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Multiscale deformation heterogeneity in twinning magnesium investigated with *in situ* image correlation

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The complex strain fields that emerge at the onset of extensive deformation twinning in Magnesium alloy AZ31B has been investigated *in situ* with multiscale digital image correlation (DIC). A two camera setup has been used to yield full-field deformation maps on two orthogonal faces of a gradually strained compression sample whose crystallographic texture favors the $\{10\bar{1}2\}\langle\bar{1}011\rangle$ tensile twin mode. Deformation fields have been determined at subgrain resolution (DIC subset and nominal grain size, 12 and 50 μm, respectively) on the primary examination face that is scanned with a microscopic lens at each load. In an *in situ* setting, a unique combination of microscopic DIC resolution and statistical significance (~5000 grains) is achieved that enables a full bridging of the crystallite and sample scales at each load point. Intense strain heterogeneity is revealed abruptly at all length scales in the form of ±45° banded strain patterns: at the macroscale, the strain in the sample is locally accommodated by the sudden emergence/advance of shear bands whose macroscale strain content at initiation is around 1%. Bands from conjugate +45° and −45° families exhibit sequential activation. At the granular scale, another set of ±45° strain patterns are resolved inside a macroscopic band of either family with very high strain heterogeneity levels, whose extent is comparable to the twin transformation strain (principal values ±6.5%). Traversing the initial regions of the twin plateau in fine steps with *in situ* observation, the strain heterogeneity is found to explode, rather than gradually evolve, yielding uniquely shaped strain histograms at the subgrain scale which persist until the next macroscopic band arrives at the particular locality.

1. Introduction

Deformation twinning is widely observed in strained hexagonal-close-packed (HCP) crystals (Christian and Mahajan, 1995). It is particularly associated with strain accommodation along the *c*-axis, which is perpendicular to the glide direction, ⟨a⟩, of all favorable slip systems. Unlike dislocation slip, twinning activation is unidirectional with the straining sense. In an HCP polycrystalline aggregate, this characteristic further specializes the subset of grains that are suitably oriented to undergo twinning. Inside such a (parent) grain, twin band(s) form abruptly, incurring a large transformation shear strain (typically >10%) associated with the crystallographic reorientation. With the high localized shear and the mechanism being available to specific grains, high intergranular (type II) and intragranular (type III) stresses are generated about the twin zones. The effect of twinning is evident in macroscopic observation by, e.g., the sharp tension–compression anisotropy in textured samples (Kleiner and Uggowitzer, 2004) which leads to highly distorted hysteresis loops in cyclic loading (Lou et al., 2007;

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Wu et al., 2008a; Yin et al., 2008; Zhang et al., 2011). A fundamental understanding of twinning, however, requires observations of this phenomenon at the grain and subgrain scales.

In this regard, the contribution of *in situ* neutron (Agnew et al., 2006; Brown et al., 2005; Clausen et al., 2008; Muransky et al., 2010; Muransky et al., 2009; Wu et al., 2008b; Xu et al., 2008) and X-ray (Aydıner et al., 2009; Merkel et al., 2009) diffraction experiments has been significant. Neutron diffraction results for lattice strain are resolved over grain sets of a certain orientation family—the direct observation is not at the grain scale. Stress state can be predicted on individual *orientations*, however, when complemented with polycrystalline modeling (Abdolvand and Daymond, 2012; Beyerlein and Tome, 2008; Clausen et al., 2008; Fernandez et al., 2011; Hama and Takuda, 2011; Oppedal et al., 2012; Proust et al., 2009; Turner and Tome, 1994) that also predict the strength of fundamental slip/twin modes. Naturally, the validity of these results is governed by the adequacy of the model as well. Neutron diffraction experiments have excellent statistics, and experiments on heavily pre-textured samples allow singling out results from emerging twin families. It is in these results, it was first observed that twins emerge with a very distinct stress state (Brown et al., 2005). A large portion of these studies consider Magnesium alloys due to the activation ease of the tensile twin mode; the critical resolved shear stress of the $\{10\bar{1}2\}\langle\bar{1}011\rangle$ twin system in lightly-alloyed AZ31 is inferred to be only higher than that of basal slip $\{0001\}\langle11\bar{2}0\rangle$ (see, e.g., Clausen et al. (2008), Neil and Agnew (2009)). Recently, with synchrotron X-ray diffraction on the same alloy, observation of an individual grain inside the bulk could be possible (Aydıner et al., 2009). Here, the *average* stress tensors of a parent grain as well as its twin children have been measured *in situ*. This direct observation confirmed neutron diffraction deductions that, in the twin-transformed volume, the stress component along the straining direction is relaxed whereas reaction stresses are incurred in the lateral direction. Acquiring statistical significance, however, is difficult with this technique; there is no intragranular resolution; and there is no morphological detail on the grain or its neighbors. With the footing in Oddershede et al. (2011) and Shmidt et al. (2004), it is in principle possible to remedy the latter two aspects; but an *in situ* loading experiment with three-dimensional *microscopy* over a considerable volume of the aggregate pushes the synchrotron beam time requirements towards impractical.

Meanwhile, morphological detail over grain neighborhoods with intragranular resolution and high statistical significance are practical with surface microscopy. This led to another field of experimentation that analyzes microscopic images for deformation. By tracking the displacement of (introduced) surface features, full field deformation is determined over the surface of a polycrystalline aggregate (Abuzaid et al., 2012; Bodelot et al., 2011; Carroll et al., 2010; Delaire et al., 2000; Di Gioacchino and da Fonseca, 2013; Efstathiou et al., 2010; Heripre et al., 2007; Littlewood and Wilkinson, 2012; Padilla et al., 2012; Raabe et al., 2001; Sachtleber et al., 2002). Efstathiou et al. (2010) provide an extensive literature review, classifying over two displacement-tracking techniques: digital image correlation (DIC) and the grid method, the latter typically employed with electron microscopy. The ensuing measurement of *total* strain (and rotation) by these methods is in contrast to the lattice strain (a measure of stress) determined by diffraction, making the two fields complementary. The particular contribution of microscopic full-field deformation has been in relating the strain localization that necessarily develops in a polycrystal (e.g., Ashby (1970)) with the morphology of the grain neighborhood. The observed plastic localization in the vicinity of grain boundaries are thus anticipated; however, further structure is revealed to the process, e.g., the formation of strain localization bands that often proceed through several grains and are approximately oriented along maximum-shear directions (±45° to a uniaxial load) in cubic (Sachtleber et al., 2002; Schroeter and McDowell, 2003) and HCP (Efstathiou et al., 2010; Heripre et al., 2007; Padilla et al., 2012) metals. Random-textured pure Titanium is considered in Efstathiou et al. (2010) where twinning was not physically pertinent. Beyond presenting the strain heterogeneities in the residual deformation patterns, this *ex situ* optical DIC study is technically significant in two respects. First, the effect of measurement length scale (typically described in terms of optical resolution: field length per detector pixel) on DIC results has been characterized. Imaging with optical resolutions ranging from 1.359 to 0.087 μm/pixel, it is clearly shown that the DIC-detected level of strain heterogeneity is higher at higher magnification. Second, scanning (see also Carroll et al. (2010)) of high-magnification optics over the surface has been implemented to cover the same field of view with lower magnifications. Scanning not only enhances grain statistics but allows linking the observations to the upper (e.g., macroscopic) length scale. Majority of microscopic full-field deformation studies are *ex situ* due to experimental complexities, e.g., in optical DIC, higher magnification comes with smaller depth of fields. The *in situ* work by Padilla et al. (2012) with 1 s^{-1} strain rate, suffered defocusing, and had to constrain attention to small regions (tens/hundreds of grains) without scanning. It presents strain heterogeneity in Zirconium alloy Zr 702 with rolling texture and its connection to local orientation distribution. Twinning consideration is not central in this work with the *relatively* subdued activity of twin modes in this HCP alloy. Efstathiou and Sehitoglu (2010) considered twinning in an FCC Hadfield steel, presenting its effect on hardening. However, this study on a single crystal, naturally, does not fit the described polycrystalline interaction focus. Martin et al. (2013) perform high-resolution strain mapping on Mg–1Zn–0.5Nd in a configuration that suppresses twinning. Regarding twinning, a recent study by Hazeli et al. (2013) on AZ31 is noteworthy despite the fact that, with its macroscale DIC measurement, it does not strictly fall in the described literature. In this work, *macroscale* strain heterogeneity in the form of shear bands (Barnett et al., 2012) has been identified. The relation to profuse twinning has been established with accompanying acoustic emission (see also Muransky et al. (2010)) and electron backscatter diffraction measurements.

In the current study, *microscopic* DIC is used to characterize the spatial distribution of *in situ* deformation fields in an HCP polycrystalline aggregate that exhibits substantial twinning. This work is particularly motivated by the anticipation that twins would promote mesoscale strain heterogeneity; the exact nature of which will be under investigation. To this end, Magnesium alloy AZ31 is considered in likeness to the diffraction studies that target HCP twinning physics. An in-plane compression