Deformation twin nucleation and twin variant selection in single crystal magnesium as a function of strain rate

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Abstract

Deformation twinning is an important deformation mechanism in a variety of materials, including metals and ceramics. This deformation mechanism is particularly important in low-symmetry hexagonal close-packed (hcp) metals such as Magnesium (Mg), Zirconium (Zr) and Titanium (Ti). Extension twins in Mg, Zr and Ti can accommodate considerable plastic deformation as they grow. Thus, the rate and the mode of twinning greatly influences the mechanical behavior including strength and ductility. Herein, we study deformation twinning in terms of nucleation, twinning mode and variant selection as a function of strain rate in Mg single crystal (considered as a model material). We show that twin variant selection is sensitive to the loading rate, with more twin variants nucleating at the dynamic strain rates. Low Schmid factor twin variants (one of them being a double extension twin variant) were also found at the dynamic strain rates. Further at high strain rates, the first twins generated do not thicken beyond a critical width. Instead, plasticity proceeds with nucleation of second generation twins from the primary twin boundaries. The rates of area/volume fraction evolution of both generations of twins are found to be similar.

Keywords: Dynamic plasticity, deformation twinning, twin variant selection, twin tip velocity, Mg single crystal
1. Introduction

Deformation twins have been identified as a critical deformation mechanism in atomic structures with low stacking fault energy and/or low crystal symmetry (such as body centered cubic and hcp metals [1]). For instance, twinning can result in significant elongation at the onset of plasticity in Mg [2], contribute to the remarkable ductility of Ti [3], cause strain hardening in Ti and Mg [4, 5], and also lead to crack initiation under low-cycle fatigue in Mg [6]. In the case of high-stacking fault energy face-centered cubic (fcc) materials, twinning has been also observed under extreme conditions such as cryogenic temperatures [7, 8], shock loading conditions [9], or when grain sizes are on the order of less than one-hundred nanometers [10]. Twinning also plays an important role in the physical properties of ordered intermetallic alloys [11]. For example, it is reported that twin geometry and spacing determines the electronic quality of layered indium phosphide wire semiconductors since the electron wave function is discontinuous at a twin stacking fault, which leads to a reduction in the mobility of charge carriers [12]. These examples highlight the need for the understanding of the energetic, morphological and kinetic aspects of deformation twinning. This paper considers the factors which influence twinning mode variant selection, twinning kinetics and volume fraction evolution.

1.1. Crystallographic Theory of Twinning

In general, plastic deformation in hcp metals takes place by dislocation slip, deformation twinning or a combination of both. Each dislocation slip system is typically assumed to have a critical value of resolved shear stress necessary to become active, commonly referred to as the critical resolved shear stress (CRSS). Unlike dislocation slip, deformation twinning does not necessarily activate at a particular CRSS [13] because the normal stresses that develop
at the twin boundary can influence mechanism activity. Therefore, a geometric approach using Schmid factors may be more appropriate \[14\]. In this context, the Schmid factor (SF) indicates the slip plane and slip direction of a loaded crystal, which sustains the greatest magnitude of shear stress. As a result of twinning, the original lattice (lattice below the twin plane \(K_1\), indicated with a horizontal red line in Figure \[1\]) is re-oriented by displacements which are equivalent to a shear of the lattice points.

The invariant plane of this shear is called the twin plane and is denoted by \(K_1\) with normal \(\hat{n}\) where the shear direction is \(\eta_1\) and the magnitude of shear is \(s\). When twinning occurs the twin plane \((K_1)\) does not change its position, instead the so-called “conjugate” plane \(K_2\) displaces to \(K_2'\) by the twinning shear. The direction \(\eta_2\) rotates to \(\eta_2'\) about a rotation axis in the \(K_1\) plane. In bcc and fcc structures twinning takes place by homogeneous shear along the shear direction. However, in hcp structures additional atom displacements are often required in a direction different from the lattice shear. These atom movements are known as *shuffles* \[15\]. In hcp crystals twinning can accommodate strain along the c-axis of the crystal lattice by either extension \(\{10\bar{1}2\}\) or contraction \(\{10\bar{1}1\}\) twinning, depending on whether the imposed strain tends to extend or contract the c-axis respectively. Since the focus of this study is on the kinetics of extension twins in Mg, we discuss contraction twins only when necessary. When the c-axis is placed in tension, two extension twin modes (systems): (1) \(\{10\bar{1}2\}[\bar{1}011]\) and (2) \(\{11\bar{2}1\}[\bar{1}\bar{1}26]\) have been observed in Zr, Ti, and Hafnium (Hf) \[16, 17, 18, 19, 20\]. However, in Mg and Zinc (Zn) only one extension twin mode \(\{10\bar{1}2\}[\bar{1}011]\) has been commonly observed \[21, 22\].

In Mg, there are six (6) possible primary extension twin variants (occurring on crystallographically equivalent planes) for each extension twin mode \[23\]. Under loading, each twin mode and twin variant can have a different SF for the same loading direction \[24\]. Therefore,
during deformation, only a limited subset of the six possible twin variants will be activated (in principle). Most descriptions of twinning, relate the favorable twin modes to a small shear strain and minimal atomic shuffling \[3, 13, 25, 15\]. Note that the twin morphology is also believed to be influenced by the twinning shear, with the twin having the traditionally observed wide lenticular shape \[15\] when the shear is small.

1.2. Significance of Twin Modes, Variants, and Volume Fraction

For the hcp structure the ideal axial ratio \((c/a)\) for perfect packing of spheres is 1.633. This ideal axial ratio separates hcp metals into two groupings: those that have an axial ratio greater than 1.633, such as Cadmium (Cd) and Zinc (Zn), and those that have an axial ratio less than 1.633, such as Cobalt (Co), Ti, Zr, Mg, and Hf. Metals with larger axial ratios tend to exhibit reduced numbers of twinning modes compared to those with smaller axial ratios \[3\]. The fact that hcp alloys made of crystals with lower \(c/a\) exhibit better ductility \[3, 2\] indicates the significance of the number of twinning modes (and thus variants) on the mechanical response as exemplified by the macroscopic stress-strain curve.

The macroscopic stress strain relationship typically depends on the strain rate, temperature and internal variables (e.g., dislocation and twin characteristics and densities). Both the dislocation substructure and the twin substructure evolve during deformation. The evolution of dislocation substructure has been described in detail in fcc systems, e.g. by \[26\]. Many of these concepts carry over to hcp systems. However, the evolution of the twinning substructure is a matter of great current interest. For instance, from the modeling perspective, the activation and volume fraction of different twinning systems as a function of strain rate (if known) can be incorporated into the widely used visco-plastic self-consistent (VPSC) anisotropic model developed by \[27\], which enables accounting for the shear contributions of
twin modes and variants. Specifically, VPSC based models must make choices on the twin mode selection under applied conditions [28]. This is also true of all other crystal plasticity based models. This highlights the need for quantitative understanding of twin variant selection and growth. There is some conflict in the literature as to how to properly incorporate such phenomena. Some work suggests that the twin volume fraction is only a function of stress and thus is independent of strain-rate [1, 29]. Other experimental studies however, suggest that the rate of growth of twin volume fraction increases at higher rates of loading [8].

It has been stated that “the central problem of the crystallographic theory is to understand the factors which influence the choice of twinning mode, and ultimately to predict which twinning mode or modes will operate in a given crystal structure.” [1]. From crystallographic theory and crystal plasticity standpoints it is equally important to understand: (1) the plastic strains associated with slip and twinning, and (2) also the change of the crystal orientation which may promote activation of other slip or twin systems. The plastic strain rate contributed by a specific twin variant in a single crystal can be estimated by:

\[ \varepsilon_{twin} = \dot{f}_{twin}\gamma\mu_{twin} \]

where \( \dot{f}_{twin} \) is the rate of evolution of that variant, \( \gamma \) is the corresponding twinning shear and \( \mu_{twin} \) is the Schmid factor for that variant. The twinning shear is purely a function of crystal lattice parameters. Hence, the strain caused by extension twinning is dependent on the twin volume fraction evolution and the specific crystal structure. Therefore, identifying and quantifying twin modes and variants is critical for accurate descriptions of plasticity. Consequently, several studies have aimed at discovering activated twin modes and variants in hcp metals (especially in Mg) both at the single and polycrystalline level.
e.g. [15, 31, 32, 33, 24, 34]. However, to the best of the authors’ knowledge, this is the first study that differentiates twin modes and variant selection in Mg single crystals as a function of loading rate.

The total twin volume fraction rate in Mg depends on the rate of nucleation, propagation and thickening of the nucleated twins. Experimental evidence points towards twin nucleation typically occurring at grain or tilt boundaries e.g., [35]. Recent molecular dynamics simulations [36] have suggested that the twin nucleation process requires the coalescence of multiple twinning dislocations to form a stable twin embryo [37]. Propagation and thickening events then appear to occur through the interactions of lattice (i.e., matrix) dislocations to create twinning dislocations that travel along the twin boundary and in so doing increase the twin thickness by two atomic layers [38, 39, 40, 41, 42]. While there have been strides made towards understanding the necessary conditions for nucleation, the processes of twin tip propagation and thickening are yet to be adequately explored. Experimental characterization of the rates of twin growth in hcp materials is rare. An exception is the work of Brunton & Wilson [43] on twinning in Zn and Sn, who measured twin tip velocities of approximately 600 m s$^{-1}$ using knife edge loading on Zn single crystals; knife edge loading leads to a complex non-uniform stress state. To the authors’ knowledge, there have been no direct measurements of twin tip velocities in single crystals under nominally uniform stress conditions until our recent work [44]. Modeling approaches have predicted twin growth velocities over a wide range, from 400 ms$^{-1}$ in MD simulations of nanopillars [45] to approaching the material shear wave speed ($c_s$) in the material in continuum models [46].

In general, the literature on twinning in metals is replete with generic statements about twin velocities approaching the speed of sound (for eg. [13, 47]). The assumed twin tip velocities approaching the speed of sound in the material are associated with another oft-used
statement: that deformation twinning, as a mechanism, is insensitive to the rate of loading, e.g., [48]. This latter statement also implies that twin nucleation processes and twin variant selection are insensitive to rate of loading. We show here that these statements are not true at high strain rates.

In this work, we perform experiments at two different strain rates of loading and characterize, through post-mortem microscopy, the crystallographic nature of twin activity as a function of strain rate. In addition, using in-situ ultra-high speed imaging, we have described in another manuscript [44] the spatial evolution of twins at high temporal resolutions at high rates of loading. While most of this paper is focused on the variant selection, some discussion of the twin nucleation and growth is necessary to place our findings in context.

2. Methodology

2.1. Quasi-static Loading

Cuboidal compression specimens were cut by electric discharge machining (EDM) from 99.999% pure Mg single crystals (Metals Crystals and Oxides Ltd, UK) following ASTM standard E9 (length to side ratio equal to 2 for a 6 mm side length). The quasi-static compression tests were carried out at constant strain rate of $\dot{\varepsilon} = 4.5 \times 10^{-4} \text{s}^{-1}$, with compression along the a-axis at room temperature. The strain field was calculated using the digital image correlation (DIC) technique. The strain uncertainty for DIC measurements reported in this article was $\pm 150 \mu\text{m/m}$, achieved by a facet size of $25 \times 25$ pixels, a step size of 12 pixels and 52% overlap. The reported strain in the quasi-static stress-strain curve shown in Figure 2 was obtained from the average full-field strains along the loading axis on the (1120) plane.
2.2. High-strain Rate Loading

Cuboidal specimens with nominal dimensions of $3 \times 3.5 \times 4 \ mm^3$ were cut using EDM. The cut surfaces were chemically polished using 10% nitric acid in water to remove the EDM recast layer. The loading faces of the specimens were lightly polished to remove surface damage and checked for planarity before high strain rate testing was performed using a compression Kolsky bar apparatus [49]. Compression was performed along the crystallographic a-axis $\langle 11\overline{2}0 \rangle$ of the hcp structure at room temperature.

Two sets of experiments were performed: (1) specimens recovered at $\sim 3\%$ macroscopic strain for post-mortem microscopy (2) specimens deformed to about $10\%$ macroscopic strain with in-situ imaging and stress-strain measurement. Recovery was performed using a steel limit collar that slides over the specimen and is slightly shorter than the specimen (in this case $97\%$ of the length of the specimen). The steel collar was cut in half along the normal to the cross-section to allow for in-situ imaging during recovery as well. The twin evolution study utilized a Kirana high speed camera capable of capturing $924 \times 768$ pixels of data at $5,000,000$ frames per second. Magnification optics are used to achieve a resolution of $\sim 5\mu$ per pixel.

It is worth noting that in such plastic high strain-rate experiments, a uniform stress state is typically achieved after the longitudinal wave (i.e., loading wave) travels at least five times back and forth through the specimen [50]. Therefore, for the experiment shown, equilibrium occurs about $\sim 5 \mu s$ after the beginning of deformation (i.e., before image 2 in Figure 4b).

2.3. Post-mortem Microscopy

Samples (of thickness $\sim 500 \ \mu m$) for post-mortem electron back-scattered diffraction (EBSD) were cut from the deformed samples (both quasi-static and dynamic loading) parallel
to the (1010) plane using a diamond wire at a low rate with low coolant water pressure. The sliced samples were then etched using a 10% nitric acid solution in distilled water to remove the wire damaged layer. Further, a surface finishing process was conducted by using electrochemical polishing in a 90% methanol and 10% nitric acid solution with an applied voltage, current, time and temperature of 10, 20V, 1min, and -40°C, respectively. The final surface finish was achieved by Ar⁺-ion milling using a Fischione Model 1050 TEM Mill. Milling was conducted for 5-10 min at 5° angle with voltages of 4 kV. EBSD data was collected using a TESCAN scanning electron microscope equipped with an EDAX EBSD detector. The accelerating voltage and working distance were set at 20 kV and 19.5 mm respectively. Orientations were recorded with a spatial resolution of 0.6 µm on a hexagonal grid.

3. Results

3.1. Mechanical Behavior

The true stress-strain curves for Mg single crystals compressed along the a-axis are shown in Figure 2 for both quasi-static (blue) and dynamic (red) rates. A characteristic sigmoidal profile is observed for both the curves. This profile has been associated with the activation of twinning and its interplay with dislocations e.g., [51]. Figure 2 suggests a considerable difference between the flow stress and strain hardening rate under dynamic loading compared to the quasi-static loading, which (as shown in the following section) cannot be solely attributed to the dislocation glide sensitivity to the strain rate previously noted by other researchers e.g., [52]. Note that at the dynamic rates, wave propagation effects in the experiment do not allow for an accurate representation of yield stresses. Flow stress beyond ~ 2% strain can be comfortably used to evaluate the stress-strain response at high rates in our experiments.
Both flow stress and strain hardening change significantly as the loading rate is increased. These rate effects are coupled to twin volume fraction evolution and twin variant selection, as we show subsequently.

3.2. Post-mortem Characterization of Twinning at Quasi-static Loading

EBSD data from a specimen that was deformed to 2.5% macroscopic strain at quasi-static rates is shown in Figure 3. Figure 3a shows an Inverse Pole Figure (IPF) map with the colors indicating the orientations of the crystal at every spatial point analyzed. Figure 3b shows the pole figure indicating the dominant poles from the data with the inset schematic describing the physical orientation of each pole with respect to the matrix (M). All the recorded twins were extension twins reorienting the matrix by 86°. Two extension twin variants (labeled T1 and T2) with minor misorientation (7°) with respect to each other were observed (Figure 3a&b).

3.3. Twin Evolution at High Strain Rate Loading

Figure 4a shows a schematic of the loading orientation, with a magnesium single crystal cuboid compressed along the a-axis ⟨1120⟩ at a nominal strain-rate of \( \dot{\varepsilon} = 1400 \) s\(^{-1} \) (with a rise time to max strain rate \( \sim 16 \) µs). This loading configuration results in an extension of the c-axis of the crystal. High-speed imaging is used to observe the (1010) crystallographic plane \textit{in situ} (Figure 4b) at 5 million frames per second during the loading (an example video is available online). As the stress level increases, extension twins nucleate and propagate along \{10\bar{1}2\} planes; nucleation is first observed 6 µs after loading begins, at a compressive stress of \( \sim 10 \) MPa (and an approximate total strain of only \( \varepsilon = 0.18\% \)). Figure 4b shows non-consecutive frames except for the second and third images, which show the movement of a twin tip during the 200 ns between frames. The fifth image is a magnified view of a region of...
the fourth image, and shows that a second generation of extension twins is nucleated from the first generation twin boundaries (blue and red arrows) at around 9 $\mu s$. Twins nucleating from existing first generation (or Gen1) twins are termed as second generation (Gen2) twins. This terminology is used for identifying the specific twins being discussed and does not describe the crystallographic nature of the twin. An extensive conjugate twin network has formed by 13 $\mu s$ (the sixth image).

The velocity history of a specific representative first generation twin tip is extracted from the image sequences and shown in Figure 4c. The twin tip initially propagates at about 1800 m s$^{-1}$, but then rapidly drops in speed to $\sim$1000 ms$^{-1}$. The first drop in velocity appears to occur after a time consistent with interaction of the propagating twin tip with the specimen boundaries through longitudinal stress waves. The subsequent twin tip velocity of $\sim$1000 ms$^{-1}$ is about one-third of the shear wave speed in Mg ($c_s = 3120$ ms$^{-1}$), well below the acoustic velocities sometimes cited in the literature [47, 13].

The second generation twins (Gen2) propagate at much slower speeds (Figure 4c) than the first generation twins, $\sim$200 ms$^{-1}$ vs. $\sim$1000 ms$^{-1}$. The lower Gen2 twin tip velocity may be related to a lower local driving stress, or to interactions with the nearby boundaries of other first generation twins. Note that there is also negligible thickening of the Gen1 twins (the typical thickness ranges between 40 – 60$\mu m$). Thus, nucleation of Gen2 twins occurs preferentially to thickening of the existing Gen1 twins at high strain rates. Recent three-dimensional x-ray diffraction experimental studies have examined the local strain state within deformation twins and neighboring regions [53, 54, 55]. These studies have found strain gradients across grains [54, 56, 57] that contain deformation twins, as well as gradients within the twinned region themselves [53], providing direct evidence of heterogeneous stress and strain states at and near twin boundaries. These heterogeneous states presumably drive
the nucleation of Gen2 twins.

Quantitative image analysis also provides the evolution of the area fraction (on the (1010) crystallographic plane) of the first and second generation twins as a function of time (Figure 4d). While the Gen2 tip velocities are an order magnitude lower than that of the first generation twins, the rates of area fraction evolution of the two classes of twins are similar. Given the difference in velocities, the rate of nucleation of the slower Gen2 twins must be substantially higher than that of the faster Gen1 twins. The dynamics of twin evolution has been discussed in greater detail in a separate manuscript [44]. Finally, it is noted that the observed nominal nucleation stresses of the Gen1 twins are of the order of 20 MPa, substantially smaller (as expected, given heterogeneities) than the GPa nucleation stresses computed from MD simulations of nanopillars [45].

While the nucleation of second generation twins has a clear significance in terms of the evolution of twin volume fraction, the crystallographic nature of these second generation twins also provide clues to the rate dependence of twin variant selection. This is the primary objective of this study.

3.4. Post-mortem Characterization of Twinning at High Strain Rates

Figure 5a shows an example specimen region on which the EBSD measurement was performed. Note that the in situ images and EBSD maps were collected from different experiments with nominally similar loading rates and boundary conditions. The resulting crystallographic orientation color map (IPF) is shown in Figure 5b. Note that this sample was also recovered at the same strain as the quasi-static experiment. The IPF map shows the formation of twins labeled T2 through T6 based on crystallographic orientation. Twin variants are characterized and named in the same manner as a previous study by Zambaldi et
al. [58]. The misorientation angles between the matrix and twinned regions are provided in Table 1. Dashed lines (figure 5b) are used to indicate the basal plane orientation within a few of the twin variants. A pole figure with the imaging surface normal to the page is provided in Figure 5c: indicating the orientations of the (0001) c-axis direction for each twin observed. The first generation twins (denoted T3 and T4) occur on (11̅02) [10̅1̅1] and (1̅012) [101̅1] crystallographic systems respectively. The second generation twins (Figure 4b) shown have orientation T2 and T6 and are characterized as (1̅102)[1̅101] and (01̅12)[011̅1] respectively. We also observed formation of twins within a primary twin labeled T5 ((01̅12)[011̅1]).

In the deformed specimen we observed four primary extension twin variants (T2,T3,T4, and T6) and one double twin/ secondary twin variant (T5) (Figure 5b&c). The pole figure in Figure 5c suggests that twins labeled T2, T3, T4, and T6 are primary extension twins reorienting the matrix by ~86°. Careful analysis (see Table 1) of the misorientation between T5 and T3 reveals that T5 is a double extension twin (i.e., an extension twin within an extension twin). Comparison of these observations with our quasi-static work (figure 3) and other work [59] at lower rates of loading demonstrates that twin variant selection is influenced by the applied strain rate.

A comparison of figure 5b and figure 3a suggests that at high strain rates the deformation proceeds by multiple twin variant nucleation from the boundary of the first generation twins, while under quasi-static rates the deformation proceeds through thickening of the first generation twins.
3.5. Schmid Factor Analysis

A Schmid factor analysis of the observed primary extension twin traces seen at the high strain-rates is presented in Figure 6 where traces are overlaid on a snapshot to provide context of the numbering used. In Figure 6b, pole figures provide the orientation of the c-axis, a-axis, and the direction perpendicular to both the c and a axis (the prismatic pole). In Figure 6c, representations of the hcp unit cell with the twin planes (gray), loading direction (vertical green arrow) and the twin direction on the twin plane (red arrow) are provided. Beneath each unit cell the corresponding Schmid factor is provided. It is clear that most of the twins that we observe have high Schmid factors, however two low Schmid factor variants (T2 and T5) have also been observed. A further analysis of these observations will be discussed in section 4.

3.6. Double Twin Variant Groupings

From the EBSD characterization data the misorientation between the primary extension twin T3 and the secondary twin T5 can be determined. For an extension twin, the misorientation relationship between the twin and matrix is $86^\circ$. For twins T2, T3, T4 and T6 the measured value from EBSD data closely matches the predicted rotation, see table 1 and figure 5.

In total there are four types or crystallographically equivalent groupings of the secondary extension twins. These four groupings are summarized in figure 8 which presents three dimensional representations of the matrix, primary twin and secondary twin orientations. For similarity the matrix orientation (M) and the primary twin (T3) have been colored to match that of the pole figures shown in figure 5. The secondary twins are colored grey for the orientations that were not observed in this study, and in orange (T6) for the orientation
that was observed in this study. The misorientation between the basal plane of the matrix material and the basal plane of the secondary twin for each grouping are $0^\circ$, $7^\circ$, $60^\circ$, and $60^\circ$, respectively. Combining the above with EBSD data of the orientation of T5 with respect to the matrix, it becomes apparent that T5 falls into what is classified as type three in figure.

4. Discussion

4.1. Twin Characteristics as a Function of Strain Rate

Figures 3 and 5 show clearly the effect of strain rate on the number of variants selected. It is seen that although the stress state and total strain are similar (though not the same) in both experiments, the governing deformation mechanisms are different, particularly the number of twin variants observed and their morphology.

After the formation of the first generation extension twins, further strain can be accommodated through a combination of the following: (1) thickening of first generation twins, (2) nucleation of additional first generation twins, (3) nucleation of second generation twins (as discovered from our experiments), and (4) secondary twinning within the first generation primary twins and dislocation motion. The *in situ* ultra-high speed imaging of twin propagation shown in Figure 4b demonstrates that the twin tip propagation is much faster than the twin thickening at high rates. Further, nucleation of additional second generation twins is preferred over twin boundary motion at higher rates. The conventional view is that lateral twin growth (i.e. thickening) continues until the twinned region consumes the available matrix, as reported over the last three decades. However, the *in situ* ultra-high-speed observations presented here suggest that this view may only be valid under low strain rate loading. From these observations it is evident that twin nucleation processes depend on the rate of the loading, and thus the deformation twinning mechanism itself thus depends on
the rate of loading at this "grain" scale. The competition between twin thickening and the nucleation of new narrow twins (as we observe) is yet to be understood.

In the dynamically deformed specimen we observe five twin variants (figure 5b&c), while in the quasi-statically deformed specimen (figure 3) only two variants were observed. The variants observed in both these cases were extension twins (causing extension of the c-axis). The activation of some twin variants with low Schmid factors was also observed only at the higher rates (figure 6). The additional twin variants observed were found to nucleate at high rates and qualitatively seem to be associated with the nucleation of second generation twins. While we cannot measure dislocation activity directly, dislocation slip is expected to play an important role in both cases as well.

In the high strain rate loading case one of the low Schmid factor variants is a single secondary twin (or a twin within a twin), identified as T5 in Figure 5b. Previous studies of the formation of secondary twins ([60, 61]) have shown that the secondary twin selection does not follow Schmid predictions and instead is favorable for some orientations, perhaps due to minimization of strain incompatibility between primary extension and secondary contraction twin planes [61, 60]. Cases of secondary extension twins within primary extension twins have also been observed under high strain rate uniaxial strain [62], but these have not been studied in sufficient detail to make similar claims.

4.2. Second Generation (Gen2) Twins at High Strain Rates

In the high strain rate experiments, after the Gen1 twins reach a thickness of \( \sim 50\mu m \), the twins continue to lengthen but do not thicken. Once twin thickening is no longer a preferred mechanism, deformation proceeds with multiple twin variants nucleating at the boundary of the first generation twins. Post-mortem characterization (figure 5) has shown some of these to
be of a lower Schmid factor. One possible explanation for this is that the local stress caused by the two lattice plane steps gives rise to the development of a non-coherent boundary. Other sources of defects and local stresses at the twin boundary arise from interactions of basal dislocations gliding into the primary twin boundary shown in Figure 7. Depending on the Burgers vector of the impinging basal dislocation, the dislocation can either cross the boundary without leaving debris at the interface [63], or dissociate at the twin boundary into interfacial defects [38]. Therefore, it is plausible that the local stress state (see Figure 7) provides appropriate conditions for the nucleation and propagation of low Schmid factor twin variants at the interface of first generation twins. Because of the restrictions placed on the rate at which deformation can be accommodated via first generation propagation and thickening, and the available high local stresses at the twin boundary, further macroscopic deformations are perhaps then accommodated through additional nucleation of Gen2 twins. The formation of second generation twins hence appears to be an alternative mechanism activated to accommodate local strain incompatibilities caused by first generation twins in the limited time available during a high strain rate experiment. The nucleation of second generation twins occurs when the thickness of the first generation twin reaches a critical value. This is also an important question for constitutive modeling at high rates, as the strain hardening is sensitive to twinning dynamics and its interaction with dislocations. It is however useful to note that the number of second generation twins with low Schmid factor (T2 and T5) is much smaller than the number of those with high Schmid factor (T6).

The reduced tip velocities of the second generation twins can be attributed to a combination of: (1) defect content in the non-twinned regions and (2) back-stresses due to the existing first-generation twin boundaries. As the Gen1 twins are growing, the dislocation density grows in the non-twinned matrix regions (primarily through basal slip due to its
low CRSS). The second generation twins must thus travel through regions that have greater defect populations. Assuming that the tips of the second generation twins are composed of an ensemble of dislocations, it follows that interactions of the tip with matrix dislocations would reduce the observed propagation velocity. Further as the second generation twins approach and interact with the twin boundaries of Gen1 twins, back stresses develop. The magnitude of the back stresses has been shown to be dependent on the strain accommodation in neighboring regions [53]. To date the precise short range interactions that occur between an advancing twin tip and a stationary twin boundary are yet to be clarified. However, a complementary scenario of an advancing twin boundary and a stationary twin tip has been imaged by Morrow et al during *in situ* straining using TEM [64]. Just prior to the interaction of the twin tip and advancing boundary, the twin tip was seen to transition from sharp to blunt. The transition of tip from sharp to blunt points to a complex interaction that could indicate propagation of dislocations along the boundary to move to the leading edge of the twin forming the blunt shape.

It is of course generally the case that both dislocation slip and twinning are occurring during these rapid deformations, and the interactions of the evolving dislocation density and the evolving twin populations are intimately related. Careful discrete dislocation (as well as discrete twinning) simulations will be useful to understand these fundamental laboratory observations.

4.3. Twinning Dislocation Dynamics at High Strain Rates

Most of the available literature on twin growth is associated with twin thickening, rather than with twin tip propagation. One common model for the process of lateral twin growth
is that thickening occurs through twinning dislocations propagating along the \{10\bar{1}2\} plane with steps having a magnitude of \(2d_{10\bar{1}2}\). These mobile twinning dislocations would have to be generated in sufficient quantities (one for every \(2d\) of advance of the interface) to accommodate the twin growth rate. Other twinning mechanisms (e.g. through shuffling) have been discussed, and there remains some controversy in the literature. All such growth models indicate that twin tip velocity and twin lateral thickening rates are correlated, effectively determined by the velocity of the dislocations along the twin plane. Very recent experimental evidence suggests that lateral twin growth indeed occurs through twinning dislocations propagating along the \{10\bar{1}2\} plane in the AZ31 magnesium alloy.

The twin nucleation and growth processes are hence intimately tied to the evolving dislocation substructure (significant plastic slip occurs at these stresses). One aspect of this is that the twin tip growth speed must correlate to the speed of twinning dislocations (the tip is composed of a propagating ensemble of such dislocations). A growth model for \{10\bar{1}2\} twinning is illustrated schematically in Figure 7. Lattice basal dislocations interact with the twin boundary (TB) as in Figure 7a, and each basal dislocation dissociates into a prismatic-basal (PB) facet and two sessile TDs (Figure 7b and c). The twin boundary thus is expected to contain TB-PB-BP-TB facets. Figure 7c shows a schematic of the emission of TDs (shown in blue) that move along the twin boundary. In this view, what we report as the twin tip velocity (figure 4c) is directly related to the twin dislocation velocity.

5. Conclusions

In summary, we have provided the first study of twin activity as a function of strain rate in single crystal magnesium. The primary conclusions in this study are,

1. Twin evolution and crystallography is found to be sensitive to loading rate.
2. The number of twin variants activated are found to be higher at the dynamic rates (five extension twin variants) in comparison to quasi-static rates (two extension twin variants).

3. Additional twin variants found at high rates are found to correlate to the nucleation of Gen2 primary twins from Gen1 twin boundaries, observed using in situ high speed imaging.

4. Low Schmid factor variants are observed at the high rates one of them being an extension twin within a primary extension twin (or a secondary twin). The low Schmid factor variants are expected to occur due to local stress concentrations and limited time available for twin growth at high strain rates.

5. Twin tip propagation velocity was measured at $\sim 1000$ m/s for first generation twins and of the order $\sim 100$ m/s for second generation twins. The rates of area fraction evolution of these two types of twins are however similar.
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![Figure 1: Schematic of twinning plane (K₁), twin conjugate plane (K₂) and its shear direction (s).](image)

Figure 1: Schematic of twinning plane (K₁), twin conjugate plane (K₂) and its shear direction (s).
Figure 2: Stress-strain response of a Mg single crystal deformed along the a-axis at a stain-rate of 1400 s\(^{-1}\) and 4.5 \times 10^{-4} s\(^{-1}\).
Figure 3: **Characterization of extension twins that form on \( \{10\bar{1}2\} \) crystallographic planes under quasi-static loading rates.** (a) An EBSD color map showing that two extension twin variants are detected and denoted as T1 and T2. The misorientation between these two variants is \( 7.3^\circ \). The twin morphology is considerably different than in the high strain rate regime (see Figure 5b). (b) Representations of the hexagonal close-packed unit cell orientations and pole figure of observed twin variants.
Figure 4: **Extension twin evolution and twin tip velocities measured on \{10\bar{1}2\} planes during dynamic loading.** a) Schematic of a cuboidal magnesium single crystal loaded dynamically in compression along the (11\bar{2}0) a-axis b) five (5) snapshots of the (10\bar{1}0) crystallographic plane during deformation captured at 5,000,000 frames per second. The white scale-bar in the first snapshot corresponds to a length of 500 µm which is standard for all non-zoomed in snapshots. c) Twin tip velocity as a function of time. d) Twin area fraction as a function of time. Twin area fractions are calculated from the imaging data using image processing techniques. The points marked by arrows a and b indicate points at which measurable rise in the area fractions of the 1\textsuperscript{st} gen. and 2\textsuperscript{nd} gen twins are detected. Note: The time values on the x-axis are displayed with respect to the time at which the elastic stress pulse reaches the specimen during the Kolsky bar experiment.
Figure 5: **Characterization of extension twins that form on the \{10\bar{1}2\} crystallographic planes under high strain-rate loading.** a) The region of the specimen where EBSD characterization was conducted. Note that this region was one where multiple twins nucleate and interact. b) An EBSD color map of the captured five extension twin variants, denoted T1 through T5. The Schmid factor (SF) analysis presented in the appendix demonstrates the presence of twins with both high SF and zero SF. c) Representations of the hexagonal close-packed unit cell orientations and (0001) pole figure of observed twin variants.
Figure 6: **Twin traces, calculated twin orientations and Schmid factor analysis at high strain-rate** a) twin traces with respect to the loading direction. b) calculated twin orientations c) Schmid factor calculation at high strain-rate for the observed twin variants. Note that 5 twin variants were detected, 4 with high Schmid factor (0.37) and 1 with zero Schmid factor. Observation of twins with zero Schmid factor (T2 in Figure 2b) and Low Schmid factor (T5 in Figure 2b) could be explained by shear transfer between two intersection twins at this early stage of macroscopic strain amplitudes. Note that the Schmid factor for T5 variants was calculated using the EBSD map and is 0.124.
Figure 7: **Growth model for \{101\} twinning** a) interaction of the basal dislocation to the TB. b) twin boundary transforms to TB-PB-BP-TB facets. c) TD’s glides along the twin boundary. d) High-resolution TEM micrographs showing TB-PB-BP-TB interfaces in \{101\} twin boundary [42]. e) Atomic structures of a \{101\} twin plane. Dashed lines represent TB. large blue spheres represent crystalline sites and smaller solid spheres represent the rumpled atoms on \{101\}. A TD is identified, with a Burgers vector $b_{twin} = b_{prismatic} + b_{basal}$. Magnitudes of the TDs Burgers vectors are presented in the main text.
Figure 8: Three dimensional representations of the four types of secondary extension twins that can form from a primary extension twin, adapted from [60]. a) type one secondary twin with a misorientation angle of 0°, b) type two secondary twin with a misorientation of 7.37°, c) type three secondary twin with a misorientation of 60.41°, and d) type four secondary twin with a misorientation of 59.85°. Matrix orientation (M) is shown in blue, the primary extension twin is shown in green. Secondary twins are shown as gray if they were not observed in EBSD maps. The lone orange secondary twin was observed in the high strain-rate deformation case, see Figure 2b.