Strain rate and temperature effects on the selection of primary and secondary slip and twinning systems in HCP Zr

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Abstract

We investigate the temperature and rate dependence of slip, twinning, and secondary twinning in high-purity hexagonal close packed α -Zr over a wide range of temperatures and strain rates (from 76 K to 673 K and 0.001 s^{-1} to 4500 s^{-1}). To reliably identify the dominant deformation mechanisms for each condition, we employ electron-backscattered diffraction (EBSD), dislocation theory, multi-scale polycrystal constitutive modeling, and a thermally activated dislocation density evolution based hardening law. We demonstrate with direct comparison with measurement that the constitutive model, with a single set of intrinsic material parameters, can predict the underlying texture evolution, primary and secondary slip and twin activity, and twin volume fraction associated with the different loading orientations and applied temperatures and strain rates. We find that the $\{10\bar{1}2\}\{10\bar{1}1\}$ twin is the preferred tension twin, either as a primary or secondary twin depending on the sample orientation, over the broad temperature and strain rate range tested. In contrast, we show that the preferred contraction twin, whether $\{11\bar{2}2\}\{11\bar{2}3\}$ or $\{10\bar{1}1\}\{10\bar{1}2\}$, is sensitive to temperature but insensitive to strain rate and whether it is a primary or secondary twin. Based on the concomitant changes in the dominant slip mode predicted by the model and revealed by the texture development, we rationalize that the temperature-induced transition in contraction twinning is due to the increased predominance of basal $\langle a \rangle$ slip at high temperatures ($> 673\text{K}$). Last, our analysis implies that all twin modes studied are rate insensitive and so the strong influence of strain rate and temperature on twinning is due to the rate-sensitivity of slip.

Keywords: Hexagonal close packed; Zirconium; Twinning; Dislocations; Crystal plasticity

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1.0 Introduction

In hexagonal close packed (HCP) metals, deformation twinning is an important deformation mechanism that governs texture evolution, hardening, and failure for many metals and their alloys and under various loading conditions [1, 2]. Within a given grain in a polycrystalline metal, twinning typically acts in combination with plastic slip to accommodate deformation. Twins abruptly reorient the crystal lattice of the parent grain. The reoriented twin domain introduces a twin boundary, which interacts with subsequent slip, and, within the reoriented twin domains, secondary slip and/or secondary twinning can occur with continued straining. Taken together, these two basic effects of twinning can significantly contribute to material strain hardening in at least three ways: (i) the hardening/softening due to crystal reorientation, from geometrically softer to harder orientations and vice versa [3-6], (ii) the Hall-Petch-like hardening due to grain subdivision [4, 7-9], and (iii) the Basinski-type hardening mechanism due to transmutations of dislocations from glissile to sessile [10]. Thus, understanding and predicting the mechanical behavior and microstructure evolution of HCP metals require appropriate models for twinning and de-twinning within polycrystal constitutive models.

To this end, four approaches currently exist in the literature for treatment of reoriented deformation twin domains within polycrystal plasticity codes: (i) the predominant twin reorientation (PTR) method [11, 12], (ii) the volume fraction transfer (VFT) scheme [12], (iii) the total Lagrangian approach [13, 14], and (iv) the composite-grain method [7]. Mean-field crystal plasticity models, such as Taylor-type [15-22] and self-consistent [23-25] schemes, are usually used as platforms in which these schemes are incorporated. Together, they provide a way to model the effects of deformation twinning on constitutive response [23, 26-30]. They typically allow for one twin type to be treated per grain. Separate more phenomenological schemes are then introduced for representing the effects of these twin domains on the three mechanisms (i), (ii), and (iii) listed above.

Despite these advances, achieving a better understanding the nature of twinning in HCP requires more refined twinning models in combination with physically based models for dislocation slip. Unlike FCC or BCC metals, multiple twins are frequently observed in HCP metals, which depend on the distribution of crystallographic orientations and loading conditions. Specifically, more than one twin mode can form in a grain or one twin type can form within another twin type (secondary or internal twinning). A good example of an HCP metal that exhibits

multiple twinning modes as well as slip modes is Zr. Four deformation twinning modes have been found in α -Zr grains including the c-axis extension twins $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ and $\{11\bar{2}1\}\{10\bar{2}\bar{6}\}$ and the c-axis contraction twins $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ and $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$. In many instances, the former two tensile twins can exist in the same grain [31]. These twins re-reorient the Zr crystal lattice by 85.22° about the $\langle 11\bar{2}0 \rangle$ direction, 34.84° about the $\langle 10\bar{1}0 \rangle$ direction, 64.22° about the $\langle 10\bar{1}0 \rangle$ direction, and 57.05° about the $\langle 11\bar{2}0 \rangle$ direction, respectively [32, 33]. Because of these large reorientations, which twin modes are selected and the twin volume fractions have a profound influence on texture evolution and macroscopic strain hardening. In addition, primary twins can favorably reorient a crystal for secondary twinning. Secondary twinning can significantly alter the deformation and failure response of Zr [31].

In addition to twinning, α -Zr is also known to generally deform by more than one slip mode. Through polycrystal and single crystal testing, prismatic $\{\bar{1}100\}\{\bar{1}\bar{1}20\}$ slip has been established as the easiest glide mode in Zr [34, 35]. This has been attributed to its c/a ratio of 1.594, which is lower than the ideal c/a ratio of 1.633. Pyramidal $\{10\bar{1}1\}\{\bar{1}\bar{1}23\}$ slip systems are harder than prismatic slip systems, but offer the additional degrees of freedom necessary to accommodate arbitrary plastic strains [36]. Basal $\{0001\}\{\bar{1}\bar{1}20\}$ slip also operates in Zr but requires higher activation stresses than the prismatic systems [37]. Moreover, these slip modes are highly rate sensitive and each has a different dependency on temperature and strain rate. For instance, pyramidal slip is much more rate sensitive than prismatic slip and basal slip is the most rate sensitive [35-38].

Like slip, temperature and strain rate are also known to affect deformation twinning in HCP polycrystals. However, the reasons underlying this apparent rate sensitivity of twinning are not as well understood as for slip. First of all, twinning often acts in combination with slip, and slip alone, as mentioned, is highly rate sensitive. Second, most of the foregoing studies on Zr (and other HCP metals) have analyzed slip and twinning within narrow windows of temperature or strain rates. Last, interpretation of relative contributions of various deformation modes is usually done based on the models that do not simultaneously consider the pronounced changes in texture and stress-strain evolution, and the underlying effects of strain rate and temperature on activation and propagation of individual deformation mechanisms.

In this work, we aim to understand the influence of strain rate and temperature on the activation of primary and secondary twinning modes in α -Zr. In particular, we seek to uncover

the role that dislocation slip and its rate sensitivity play on the types of primary and secondary twins activated. We use a comprehensive set of experimental data collected in the temperature range of 76 – 673K and strain rate range of 0.001 – 4500 s⁻¹ using standard and Split-Hopkinson Pressure Bar mechanical testing, electron backscattered diffraction (EBSD) and X-ray diffraction. The variety of testing conditions as well as the large strain deformation levels achieved (> 30% strain) allowed us to examine rate dependencies of three slip modes: prismatic $\langle a \rangle$ slip (easiest), pyramidal $\langle c+a \rangle$ slip (harder), and basal $\langle a \rangle$ slip (hardest) and three twinning modes. For inferring the underlying deformation mechanisms, we employ the visco-plastic self-consistent (VPSC) model with a strain rate and temperature hardening law based on the explicit evolution of dislocation density to the material under investigation and lattice reorientation by primary and secondary twinning. The VPSC model is shown capable of reproducing both the stress-strain response and microstructure evolution for all test conditions using a single set of material parameters. The model predictions are used to determine the underlying deformation mechanisms and their effect on mechanical response and texture evolution in α -Zr. We find that over the wide temperature and strain rate range tested the preferred tension twin is $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twinning whether it is a primary or secondary twin. Also, as reported in prior single crystal experiments, we observe that the preferred compression twin changes from $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ to $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ as temperature increases. We additionally show that this transition is not sensitive to strain rate and whether the contraction twin is a primary or secondary twin. Based on the dominant mechanisms revealed in modeling and texture evolution, we rationalize the temperature-induced transition in contraction twinning is due to the increased activity of basal $\langle a \rangle$ slip at high temperatures (> 673K).

2.0 Material and experimental methods

2.1 Material

The material studied here is a cold-rolled and fully recrystallized plate of Zr with the chemical composition expressed in weight parts per million (Wt. Ppm) of: O < 50, C < 22, N < 20, Fe < 50, Al < 20, V < 50, Hf 35, Ti < 25 and Zr balance. The texture and microstructure of the initial material were examined as part of an earlier study [39]. Electron backscattered diffraction (EBSD) and optical micrographs revealed a nearly equi-axed and twin-free grain structure with a mean grain size of 25 μm (Fig. 1a).

The initial crystallographic texture was measured by both X-ray diffraction (XRD) [39, 40] and EBSD over a large area and both measurements show that most of the basal poles are oriented within 20° from the normal direction of the rolled sheet (the so-called strong basal texture). Since EBSD measures micro-texture and XRD measures macro-texture, we calculated the orientation distribution function (ODF) from the XRD data (using popLA software) and used it to generate the discrete texture file needed for the input initial texture for the crystal plasticity simulations reported here. The pole figures of the measured and converted textures are compared in Fig. 1b. Plotting was done in the MTEX software package [41]. As shown, they are very similar except the maximum intensity of the measured one is slightly higher. Evidently, the conversion from the measured texture into the polycrystal data file caused the loss of intensity from ~ 5.2 to 4.8.

2.2 Mechanical testing

The mechanical response of the Zr plate was measured in compression and tension under quasi-static conditions and in compression under high-strain rate conditions. Compression samples were machined from the plate as cylinders 5 mm in diameter by 5 mm in length. The axis of the specimens was either aligned with the in-plane (IP samples) or through-thickness (TT samples) direction of the rolled Zr sheet. Tensile samples were machined as dogbone specimens with a 3.2 mm square cross-section. In what follows, these samples will be labeled as IPC (in-plane compression), IPT (in-plane-tension), and TTC (through-thickness compression). Table 1 lists the testing conditions for each test. Constant velocity mechanical tests were performed using a screw-driven Instron. To reduce frictional effects during loading of the compression samples, we used polished tungsten carbide plates lubricated with molybdenum disulfide grease. The raw load-displacement data were collected and corrected for machine compliance before computing the true stress-true strain curves. Dynamic compression tests were performed using a Split-Hopkinson Pressure Bar [42, 43]. The inherent oscillations in the dynamic stress-strain curves make the determination of the yield stress very approximate at low strains.

2.3 EBSD characterization

EBSD maps were measured in an FEI XL30 SEM equipped with TSL/EDAX data acquisition software. Following deformation, the samples were cut vertically in half with the section plane containing the loading direction. The specimens were prepared by grinding to an 800 grit SiC finish followed by chemically polishing with a solution of the ratio

45H₂O:45HNO₃:10HF. Scans were taken at 20 kV with a 0.2 μm step size in a hexagonal grid. Twin statistics were analyzed using the OIMTM Analysis software and a twin program written at Los Alamos National Laboratory [31, 44].

3.0 Multi-scale modeling approach

In this section, we review the multi-scale modeling approach we employ to distill the individual dependencies of dislocation evolution, twin selection, and twin growth on strain rate and temperature. At the largest scale, we model the constitutive response of the polycrystal and its relationship to the deformation of the individual constituent crystals via the viscoplastic self-consistent (VPSC) scheme. At the grain scale, we use crystal plasticity for slip and a composite grain model for the lattice reorientation induced by the primary twins. At the subgrain scale, we introduce a thermally activated dislocation density law for the evolution of slip resistance for the individual slip systems as a function of temperature and strain rate. The barriers for twin activation and propagation within each crystal follow a different model than slip and this twinning model will be reviewed last. In several prior studies, this same basic modeling framework has been successfully applied to a variety of metals of different crystal structures: Zr [45, 46], Be [47, 48], and Mg [49, 50], BCC Ta [51-53], an FCC cobalt alloy [54], and orthorhombic uranium [55, 56]. However, to meet the objectives of this work, we found it necessary to extend the framework to treat multiple modes of twins per crystal, interactions between slip modes, and lattice reorientation by secondary (internal) twinning. These additions will be highlighted in what follows.

3.1 Visco-plastic self-consistent (VPSC) model

In VPSC, a polycrystal is represented by a set of crystals or model grains (each having an orientation, shape and a volume fraction). Each grain is treated as a visco-plastic inclusion embedded in a homogeneous effective medium. The elementary inclusion problem is solved using a Green's function approach [23]. Enforcing the macro-homogeneity condition of self-consistency between the macroscopic stress and grain average stresses allows for calculation of the viscoplastic properties of the effective medium. The effective medium response corresponds to that of the polycrystal and is to be compared with that of the tested sample.

Plasticity in each grain is carried via the activation of slip and twinning. The corresponding slip or twin shearing rate, $\dot{\gamma}^s$, on a given system, s , is modeled using the power-law relation:

$$\dot{\gamma}^s = \dot{\gamma}_0 \left| \frac{\tau^s}{\tau_c^s} \right|^{\frac{1}{m}} \text{sign}(\tau^s). \quad (1)$$

Here, $\dot{\gamma}_0$ is the reference slip rate set to be the macroscopic strain rate, m is the rate sensitivity parameter set to be 0.05, τ^s is the resolved shear stress, and τ_c^s is the threshold stress on the slip system. The twin propagation is treated as a pseudo-slip mechanism, as originally proposed in [11].

According to Eq. (1), the crystal plasticity constitutive law requires a threshold resistance to activate each slip mode and each twinning mode. In this modeling framework, these activation stresses can evolve with strain separately for each system within each grain in the polycrystal. Twin activation is modeled separately from slip and the expansion of twin domains may be indirectly affected by the stored dislocation density. The models related to slip are described next and those related to twinning as well as to the interactions between twins and slip dislocations are described in the next section.

3.2 Dislocation density hardening law (DD model)

For dislocation slip, resistances change as dislocations are stored in the crystal. The dislocation density for each slip system s in each slip mode α evolves following its individual law and possesses its own dependencies on strain rate and temperature. We adopt a thermally activated dislocation density based hardening law (DD model) that was developed previously [50, 57] and therefore, is only briefly reviewed below since the physically based parameters that the law uses (athermal stress, activation barriers, drag stress, etc.) are important for interpreting our experimental results. In the description that follows, the superscripts s and s' span the slip systems and the Greek superscripts α, β represent modes to which s and s' belong.

The resistance to slip τ_c^s includes a friction stress τ_o^α (dependent on the Peierls stress and the initial content of dislocations), a barrier term $\tau_{o,HP}^\alpha$ (dependent on initial grain size) and two terms that evolve with strain, i.e., the forest and debris interaction stresses τ_{for}^s and τ_{deb}^α (dependent on a spatially random and ordered distribution of stored dislocations, respectively) [45, 58]:

$$\tau_c^s = \tau_o^\alpha + \tau_{o,HP}^\alpha + \tau_{for}^s + \tau_{deb}^\alpha. \quad (2)$$

In this work, we are concerned with strain rate and temperature sensitive deformation. The initial slip resistances $\tau_o^\alpha(\dot{\epsilon}, T)$ per slip mode are found to decay exponentially with temperature and increase logarithmically with strain rate as:

$$\tau_o^\alpha(\dot{\epsilon}, T) = A^\alpha (1 + B^\alpha \log(\dot{\epsilon})) \exp\left(-\frac{T}{C^\alpha}\right) (1 + D^\alpha \text{lognormal}(\sigma^\alpha, \nu^\alpha)), \quad (3)$$

where A^α , B^α , and C^α are constants and T and $\dot{\epsilon}$ are the current temperature and strain rate, respectively. Such expressions were used for several other metals in earlier studies [5, 45, 54, 56, 59]. The constant D^α and lognormal terms, σ^α and ν^α , are used to capture the saturation of the resistances at high temperatures seen experimentally. The grain size barrier term, $\tau_{o,HP}^\alpha$, is formulated through a Hall-Petch like equation:

$$\tau_{o,HP}^\alpha = \mu^\alpha(T) HP^\alpha \sqrt{\frac{b^\alpha}{d_g}} \quad (4)$$

where b^α is the magnitude of the Burgers vector, d_g is the initial grain size, $\mu^\alpha(T) = 40.06 - 0.022T$ is the effective shear modulus taken to be the same for all shear directions [60], and HP^α is the Hall-Petch parameter. When twins are present in a grain, the $\tau_{o,HP}^\alpha$ term is defined per slip system, s , as $\tau_{HP}^s = \frac{f^{pts} - f^{pts,0}}{f^{pts,max} - f^{pts,0}} \mu^\alpha(T) HP^\alpha \sqrt{\frac{b^\alpha}{d_{mfp}^s}}$ and d_g is replaced by the mean-free-path distance calculated using $d_{mfp}^s = \frac{(1 - f^{pts})d_c}{\sin(\alpha)}$ [7, 45]. The angle α is the angle between slip plane and the predominant twin system (PTS) plane and $d_c = \frac{d_g}{n^{lamellas}}$ is the separation between twin lamellae calculated as the ratio between the grain size, d_g , and a specified number of lamellae, $n^{lamellas}$, which is assigned a nominal value of 5. The term, $\frac{f^{pts} - f^{pts,0}}{f^{pts,max} - f^{pts,0}}$, represents the barrier factor and is based on the $f^{pts,0}$ and $f^{pts,max}$ constants, which represent the minimum volume fraction identifying the PTS and the maximum volume fraction that a twin can occupy within a given grain. The former $f^{pts,0}$ is taken to be 0.02 and the latter $f^{pts,max}$ is taken to be 0.9.

The evolution of $\tau_{for}^s(\dot{\epsilon}, T)$ and $\tau_{deb}^\alpha(\dot{\epsilon}, T)$ are governed by the evolution of the forest $\rho_{for}^s(\dot{\epsilon}, T)$ and debris $\rho_{deb}^\alpha(\dot{\epsilon}, T)$ dislocation densities, as described below. The effects are given by Taylor-type laws [61, 62]:

$$\tau_{for}^s(\dot{\epsilon}, T) = b^\alpha \mu^\alpha(T) \sqrt{\chi^{ss'} \rho_{for}^{s'}(\dot{\epsilon}, T)}, \quad (5a)$$

$$\tau_{deb}^\alpha(\dot{\epsilon}, T) = k_{deb} \mu^\alpha(T) b^\alpha \sqrt{\rho_{deb}^\alpha(\dot{\epsilon}, T)} \log\left(\frac{1}{b^\alpha \sqrt{\rho_{deb}^\alpha(\dot{\epsilon}, T)}}\right) \quad (5b)$$

where $\chi^{ss'}$ is a dislocation interaction matrix. Accurate characterization of this parameter would require specialized experiments and dislocation dynamics simulations, e.g., [63]. To eliminate any uncertainty that might be introduced by this parameter and to keep the number of fitting parameters minimal, we utilize only the diagonal terms ($s=s'$) and set them all to a typically used value of 0.81 [61]. However, in principle, it is important that the model is capable of accounting for dislocation-dislocation interactions via the off-diagonal components of the matrix.

The evolution of the stored forest density, $\rho_{for}^s(\dot{\epsilon}, T)$, is governed by competition between the rate of storage and the rate of dynamic recovery [61, 64]:

$$\frac{\partial \rho_{for}^s}{\partial \gamma^{s'}} = \frac{\partial \rho_{gen,for}^s}{\partial \gamma^{s'}} - \frac{\partial \rho_{rem,for}^s}{\partial \gamma^{s'}} = k_1^s \sqrt{\rho_{for}^s} - k_2^s(\dot{\epsilon}, T) \rho_{for}^s. \quad (6)$$

Here, k_1^s is a rate-insensitive coefficient for dislocation storage by statistical trapping of mobile dislocations and k_2^s is a rate-sensitive coefficient that accounts for dynamic recovery by thermally activated mechanisms. Following the derivation given in [45], we define the coefficient, k_2^s , to include not only an averaged interaction coefficient χ but also the anisotropic interactions through $\chi^{ss'}$:

$$\frac{k_1^s}{k_2^s} = \frac{1}{\sqrt{(\chi^{-1})^{s's} \left(\frac{\tau_{sat}^s}{b^\alpha \mu^\alpha} \right)^2}}, \quad \tau_{sat}^s = \frac{D^\alpha (b^\alpha)^3 g^\alpha \mu^\alpha}{D^\alpha (b^\alpha)^3 - k T \ln\left(\frac{\dot{\epsilon}}{\dot{\epsilon}_0}\right)}. \quad (7)$$

where k , $\dot{\epsilon}_0$, g^α , and D^α are the Boltzmann constant, a reference strain rate taken to be 10^7 s^{-1} , an effective activation enthalpy, and a drag stress, respectively. Dynamic recovery is often associated with thermal activation of dislocation cross-slip and climb, and the formation of

dislocation debris is concomitant with recovery processes. The rate of debris development is coupled to the rate of recovery of all active dislocations through:

$$d\rho_{deb} = \sum_s q^\alpha b^\alpha \frac{\partial \rho_{rem,for}^s}{\partial \gamma^{s'}} |d\gamma^{s'}| \quad (8)$$

where q^α as a rate coefficient defining a fraction of dislocations that leads to debris formation and the remaining fraction that annihilates.

3.3 Twin activation

Twin nucleation occurs at the atomic length scale where a unique strength for forming a twin nucleus can be potentially calculated [65]. At a larger scale, such as the one we treat here, it is difficult to experimentally characterize a unique critical resolved shear stress for twinning [57]. The local stresses and defect structures from which twins arise are statistically varying [66]. Therefore, although we provide model parameters associated with twin formation, we cannot state that these are singular values tied directly to twin nucleation at the atomic scale. Despite these limitations, however, these model parameters provide insight into the relative propensity of twinning among the three twin types and three slip modes considered for Zr and the individual sensitivities of twinning to strain rate and temperature.

In the description that follows, we use t to designate the twin variant and β the twin mode to which t belongs. The twin activation stress accounts for a temperature-independent friction term τ_0^β , a Hall-Petch-like term $\tau_{o,HP}^t$, and a latent hardening term that couples slip and twin systems:

$$\tau_c^t = \tau_o^\beta + \tau_{o,HP}^t + \tau_{slip}^t. \quad (9)$$

The terms are defined as follows:

$$\tau_o^\beta = \tau_{prop}^\beta + (\tau_{crit}^\beta - \tau_{prop}^\beta) \exp\left(-\frac{\sum \rho_{for}^s}{n^\alpha \rho_{sat}^s}\right), \quad (10a)$$

$$\tau_{o,HP}^t = \frac{HP^\beta}{\sqrt{d_g}}, \quad (10b)$$

and

$$\tau_{slip}^t = \mu^\beta(T) \sum_j C^{\beta\alpha}(\dot{\epsilon}) b^\beta b^\alpha \rho_{for}^s \quad (10c)$$

where τ_{crit}^β and τ_{prop}^β are the nucleation and propagation stresses, whose contribution to twin activation is leveraged by the probability term $\frac{\sum \rho_{for}^s}{n^\alpha \rho_{sat}^s}$ [45]. The probability term is defined by an average value of dislocation density on slip systems, which is reflected in the division by n^α , which is the number of slip systems in a given mode α . For $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ and $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twins, the dislocation density of prismatic slip systems enters the probability equation, whereas for $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twins, the dislocation density of basal slip systems enters into the probability equation. Constants in the interaction term are the elastic shear modulus on the system, μ^β , the Burgers vector of a given twin system, b^β , and the latent hardening matrix in function of strain rate, $C^{\beta\alpha}$. The six twin variants, t , belonging to the same twin mode, β , have their own activation stress and relationship to the dislocations stored within the crystal.

3.4 Primary twinning

Lattice re-orientation by twinning is modeled via the composite grain (CG) approach [7]. In this approach a twin system with the highest shear-rate among all active twin systems, i.e., the predominant twin system (PTS), is identified in a given grain. The selection criterion for the twin variant is not entirely deterministic. Specifically we use the following expression: $0.2f^t + 0.8 \cdot r \cdot f^{pts,0} \geq f^{pts,0}$, where r is sampled from uniform distribution and ranges between 0 and 1, f^t is the twin volume fraction of the t^{th} twin system and $f^{pts,0}$ is the threshold defined earlier. The first twin variant that satisfies this condition is selected and is termed the “predominant twin system. The grain is then partitioned into stacks of flat ellipsoids having the crystallographic orientation of the predominant twin and the matrix grain. The short axis of the parent and twin ellipsoids is perpendicular to the twin plane. Twin growth is modeled by volume fraction transfer from the parent grain to the twin domain. As the ellipsoids representing the twins thicken, the ones representing the parent shrink.

We note that the PTS criterion is a simplifying assumption because it neglects many of the factors that are believed to contribute to twin variant selection. Several experimental studies have shown that a non-negligible fraction of the twins do not correspond to the variant with the highest Schmid factor. Several reasons have been proposed, such as those related to grain-grain interactions, local grain boundary defects, parent grain properties, neighboring grain properties, slip-twin and twin-twin transmission across grain boundaries, and stress fluctuations at grain

boundaries [67-71]. Experiments and numerical simulations in [67, 69] have studied the role that strain accommodation at grain boundaries play. Other studies have examined the relation between twin variant selection and the backstresses [70] or the work of accommodation due to reaction of the twinning shear induced in a neighboring grain [71]. Generally, accounting for such effects in twin formation within constitutive modeling require supplemental or integrated calculations of the local strains and stresses at and in the vicinity of the grain boundaries and the response of the neighboring grains. Such calculations are possible with full-field models based on finite elements [17, 72] or Green's functions [73] that consider explicitly the grain structure of the metal [74] but not the mean-field formulation adopted here. Nevertheless, to first order, prior work using mean-field models have demonstrated that assuming that the twins associated with the highest Schmid factor variant can lead to reasonable predictions for twin volume fraction and texture for many low-symmetry metals including Zr [7, 23, 45, 46], Be [47, 48], Mg [49, 50, 75], Ti [6] and U [55, 56]. This success likely results because the thicker twins usually correspond to twins with the higher Schmid factor [68, 76].

3.5 Secondary twinning

As a necessary new feature, we introduce twin reorientation by secondary twinning. In the model we allow secondary twins to nucleate when a primary twin fraction in a given grain reaches a fraction of 0.5. Nucleation and propagation of secondary twins is modeled in a similar manner as that of the primary twins, with the difference being that the role of the parent grain is replaced by a primary twin.

In the next section we calibrate and critically validate the model using a comprehensive set of rate and temperature sensitive experimental data for Zr.

4.0 Material parameter characterization

To have a model capable of accurately describing the mechanical behavior and microstructure evolution over a broad range of temperatures, strain rates, and loading conditions requires making available the following deformation mechanisms: prismatic $\langle a \rangle$ slip, first-order pyramidal $\langle c+a \rangle$ slip, basal $\langle a \rangle$ slip, $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twinning, $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twinning, and $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning. The slip and twin activation models we employ introduce a number of physically based material parameters. Although we can build on parameters characterized in prior work, it is necessary to extend the characterization effort to ensure that the parameters apply at much higher temperatures and strain rates. Our approach is to obtain a single set of

material parameters by only fitting the calculated stress-strain curves with the measured ones for all mechanical tests in this study. Other data reported here (texture, twin statistics, variant selection) are used for validation.

We should mention briefly, that for high-purity Zr, some of these parameters have been characterized previously. They have been obtained for prismatic slip and pyramidal slip at low strain rates for low to moderate strain levels ($< 30\%$), where basal slip does not contribute significantly [57] and additionally for basal slip for large strains ($> 30\%$), where the basal-slip contribution becomes non-negligible [46]. The parameters for $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ and $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twinning have also been characterized previously for Zr at relatively low temperatures (76 K to 400 K) at quasi-static strain rates [45, 66]. However those for $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning are not available and hence are newly determined here. In the modeling, we also permit de-twinning to occur [48, 77], but under the monotonic loading conditions studied here, we find that not surprisingly it was not activated.

The starting microstructure of the model polycrystal was constructed using the measured initial texture (Fig. 1b), represented by 2944 distinct orientations (grains) each with a corresponding volume fraction. Consistent with the initial grain structure seen in Fig. 1, we assigned the initial grain shape to be equi-axed and its diameter d_g to be 25 μm . In the simulations, we impose homogeneous boundary conditions corresponding to uniaxial compression or tension in the prescribed load direction (with respect to the initial texture) and traction-free surfaces in the other two orthogonal directions.

Figure 2 compares the measured and calculated stress-strain curves. We note that the calculations achieve agreement in both the flow stresses and hardening rates for the entire suite of tests. Table 2 lists the single set of material parameters corresponding to these calculations. In particular, Figure 3 compares the variation in the initial activation stresses, τ_0^α , with temperature for each slip mode with experimental data on single crystals found in the literature [35-37, 78]. Other relevant model outputs, such as slip and twin activity as a function of strain, strain rate, and temperature; texture evolution; and twin volume fraction, were not used for parameter characterization, and thus, are model predictions to be analyzed.

5.0 Results

5.1 Influence of strain rate and temperature on tensile twin selection

Of the seven observed twin types in HCP metals [1, 2, 79], three are tensile (or extension) twins in Zr. Tensile twins are thus named because they accommodate a tensile strain along the c-axis. Accordingly tensile twins can form under tensile loading parallel to the c-axis. Compression perpendicular to the c-axis can also result in a positive resolved shear stress on many of the tensile twin variants, and hence tensile twinning is often observed for compressive loading perpendicular to the c-axis. In the present material, due to the strong initial texture (Fig. 1b), in-plane compression (IPC) is favorable for tensile twinning. Prior work carried out on Zr of similar composition and initial texture after IPC found that the preferred twin type is the $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twin for a temperature range of 76 K to 298 K and quasi-static strain rates [31, 77]. For this material, Figure 4 shows the EBSD maps after compressive deformation at a) 76 K and b) 298 K at 0.001 s^{-1} . In agreement, the $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twin is the preferred tensile twin. A much smaller fraction of the $\{10\bar{2}1\}\{11\bar{2}\bar{6}\}$ twins is also observed at 76 K. The preferred tensile twin type at higher temperatures, however, is not known since, under commonly used quasi-static testing conditions, slip dominates over twinning as temperature increases. To explore the selection of tensile twin type in HCP Zr as a function of temperature, we simultaneously apply high strain rates to promote twinning at high temperatures and use the model to separate the effects of strain rate and temperature.

Figure 4c shows the microstructure after room temperature compression deformation at high rate (3500 s^{-1}). Prior to deformation, the grains had orientations such that their $\langle 11\bar{2}0 \rangle$ and $\langle 10\bar{1}0 \rangle$ (green to blue), directions were nearly aligned with the compression direction. After deformation, the microstructure has been almost completely transformed to near $[0001]$ orientations by $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twins. Next, we varied the test temperature from 298 K to 673 K while keeping the strain rate high (4500 s^{-1}). Figure 4d shows the EBSD maps of the deformed sample at 673 K. Again, $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twins have nearly completely transformed the orientations of the grains. Considering all tests performed, the preferred tensile twin is the $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twin for a broad range of temperature (76 K to 673 K) and rates (10^{-3} s^{-1} to 10^3 s^{-1}). We do not see a temperature- or strain rate-induced transition in the type of tension twin that prevails. Shortly we will discuss tests to determine whether this preference persists when $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twinning serves as a secondary twin.

Figure 5 and 6 compare measured and predicted texture evolution. It can be seen that texture evolution is predicted well. In particular, the texture reorientations in IPC after large strains and

under high strain rates due to profuse twinning are also effectively captured (Fig. 5 c and d). Under these conditions, the basal $\{0001\}$ poles have almost completely reoriented toward the compression direction.

5.2 Deformation within $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ twins

The presence of twins in the grain can cause a change in the deformation mechanisms. The twins introduce twin boundaries, which can serve as barriers to certain slip systems. The twins also form reoriented domains that can promote different slip or twin modes within the twin than were active in the original, untwinned grain (matrix) under the same imposed stress state. Particularly, within $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ twins, the c-axis reorients by 85.22° and aligns close to the imposed compression axis (Fig. 5c and d). Consequently, to accommodate compression, the twin domains can deform by slip and/or contraction twinning. To examine the temperature- and strain-rate dependence of the deformation mechanisms within the $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ twins, we examined the large-strain behavior under two conditions where profuse primary $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ twinning occurs: a) in-plane compression at cold temperature (76K) and quasi-static strain rates and b) in-plane compression at high temperature and high strain rates ($> 10^3 \text{ s}^{-1}$).

For the first case a), Fig. 4a shows the microstructure after large strain at 0.001 s^{-1} and 76K. By a strain of 0.08, secondary $\{11\bar{2}2\}\langle 11\bar{2}\bar{3}\rangle$ twins form within the primary twins. This result is similar to that reported previously [31]. With the present model, we predict that the $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ twin domains deform primarily by $\{11\bar{2}2\}\langle 11\bar{2}\bar{3}\rangle$ twinning at 76K (see Fig. 7b). Both microstructural characterization and the model find that the volume fraction of secondary $\{11\bar{2}2\}\langle 11\bar{2}\bar{3}\rangle$ twinning reaches around 3% at a strain of 0.18 and increases with further straining (see Fig. 9).

For the second case b), Fig. 4d shows the microstructure after the same deformation mode, in plane compression (IPC), but at a higher rate and higher temperature (673K and 4500 s^{-1}). Here we find that the $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ twins deform by different mechanisms than at colder temperatures and slower rates. We observe that secondary twinning occurs via another HCP contraction twin, the $\{10\bar{1}1\}\langle 10\bar{1}\bar{2}\rangle$ twin, and not by the $\{11\bar{2}2\}\langle 11\bar{2}\bar{3}\rangle$ twin. EBSD analysis and the model find that the volume fraction of secondary $\{10\bar{1}1\}\langle 10\bar{1}\bar{2}\rangle$ twinning reaches approximately 7% by a strain of 0.44. Thus, it appears that the choice of compression twin type as a secondary twin within the primary $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ twins exhibits a rate sensitivity.

To determine whether the twin-type selection was caused by the increase in strain rate or in temperature, we tested the material in IPC at room temperature and high rate (3500 s^{-1}). Figure 4c shows the deformed microstructure from EBSD. As shown, the high rate was sufficient to activate profuse primary $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twinning. More importantly, secondary contraction twinning occurred via $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twinning. The volume fraction of secondary twinning was estimated to be 6% at a strain of 0.32.

Therefore, we see an interesting dependence of contraction twin type on temperature, not seen in the selection of the tensile twin type. With the same loading state, similar high-rate conditions, and the same primary $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twin type, it appears that the preferred contraction twin type changes from $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twinning at low temperatures (room temperature and lower) to $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning at high temperatures (673K).

This phenomenon has been reported in earlier work on single crystals of Ti and Zr, which were compressed along the c-axis at different temperatures and low strain rates [36, 80]. It was seen in Ti that below 673 K, primary $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twinning was favored and above it, primary $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning was favored [80]. In single crystal Zr, the transition occurs at 800 K [36]. The present results on polycrystalline Zr show that a similar transition occurs within primary twin domains as well and around 673 K (and possibly lower).

5.3 Temperature sensitive contraction twin selection

An understanding of twin selection among the various HCP twin types has yet to be developed [2]. For instance, it is not known why $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ contraction twins are preferred at lower temperatures and $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twins at higher temperatures in transition metals such as Ti and Zr and not in Mg. Many studies have speculated that the answers lie in how each twin nucleates. As mentioned earlier in the context of tensile twinning, some nucleation mechanisms proposed for deformation twinning involve special boundary defect structures or slip dislocation-boundary interactions [81, 82]. With this in mind, it could be surmised that the transition in contraction twin type reported here may be the result of interactions of impinging slip dislocations from either the parent matrix or twin domain with the primary $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twin boundary. This possibility leads us to the question: Is the observed transition in contraction twin type from $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twinning at low temperatures to $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning at high temperatures limited to secondary twinning? To test whether contraction twinning exhibits the

same temperature dependence as a primary twin, we carried out uniaxial compression tests through- thickness of the rolled sheet (TTC tests).

At a low temperature of 76 K, Figure 2d shows the stress-strain response for TTC at two rates, 0.001 s^{-1} and 2000 s^{-1} . Both exhibit similar responses characteristic of deformation dominated by twinning in that the hardening rate is constant or increases with strain. As evidence, figure 4g shows the EBSD micrograph at 76K and low rate (0.001 s^{-1}) after 0.08 strain, where the microstructure is populated by $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twins.

To activate twinning at higher temperatures, high-rate TTC was needed. Figure 2e shows the stress-strain curves at 298 K and at different rates: 0.001 s^{-1} , 0.1 s^{-1} , and 2700 s^{-1} . Only at the highest rate does the material exhibit a deformation response characteristic of $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twinning. Therefore, $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twinning is preferred at low temperatures and for high rates at room temperature.

To explore even higher temperatures and high rates, Fig. 2f compares the TTC response at 473 K at 3000 s^{-1} and 673 K at 3500 s^{-1} . The former curve does not suggest substantial amounts of twinning occurred, but the latter one does. We see in Figure 4h, which is the EBSD micrograph at 673 K at high rate (3500 s^{-1}) after 0.34 strain, that the deformed structure is dense with $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twins. Compared to the initial texture the deformation textures in Fig. 6b show an increased intensity in the basal poles near the TT direction due to basal slip activity, as will be discussed later. It is sufficient to counteract the effect of compressive twinning, which tends to diminish the intensity of the basal poles in the TT direction. In this case, the basal poles of the twinned domains reorient by $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning (57.05° away from the compression axis). The reorientation does not produce increased intensity near the perimeter of the basal pole figure for several reasons. First, the volume fraction of primary twins is relatively low. Second the twin orientations are distributed for 360 degrees about the perimeter and thus do not produce a large increase in intensity at any specific spot around the perimeter. Finally, there is also some reorientation due to $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ secondary twinning (85.22° back toward the compression axis).

These results indicate that contraction twinning in Zr, as a primary twin, has the same temperature dependence as it does as a secondary twin: $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ contraction twinning is the preferred twin mode at room temperature (and high rate) and below and $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ contraction twinning is preferred at the higher temperature, 673 K. Therefore, it cannot be

concluded that the temperature-induced transition in contraction twin type is solely related to the primary $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ twin domain or requires interactions only with the $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ twin boundary.

The domains of contraction twins are reoriented well for strain accommodation by tensile twinning. Characterization of both the $\{11\bar{2}2\}\langle 11\bar{2}3\rangle$ and $\{10\bar{1}1\}\langle 10\bar{1}2\rangle$ twinned structures in Fig. 7a and 7b, respectively, reveal $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ secondary twins. Thus, like $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ primary twinning, $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ secondary twinning also persists over a wide range of temperatures.

6.0 Discussion

6.1 Model interpretations on the influence of strain rate and temperature on the formation and deformation of twins

In this work, the model was used to interpret the mechanisms underlying the evolution of stress and texture during straining.

In all cases of temperature and strain rate, the IPC stress-strain curves exhibit typical signatures of twinning, where the work hardening rate is constant or increases with strain. The details in the shape of the deformation response, however, depend on the relative amounts of twinning and slip. HCP deformation textures are highly dependent on the relative contributions of slip and twinning as well as the type of twin and twin variants selected. Accordingly, texture can serve as an excellent signature of the active deformation mechanisms as well as a stringent test of our multi-scale model predictions. Figure 5 compares the calculated texture with the measurement for the IPC tests in Fig. 2. We can see that good agreement has been achieved.

From the model, we can extract predictions of the contributions of slip and twinning to deformation. Figure 8 shows the evolution of the relative activities of slip and twinning in the grains and in the twin domains corresponding to the IPC stress-strain responses. As a reference point, we begin with the case of ambient conditions (low rate and room temperature), where there is little twinning (Fig. 8a). The corresponding measured and predicted textures at a strain of 0.065 are provided in Fig. 5b. Both textures appear similar to the starting texture (Fig. 1b) due to the little reorientation due to twinning and predominance of prismatic slip.

At a lower temperature, 76 K, the model predicts an increasing evolution of twin volume fraction with IPC strain, which quickly becomes profuse (50% volume fraction) by a strain of 0.2 (Fig. 8b). We validate this prediction in Fig. 10, which compares the calculated and measured

strain evolution of the twin volume fraction. Part of the measured data points are taken from [31]. Likewise, at higher rates (3500 s^{-1}), the model indicates that twinning increases with IPC strain and eventually becomes profuse reaching 50% volume fraction by a strain of 0.14 (Fig. 8c). At room temperature and high strain rate (3500 s^{-1}) twinning is also profuse (Fig. 8d). We compare the calculated and measured textures at 0.32 strain in Fig. 5c. As shown, the texture has completely transformed by twinning and the basal poles have reoriented close to the compression direction.

For high strain rates and higher temperatures (473 K and 673 K), the model continues to predict increasing tensile twin fractions with strain (Fig. 8e and f). The textures corresponding to 673 K are shown in Fig. 5d and appear, to first order, similar to those in Fig. 5c; that is, a texture indicative of profuse $\{10\bar{1}2\}\{10\bar{1}1\}$ twinning.

In the TTC tests, twinning did not occur in every condition tested. Figure 6 compares the calculated and measured textures and Fig. 9 presents the predicted slip and twinning activities. As a reference, little to no twinning is observed at quasi-static rates and room temperature and c-axis compression deformation is accommodated by pyramidal slip. The model generally finds and, most importantly, quantifies that the relative amount of twinning relative to slip increases with lower temperatures (76K) and higher strain rates ($>3500 \text{ s}^{-1}$). We validate the 76 K prediction in Fig. 11, which compares the calculated and measured strain evolution of the twin volume fraction in the TT compression. Regardless of strain rate, for temperatures 473 K and below, the model predicts that $\{11\bar{2}2\}\{11\bar{2}3\}$ twinning is preferred over $\{10\bar{1}1\}\{10\bar{1}2\}$ twinning. It is not until 673 K that $\{10\bar{1}1\}\{10\bar{1}2\}$ twinning occurs.

6.2 Is twinning rate sensitive?

In testing Zr over a wide range of loading conditions, we observe that strain rate and temperature strongly influence the amount and type of twin mode selected. However, these results do not necessarily imply that contraction and extension twinning possess individual dependencies on strain rate and temperature. As we have shown, the material stress-strain responses and associated texture evolution arise either from slip or from a combination of slip and twinning. Slip alone, both the amount and type of slip mode activated, is known to be highly sensitive to strain rate and temperature [32, 45, 48, 79]. Thus, it is possible that the observed rate sensitivity in the macroscopic responses is a consequence of the rate sensitivities of the slip modes activated.

With the model, we can de-convolute the macroscopic response into the individual rate sensitivities of slip and twinning. We can readily achieve this by examining the material parameters characterized by the model (Table 3). As shown in Table 3, the characteristic activation stresses for all three twinning modes used in the model are rate independent. Thus, the strain rate and temperature dependencies of stress-strain, twin volume fraction, texture evolution, and even the transition in contraction twin type captured by the model can be explained entirely by the strain rate and temperature dependence of slip.

According to the model, the easiest to form (lowest τ_{crit}^β) and most mobile (lowest τ_{prop}^β) is the $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twin. This result is in complete agreement with expectation. Its low activation barrier can be attributed to the fact that this twin has the smallest Burgers vector and twin shear S . The topologies and kinetics of these twins have been studied using atomistic simulations [81, 83, 84]. With molecular dynamics (MD), it has been predicted that the $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twin is more glissile than the other two twins $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ and $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ [81, 85].

The model finds that the $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twin is the hardest to activate and the $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twin is the hardest to propagate (highest τ_{prop}^β). This trend can be linked to basic topological properties of the respective twinning dislocations for the twin types. The former has the highest twin shear and the twinning dislocation of the latter has the largest number of layers, with three layers as opposed to two [85, 86].

Establishing the rate sensitivity of twinning is directly relevant to understanding the effect of temperature on contraction twinning. Both earlier work on single crystals [36] and the present work on polycrystals find that contraction twinning in Zr exhibits a temperature-induced transition from $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twinning at lower temperatures and $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning at higher temperatures. To date, the reason for the transition has yet to be clarified. It was first proposed that twinning is rate sensitive, which would imply that the “CRSS” for $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twinning increased with temperature and $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning decreased with temperature [80], but this explanation assumes the ideal case in which slip dislocations were not active or contributing to deformation. Later, using a homogeneous twin nucleation model, it was argued that $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twinning is preferred over $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning when the twin-boundary energy for $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twins is within 10% of or greater than that for $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twins

and that transition arises because their relative boundary energies change with temperature [87]. However, both MD and first principles calculations find that the $\{10\bar{1}1\}\{10\bar{1}2\}$ twin boundary energy is substantially lower than the $\{11\bar{2}2\}\{11\bar{2}3\}$ twin boundary energy [88-91], with 74.3 mJ/m² compared to 123.8 mJ/m². Likewise, differences in the mobility of $\{10\bar{1}1\}\{10\bar{1}2\}$ and $\{11\bar{2}2\}\{11\bar{2}3\}$ twinning dislocations cannot provide an explanation [90]. Atomic-scale simulations show that the $\{10\bar{1}1\}\{10\bar{1}2\}$ twin is the more mobile twin [81]. Last, as we have just discussed, the present model calculations indicate that twinning is rate insensitive and the rate-insensitive activation parameters found for these three twin types are consistent with atomistic calculations.

Based on the above discussion, we hypothesize that the transition in contraction twinning with temperature is a consequence of slip. Slip assists in the formation of twins [1, 2, 67, 82, 92-94] and thus, when slip is altered by temperature, twinning would then be altered by temperature. To this end, we discuss next the rate sensitivity of slip suggested by the model before returning to the issue of the temperature-induced transition on contraction twinning.

6.3 Special twin and slip combinations

An interesting finding made by the model is the differences in the choice of slip mode activated between the high- and low-temperature conditions. Unlike twinning, results on slip are not apparent from EBSD microstructure characterization or readily obvious from texture analysis and thus the model plays an important role in uncovering the effects of rate on the relative contributions of the different slip systems.

In IPC, regardless of rate, the model indicates that tensile twinning is accompanied by prismatic slip at room and lower temperatures, and by a mix of prismatic and basal slip at higher temperatures (Fig. 8). Likewise, the twin domains deform predominantly by pyramidal slip and $\{11\bar{2}2\}\{11\bar{2}3\}$ (secondary) twinning at lower temperatures (including room temperature) and basal slip and $\{10\bar{1}1\}\{10\bar{1}2\}$ (secondary) twinning at higher temperatures. The former finding has been reported previously for polycrystalline Zr [31], but not the latter.

In TTC, a similar coupling of slip and twin systems is also seen. Figures 9 show the relative slip activities calculated by the model for the selected TTC tests. As temperature increases from 76 K to room temperature, the deformation mechanisms change from prismatic $\langle a \rangle$ slip and $\{11\bar{2}2\}\{11\bar{2}3\}$ twinning to prismatic $\langle a \rangle$ and pyramidal $\langle c+a \rangle$ slip (Fig. 8a). As temperature increases further to 473 K, prismatic $\langle a \rangle$ and pyramidal $\langle c+a \rangle$ slip still prevail, but basal $\langle a \rangle$

slip begins to contribute (Fig. 9e). At 673 K, basal $\langle a \rangle$ slip, some pyramidal $\langle c+a \rangle$ slip and $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning accommodate the deformation (Fig. 9f). Again, we see that at high temperatures, basal $\langle a \rangle$ slip and $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning occur concomitantly.

The paired twin-slip mechanisms at higher temperatures (basal slip and $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning) predicted by the model are supported by the good agreement achieved in the flow stress-strain curve for different test conditions (Fig. 2). Reinforcement for this prediction can also be found in subtle but important features of texture development. Consider the texture in Fig. 5d, measured at 673 K after high rate deformation to a strain of 0.44. With modeling we can correlate features of this texture with the occurrence of basal slip within both the matrix and twin domains of the twinned grains. Figure 12 shows textures resulting from simulations a) with basal slip suppressed, b) with basal slip only within the matrix, and c) basal slip only within the $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twin domains. By comparing a) and b), we see that allowing for basal slip to occur in the matrix stabilizes those twin domains that have reoriented the c -axis by $\sim 64^\circ$ from the TT compression direction, again consistent with the measurement. Comparing a) and c), we find that permitting basal slip to occur within the twinned domains reorients some crystals towards the IP1 axis as shown experimentally. Clearly allowing for basal slip to activate in both the matrix and twin as in the calculation of Fig. 5d best explains the texture development seen experimentally. It is another piece of evidence to support that basal slip and $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning are both associated with high temperatures, but not lower ones.

6.4 Basal slip at high temperatures

In this work, we report evidence of uncommonly large activity of basal slip in high rate, high temperature conditions. Analysis of flow stresses (Fig. 2) and details of texture development (Fig. 5d) together strongly indicate that basal slip prevails in both the grains and twin domains. Prior joint experimental and modeling studies have shown that basal slip in Zr is the hardest of the three common slip modes in HCP metals (prismatic, pyramidal, and basal) to activate. In low strain, ambient conditions, basal slip is not activated in single crystals [37], and typically occurs only at localized regions of the microstructure, such as near cracks [32, 37], near twins and kink bands [95], and free surfaces [37, 96-98]. Anomalously high contributions of basal slip in Zr have been reported in ambient conditions in large strain and severe plastic deformation, e.g., > 1.0 [46, 99], which are much higher than the strain levels achieved here. Recent *ab initio* calculations reveal that basal slip in Zr involves the thermally activated motion of a prismatic

stacking fault normal to the prismatic plane [100], implying that high temperatures would promote basal slip. Consistent with these prior works, the present model finds that the activation stresses for basal slip below 673 K are significantly higher than those for prismatic and pyramidal slip (see Table 2).

The reliability of our finding on the prevalence of basal slip at high temperatures depends on the model's ability to capture the temperature dependence of the other slip modes. Thus, we seek to validate our activation stresses used by the model with available experimental data. Most of the data use single crystals and test for the rate and temperature dependence of prismatic $\langle a \rangle$ slip, which the easiest and most prevalent slip systems in Zr [35-37, 78]. Figure 3 compares the model values for the initial activation stresses for prismatic slip with the CRSS values reported in these works. Both the measured CRSS and the model τ_0^α are intended to be a characteristic threshold stress that is separate from the effect of grain boundaries and dislocation-dislocation interactions. As shown, the measured CRSS for prismatic $\langle a \rangle$ slip drastically decreases as temperature increases and notably, the model τ_0^α varies with temperature in a similar way.

Measurements are not available for the full range of temperatures tested here for pyramidal $\langle c+a \rangle$ slip and basal $\langle a \rangle$ slip in Zr, and therefore, we include experimental data outside the range in Zr and within the range from other HCP metals. For pyramidal $\langle c+a \rangle$ slip, a strong temperature-dependent behavior in the CRSS has been reported for single crystals of Co and Be [101], which respectively have c/a ratios higher and lower than Zr. To compare with the model pyramidal $\langle c+a \rangle$ slip τ_0^α for Zr, we considered the Co measurements and reduced all the values consistently by 2.5 times. As Fig. 3b shows, the temperature dependence is captured well by the model values for the pyramidal $\langle c+a \rangle$ slip τ_0^α . (Co exhibits an anomalous increase in CRSS at high temperature from ~400 K to 600 K that we do not forecast for Zr.) For basal $\langle a \rangle$ slip, the model comparison is limited since basal slip activity was not detected in single crystals of Zr until above 800 K [37]. As seen in Fig. 3c, the model predicts a reduction in basal slip τ_0^α to values in agreement with the measurement as temperature increases. The favorable comparison made implies prevalence of basal slip in the high temperature-high rate observations can be explained by a significant reduction in the CRSS at high temperatures.

6.5 The role of slip in the $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ - $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twin transition

To date, the mechanisms underlying twin nucleation remain elusive. While it is possible to observe the formation of a twin in-situ, it is not possible to see the atomic scale details of the nucleation mechanism [102, 103]. Throughout the years dislocation theory and atomic-scale simulations have been used and many of the more recent efforts are confirming some of the earlier notions that twinning results from slip dislocations [81, 82, 92]. Mendelson proposed that twinning dislocations could be produced from non-planar dissociations of slip dislocations [86, 93]. Based on detailed TEM analyses, Vaidya and Mahajan proposed that dislocations within slip bands can create embryonic $\{10\bar{2}1\}\{10\bar{2}\bar{6}\}$ twins [94]. Yoo proposed several slip-twin boundary interactions that would support twin thickening [82]. In studying the events leading up to twin nucleation for different HCP twin types, MD simulations have supported many of these ideas [2, 65, 81, 104-106]. On this basis, the origin of the $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ - $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twin temperature-induced transition could be a direct consequence of the temperature dependence of slip. In all tests of loading direction and strain rate, a predominance of prismatic $\langle a \rangle$ slip occurs at low temperatures (below 673 K) when $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ the twin prevails and basal $\langle a \rangle$ slip at higher temperatures when the $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twin is preferred.

A possible link between prismatic slip and the $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twin and basal slip and the $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twin has been made in a prior modeling study, which investigated the mechanism of twin nucleation via non-planar dissociations of $\langle a \rangle$ slip dislocations from either the basal or prismatic planes onto seven possible twinning planes [67]. Using continuum dislocation theory, a simple analytical model was developed that considered the energy and stability of the extended core of the twinning dislocation after the dissociation to determine whether slip-assisted twin nucleation was feasible. It was found that a suitable size dislocation pile-up was necessary but not sufficient to form a stable twin fault by a non-planar dissociation of an $\langle a \rangle$ slip dislocation. The calculations revealed that elastic dislocation interactions were a key factor for twinning. Specifically when the interaction force between the slip and twinning dislocations is repulsive, a pile-up, or any suitable defect arrangement, can potentially lead to a twin formation, whereas with no such repulsive force, no value of twin fault energy or pile up size can produce a twin. For Zr, the calculations indicated that pile-ups of prismatic $\langle a \rangle$ dislocations can trigger a non-planar dissociation reaction of the lead prismatic dislocation that produces twinning dislocations for $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twins but not for $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twins. However pile-ups of basal $\langle a \rangle$ dislocations can result in the non-planar dissociation of the lead basal dislocation that produces

$\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning dislocations. On this basis, it appears the substantially enhanced basal slip activity at high temperatures may have promoted the formation of $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ contraction twins over $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twins. On this basis, one would expect that $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ compression twinning would be profuse in materials where basal slip is predominant such as in Mg alloys. However $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twins in Mg alloys tend to be very fine since they readily form internal $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twins (a double twin sequence) [3, 104, 107]. Consequently they appear in low volume fractions, and are hard to detect in texture measurements.

We note that although providing a plausible reason, this idea only considers twin formation via a non-planar dissociation mechanism. Other dissociation types as well as other slip-assisted twin nucleation mechanisms may operate as well and these may have different dependencies on temperature. For instance, it is possible that dissociations from $\langle c \rangle$ or $\langle c+a \rangle$ dislocations can produce twinning dislocations [86, 93, 104].

7.0 Conclusions

In this work, we use a combination of experiment and multi-scale modeling to study the influence of strain rate and temperature on the activation of primary and secondary twinning modes in HCP α -Zr. Numerous mechanical tests and subsequent characterization are carried out at different loading directions, strain rates, and temperatures. The model predictions achieve good agreement with measurements of twin-type selection and twin volume fraction with a single material parameter set. The model shows that the relative contributions of prismatic $\langle a \rangle$, pyramidal $\langle c+a \rangle$, and basal $\langle a \rangle$ slip and $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$, $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$, and $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning to strain accommodation depend strongly on strain rate and temperature. These results can notably be explained by rate-independent twin activation and rate-dependent slip. We find that the $\{10\bar{1}2\}\{10\bar{1}\bar{1}\}$ twin is favored over a wide range of temperatures (76 K to 673 K). In contrast, we show that the preferred contraction twin depends on temperature with $\{11\bar{2}2\}\{11\bar{2}\bar{3}\}$ twinning at lower temperatures and $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twinning at higher temperatures. Moreover, these preferences are found to be insensitive to whether the twin is a primary twin or secondary internal twin and thus are not a special consequence of interactions with a pre-existing twin boundary. We rationalize that the preferences are likely a result of the slip systems activated. Based on dislocation theory, it was found previously that dissociations of the lead dislocation in a basal dislocation pile-up, but not a prismatic dislocation pile up in Zr can result in twinning dislocations for $\{10\bar{1}1\}\{10\bar{1}\bar{2}\}$ twins. This concept suggests that the

favorability of $\{10\bar{1}1\}\langle 10\bar{1}\bar{2}\rangle$ twinning at high temperatures is likely linked to the emergence of prevalent basal slip at high temperature.

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Figure 1 (a) An orientation map and (b) the pole figures of the initial microstructure and texture of the clock-rolled and recrystallized zirconium plate. The initial texture has been measured by X-ray diffraction.

Figure 2 True stress-true strain responses of clock-rolled and annealed samples of α -Zr as a function of temperature and strain rate. The loading direction is indicated: TTC is through thickness compression, IPC is in-plane compression, and IPT is in-plane tension. Measurements are solid lines and predictions are dashed lines.

Figure 3 Variation of the initial slip resistance with temperature for (a) prismatic, (b) pyramidal $\langle c+a \rangle$, and (c) basal slip predicted by the model (lines) and data taken from the literature (symbols) [35-37, 78]. Data for Co [101] was divided by 2.5 to scale with Zr.

Figure 4 Orientation maps showing the microstructures of the Zr samples deformed to various strain levels, at several temperatures and under several strain rates as indicated in the figure. The colors in the maps indicate the orientation of the compression axis with respect to the crystal reference frame according to the inverse pole figure (IPF) triangle. Twin boundaries for four twinning modes present in the maps are highlighted in the IPF maps for (a) and (b) and in the image quality maps (e), (f), (i) and (j) for corresponding IPF maps (c), (d), (g) and (h) with a different color as specified in the legend. The scale bar of 25 μm on figure (b) is valid for all the maps.

Figure 5 Basal and prismatic pole figures showing the measured and predicted texture evolution of α -Zr deformed in in-plane compression (IPC) to the strain levels, temperatures and strain rates indicated in the figure.

Figure 6 Basal and prismatic pole figures showing the measured and predicted texture evolution of α -Zr deformed in through-thickness compression (TTC) to the strain levels, temperatures and strain rates as indicated in the figure.

Figure 7 Selected regions of the orientation maps shown in Fig. 4g and h: (a) TTC at 76 K under 0.001 s^{-1} showing secondary $\{10\bar{1}2\}\{10\bar{1}1\}$ twins (blue boundaries) within the $\{11\bar{2}2\}\{11\bar{2}3\}$ primary twins (brown boundaries) and (b) TTC at 673 K under 3500 s^{-1} showing secondary $\{10\bar{1}2\}\{10\bar{1}1\}$ twins (blue boundaries) within the primary $\{10\bar{1}1\}\{10\bar{1}2\}$ twins. In (b), the regions (not boundaries) that are colored different than red are $\{10\bar{1}1\}\{10\bar{1}2\}$ twins. Twin boundaries are highlighted with the colors consistent with labeling scheme in the Fig. 4.

Figure 8 Predicted relative activities of each slip or twin mode contributing to plasticity in the parent grain and in the primary twin domains for the IP conditions indicated in the figure. Also plotted are the parent and the primary twinned material volume fractions.

Figure 9 Predicted relative activities of each slip and twin mode contributing to plasticity in the parent grain and in the primary twin domains for the TT conditions indicated in the figure. Also plotted are the parent and the primary twinned material volume fractions. The same legend from Fig. 7 applies here.

Figure 10 Evolution of the primary and secondary twin volume fraction during IPC and TTC at 76 K and under a strain rate of 0.001 s^{-1} predicted by VPSC (lines) and measured by EBSD (symbols) as a function of strain.

Figure 11 Evolution of the primary and secondary twin volume fraction during TTC at 76 K and under a strain rate of 0.001 s^{-1} predicted by VPSC (lines) and measured by EBSD (symbols) as a function of strain.

Figure 12 Effect of basal slip on texture evolution at 673 K (a) without basal slip (b) with primary basal slip in the parent grains (c) with secondary basal slip in the twinned regions. Basal slip both in parent and twinned regions were allowed in the results presented in Figs. 2, 5 and 8.

Table 1: The matrix of tests: IPC – in-plane compression, IPT – in-plane tension, and TTC – through-thickness compression at specified strain rate and temperature conditions that were carried out in this work. [†] indicates that multiple samples were deformed and tests interrupted at several strain levels for microstructural examination.

$\begin{matrix} \epsilon \\ T \end{matrix}$	$0.001s^{-1}$	$0.1s^{-1}$	$2000s^{-1}$	$2700s^{-1}$	$3000s^{-1}$	$3200s^{-1}$	$3500s^{-1}$	$3750s^{-1}$	$4500s^{-1}$
76K	IPC [†] , TTC [†] , IPT	-	TTC	-	-	-	IPC	-	-
298K	IPC, TTC	IPC, TTC	-	TTC	-	-	IPC	-	-
473K	-	-	-	-	TTC	IPC	-	-	-
673K	-	-	-	-	-	-	TTC	IPC	IPC

Table 2 Material parameters used in the model for the evolution of slip system resistance.

	$\alpha=1 \{ \bar{1}100 \} \langle \bar{1}\bar{1}20 \rangle$	$\alpha=2 \{ 10\bar{1}1 \} \langle \bar{1}\bar{1}23 \rangle$	$\alpha=3 \{ 0001 \} \langle \bar{1}\bar{1}20 \rangle$
$b^\alpha [m]$	3.231×10^{-10}	6.0771×10^{-10}	3.231×10^{-10}
$\tau_0^\alpha [MPa]$	Eq. 3	Eq.3	Eq.3
HP^α	100	170	100
$k_1^\alpha [m^{-1}]$	5.7×10^8	3.0×10^9	5.7×10^9
g^α	0.002	0.005	0.002
$D^\alpha [MPa]$	700	1400	700
q^α	$12 \log(1 + T/30)$	0.1	$12 \log(1 + T/30)$

	$\alpha=1 \{ \bar{1}100 \} \langle \bar{1}\bar{1}20 \rangle$	$\alpha=2 \{ 10\bar{1}1 \} \langle \bar{1}\bar{1}23 \rangle$	$\alpha=3 \{ 0001 \} \langle \bar{1}\bar{1}20 \rangle$
A	40.5	800	7500
B	0.04	0.02	N/A
C	100	190	150
D	235	36000	N/A
σ	7.2	7.45	N/A
ν	0.5	0.5	N/A

Table 3 Material parameters used in the model for the evolution of twin resistance.

	$\beta=1 \{ 10\bar{1}2 \} \langle 10\bar{1}\bar{1} \rangle$	$\beta=2 \{ 11\bar{2}2 \} \langle 11\bar{2}\bar{3} \rangle$	$\beta=3 \{ 10\bar{1}1 \} \langle 10\bar{1}\bar{2} \rangle$
$b^\beta [m]$	6.33×10^{-11}	9.24×10^{-11}	2.56×10^{-11}
S	0.167	0.225	0.104
$\tau_{crit}^\beta [MPa]$	140	350	750
$\tau_{prop}^\beta [MPa]$	10	350	50
HP^β	135	100	100
$C^{1\beta}$	N/A	$1300 - 100 \log(\dot{\epsilon})$	N/A
$C^{2\beta}$	100	10	N/A
$C^{3\beta}$	N/A	$1300 - 100 \log(\dot{\epsilon})$	N/A