12. (a) Variation of the maximum twin Schmid factor (SF_1) in each modeled grain and comparison with measured values from 3DXRD at Step-4. Blue dots represent the calculated values at each integration point using calculated local stress, red crosses represent SF_1 calculated using the calculated average stress in each grain, and black dots represent SF_1 calculated using local stress measured for each grain. (b) Variation of SF_1 calculated at each integration point using the macroscopic applied stress (blue dots) and their average values for each grain (red crosses)

5.4. Stress development in twin and parent pairs

Stress development in some selected parent and twin pairs are studied in Fig. 11. It should be remembered that the stresses measured using 3DXRD are the average stress over the whole grain, and do not resolve stress variation within a grain. CPFE simulations indicate that there is a significant stress variation within each grain (Fig. 7 and 8). Additionally, in the recent work by Balogh et al. (2013), a significant stress variation across a twin boundary in the parent grain is reported. Nevertheless, the trends reported here for the selected 15 twin and parent pairs were also all observed for one twin and its parent in the work by Aydinler et al (2009). In both works, the stress normal to the twin habit plane in the parent and twin are close (Fig. 11c, 11g) whereas there is a significant difference between the shear stresses acting on the twin plane (Fig. 11d, 11h). This difference could originate from the back stresses that are imposed on the twin from the neighboring grains, and depending on the misorientation, neighboring grains can act as a barrier against twin propagation. The high concentration of geometrically necessary dislocations (GND) at the twin tip and in the neighboring grain observed by Khosravani et al (2013) confirms this hypothesis; additionally the rotation induced in neighboring grains by many twins reported by Lind et al. (2014) actually indicate the significant interaction that twin and neighboring grains can have.

6. Conclusions

A statistically comprehensive study on the effect of stress in the development of tensile twinning in a bulk Mg AZ31B polycrystalline was presented. Tensile specimens with axes cut normal to a hot-rolled and annealed plate with strong basal texture were deformed. The center-of-mass position, volume, orientation, elastic strain, and stress of more than 6300 grains in the preload step to 1400 grains in the final loading step were studied in situ using 3DXRD, and modelled using CPFE. The 3DXRD technique imaged a large number of internal grains over several strain steps and identified twins with their parent grains. In MgAZ31B, twinning occurs very early in the deformation, with twins present by macroscopic yield.

A microstructure consisting of more than 1000 grains identified from the measured center-of-mass-positions and grain volumes from 3DXRD was imported into a CPFE code to model stress heterogeneity

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4 within parent grains. The CPFE provides complementary information on stress variation within each
5 grain compared to the average stress obtained by 3DXRD.

6 The error in the stress tensor was large relative to the applied stress, because of the low flow stress
7 characteristic for Mg AZ31B. However, the measured 3DXRD values all fall into the variation zone
8 calculated by CPFE. In both experiment and CPFE modeling it is shown that there is a small variation in
9 stress within each grain in the elastic zone. However, a significant stress heterogeneity is predicted by
10 CPFE which is also reflected in the size of the error bars from 3DXRD measurements. The stress
11 heterogeneity was observed at the onset of plasticity.

12 As expected, crystallographic orientation plays an important role in twin formation with grains oriented
13 with their c-axis parallel to the tensile axis expected to twin. In both the CPFE simulation and the 3DXRD
14 measurement, the number of twinned grains increase with deformation, but the CPFE over-estimates the
15 number of twinned grains at higher strain levels, likely due to stress concentrations at the grain
16 boundaries. Some twins formed in the simulated microstructure were not observed in the 3DXRD
17 experiment, presumably due to resolution limitations in the experiment.

18 The Schmid factor of each twin variant was calculated using both the average local measured stress and
19 the macroscopic stress. Both the 3DXRD experiment and CPFE simulation suggest that twin inception is
20 controlled by more local factors that were not captured in the 3DXRD experiment.

21 Crystallographic orientation itself is unable to account for all twinning behavior. It was shown that not all
22 of the twins formed based on the predominant variant. The lower rank variants can also twin.

23 A significant outcome of this work is the identification of the importance of the interaction between twin
24 and parent grains. In this experiment a population of twin and parent pairs were measured that could be
25 followed through different deformation steps. It is shown that, in average, there is a small difference
26 between stress normal to the twin habit plane in the parent and twin; however this is not the case for the
27 shear acting on the habit plane where the difference is not negligible. It is believed that the back stresses
28 imposed on the twin, by neighboring grains, correspond to the difference in the average shear stress in the
29 parent and twin.

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