QUASI-FOUR-DIMENSIONAL CHARACTERIZATION OF DISLOCATION INTERACTIONS IN FCC AND HCP SYSTEMS

BY

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DISSERTATION

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ABSTRACT

Dislocation/grain boundary and dislocation/dislocation interactions have been investigated in austenitic stainless steel and α-Ti systems using in situ deformation in a transmission electron microscope, diffraction contrast electron tomography for three-dimensional analysis, and electron backscatter diffraction for orientation determination. In situ deformation experiments were conducted at room temperature and elevated temperature, as well as combined experiments where the temperature was varied over the course of an experiment. A novel technique to combine in situ deformation experiments with periodic three-dimensional snapshots is introduced and its application to understanding dislocation interactions is demonstrated. This technique removes the requirement inherent to diffraction contrast electron tomography of maintaining consistent diffraction conditions during image acquisition by using manual digital processing of the micrographs making up a tilt series prior to image alignment and tomographic reconstruction.

In the study of stainless steel, it was found that previously determined criteria for slip transfer across grain boundaries hold true at elevated temperature as well as room temperature. Details of the interactions observed at elevated temperatures, however, varied in that raising the temperature resulted in the interactions reaching higher levels of complexity earlier as well as a reduction in the barrier strength of grain boundaries. The slip transfer criteria were extended to include the emission of partial dislocations at grain boundaries. It was found that only the lead partial dislocation need be considered initially. If the trailing partial dislocation significantly increases the strain energy in the boundary, the primary system shuts down and a different
system activates. Thin film effects were not found to play a significant role in determining dislocation interactions at grain boundaries in stainless steel.

Dislocation/grain boundary interactions in α-Ti deformed at room temperature were found to adhere to previously determined slip transfer criteria during \textit{in situ} deformation. In all interactions characterized, minimization of the strain energy at the boundary was found to dictate the evolution of the interaction. Thin film effects were found to facilitate dislocation glide on planes not usually active at room temperature, increasing the number of available slip systems and potentially simplifying the interactions. Dislocation/grain boundary interactions initiated during deformation of bulk samples were found to be much more complex, emitting multiple dislocation systems simultaneously. The majority of these interactions were found to reduce the grain boundary strain energy, though not in all cases.

Dislocation interactions previously observed only \textit{post mortem} including double cross-slip and dislocation/dislocation interactions were observed \textit{in situ}. It was found that the development of jogs and cross-slip events led to dislocation generation. Sequential double cross-slip events were also seen to occur, resulting in the emission of multiple loops and half-loops from a single gliding dislocation.
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CHAPTER 1

INTRODUCTION

The importance of grain boundaries in material properties has long been understood and exploited in the development of high-strength metals. This has mainly been accomplished through grain size reduction, either by mechanical deformation or at the synthesis stage. However, recent work has shown that certain types or combinations of grain boundaries can have beneficial effects on the mechanical behavior of materials in application as well. For example, having a high density of parallel twin boundaries or interphase boundaries between dissimilar metals has been shown to increase the strength of metallic systems without sacrificing ductility [1-4]. Concomitantly, materials scientists and engineers are developing novel processing methods capable of more fully taking advantage of these new findings [5, 6].

Although significant progress has been made in experimental, computational, and theoretical work in understanding grain boundary/dislocation interactions, much is still being debated such as the role of grain boundaries in crack nucleation and propagation [7], the exact mechanisms involved in dislocation transfer across grain boundaries [8, 9], and the barrier effect of twin boundaries on dislocation motion [10]. This dearth in knowledge is due in part to limitations in both the experimental and computational techniques available. Transmission electron microscopy (TEM) has been vital in providing direct visualization of dislocation interactions, both as snapshots in time as well as dynamic in situ straining experiments (see for
example [9]). This characterization, however, is limited to two-dimensional (2D) analysis, frustrating attempts to fully characterize interactions. The present study expands the applicability of TEM characterization by combining traditional TEM imaging techniques with recently developed diffraction-contrast electron tomography, which allows a full three-dimensional (3D) investigation of the interactions [11]. This allows direct access to vital information such as the line direction and slip plane of the interacting dislocations, as well as more qualitative information such as the true 3D spatial distribution of the defects. The feasibility of combining time resolved information with the tomography-based 3D characterization will also be shown and its usefulness in understanding complex dislocation/grain boundary and dislocation/dislocation interactions will be demonstrated.
CHAPTER 2

BACKGROUND

2.1 GRAIN BOUNDARY STRENGTHENING AND POLYCRYSTALLINE DEFORMATION

Manipulation of material grain size, distribution, and orientation represent important tools in tailoring material properties to specific applications. Much of the initial work towards understanding the precise mechanisms which link grain characteristics to material properties stems from the work of Hall and Petch who independently showed that the yield strength of a polycrystalline material could be related to the grain size according to the following relationship [12, 13]:

\[ \sigma_y = \sigma_0 + kd^n \]

Here, \( \sigma_0 \) refers to the lattice friction opposing dislocation motion, \( d \) is the grain size, \( n \) is a constant fit to the experimental data, and \( k \) is a proportionality constant taken to be related to the barrier strength of the grain boundary. Currently the Hall-Petch relationship is used as an empirical formulation, where the coefficient \( k \) is calibrated through extensive mechanical testing.

Efforts to take advantage of the strengthening effects of grain boundaries have led to the development of novel materials and processing techniques with the intent of decreasing the grain size to increase strength. Examples include the development of pulsed-laser and
electrodeposition techniques that allow direct synthesis of nanocrystalline materials [14-16], magnetron sputtering for making nanotwinned materials [6], synthesis of multilayer, including nanolayer, dual phase systems [17-19], and severe plastic deformation techniques such as equal-channel angular pressing (ECAP) [20-22]. When grain orientation and boundary character is included along with grain size, it becomes possible to further tailor the material properties to application-specific requirements [6, 23-26]. The ability to predict both long and short-term material behavior in a given application without extensive testing is still lacking due in part to an insufficient understanding of grain boundary/dislocation interactions. For example, currently the $k$ factor in the Hall-Petch relationship is treated as a global constant. However, experimental work has shown that the type of boundary can have a large influence on its ability to accommodate deformation, leading to the classification of ‘hard’ and ‘soft’ boundaries [27-31]. Methods for characterizing the capacity for a boundary to accommodate deformation include electron backscatter diffraction (EBSD) [29], digital image correlation (DIC) [28, 32], micro and nanohardness tests [27], scanning electron microscopy (SEM) imaging [33], electron channeling contrast imaging (ECCI) [34-36], atomic force microscopy (AFM) [35], optical microscopy coupled with etching [37], and TEM [9, 31, 38, 39]. These methods have shown that the barrier strength of grain boundaries can vary strongly according to boundary type and dislocation type as well as due to environmental effects, leading to strain heterogeneities during deformation [10].

A persistent challenge in understanding polycrystalline deformation has been incorporating the influence of grain boundaries, including their role in heterogeneous deformation and initiation of damage nucleation sites. Strategies for incorporation of grain boundaries into plasticity models have varied strongly, with the most basic representations treating the grain boundaries as impenetrable barriers to dislocation motion [40, 41]. Using a line
tension approach, Kumar et al. showed this basic approach leads to high strain hardening rates [42], although Puri et al. showed that such a treatment of grain boundaries may be appropriate when considering high angle grain boundaries [43]. More advanced computational approaches utilize multiscale modeling techniques, with atomistic and finite element models communicating to represent the local grain boundary and mesoscale interactions, respectively [10, 44, 45]. However, the relationship between slip transfer across boundaries and damage nucleation and accumulation is still not clear, making it difficult to accurately describe the role of grain boundaries in the deformation process.

Sangid et al. showed using atomistic simulations of slip transfer across grain boundaries that the barrier strength of the boundary was proportional to the magnitude of the Burgers vector of the residual grain boundary dislocation left after slip transfer [46]. The grain boundary energy was considered to be an important factor in the barrier strength, which has motivated a number of molecular dynamics based studies on extracting grain boundary energies [47-50] with much of this work being framed in terms of the coincident site lattice (CSL) index [51]; see for example Figure 2.1 displaying variations in grain boundary energy with rotation angle about the [011] direction in Ni. Bieler et al. similarly focused on local slip conditions around a boundary, with slip incompatibilities across the boundary indicating potential fracture initiation sites. This information was incorporated into a finite element model to predict heterogeneous strain developments and damage initiation during deformation of polycrystalline materials [10]. Clayton and McDowell used strain incompatibilities across grain boundaries to develop a fracture parameter which, when coupled with damage nucleation from preexisting pores and microcracks, could be incorporated into a finite element framework to similarly predict fracture initiation [44].
Figure 2.1. Graph constructed by Sangid et al. showing the grain boundary energy as a function of tilt angle about the [110] direction in Ni [46].

Other modeling attempts at understanding heterogeneous deformation have considered in particular the role of triple junctions in arresting dislocation motion [52, 53], accumulation of geometrically necessary dislocations around grain boundaries [54], the interaction strength and energy associated with different dislocation/grain boundary combinations [55, 56], anisotropy of grain boundary energies [57], cascading effects as dislocation interactions with grain boundaries can lead to dislocation ‘avalanches’ in neighboring grains [58], and the effects of local grain boundary features such as ledges [7, 59, 60]. What is clear from these studies is that to accurately model polycrystalline deformation in a manner which allows predictive capabilities of material behavior over extended time periods, a fundamental understanding of grain boundary/dislocation interactions is essential [61, 62].
2.2 UNDERSTANDING DISLOCATION/GRAIN BOUNDARY INTERACTIONS

Four distinct responses to dislocation impingement on a grain boundary have been identified experimentally: (1) Dislocation absorption into the boundary plane, upon which the dislocation is either converted into an extrinsic grain boundary dislocation or retains its lattice Burgers vector; (2) direct transfer across the boundary into the neighboring grain; (3) absorption of the dislocation into the grain boundary and subsequent emission of a dislocation into a neighboring grain; and (4) dislocation absorption into the boundary and emission back into the original grain [9]. The reaction at the boundary is thought to be governed by the character of the incoming dislocations and grain boundary, including the line direction, Burgers vector, and slip plane, as well as the stress state, including both the local and macroscopic influence. For example, direct transfer across the grain boundary requires the incoming dislocations to be screw in nature and for the slip planes in the incoming and outgoing grains to be aligned such that the Burgers vector can be fully transferred with no residual dislocation created in the boundary. Satisfying this condition requires the slip planes to share a common line of intersection in the grain boundary [63], though Brandl et al. have claimed based on observations made during atomistic computer simulations that local bending of planes in grain boundaries can relax this condition [64]. Additional factors shown to influence the system response include the system temperature [65], strain rate [66-68], grain size, especially in the nanoscale regime [69-73], and presence of a hardened matrix as may be encountered in irradiated materials [74].

Efforts in understanding grain boundary/dislocation interactions have been directed towards developing predictive capabilities of both the barrier strength of a grain boundary and
the likely grain boundary response if given a defined incoming dislocation system. The majority of progress in furthering our understanding of these interactions has been focused on FCC materials which will be summarized below, with more limited studies conducted on BCC, HCP, and intermetallic systems, which will be reviewed following the review of FCC systems. A separate section is devoted to dislocation interactions with $\Sigma3$ boundaries as they are of special interest to the materials engineering community.

2.2.1 Slip transfer in FCC systems

Livingston and Chalmers developed a predictive model for dislocation emission from a grain boundary through investigations of aluminum bicrystals deformed in tension [75]. By considering strain continuity at the boundary and the resultant shear stress state, they proposed the following variable $N$ to be maximized:

$$N = (n_{in} \cdot n_{out})(g_{in} \cdot g_{out}) + (n_{in} \cdot g_{in})(n_{out} \cdot g_{out})$$

where $n$ refers to the slip plane normal of the incoming (in) and outgoing (out) slip planes and $g$ refers to the slip directions. Using these criteria, they were able to successfully predict the outgoing slip system when given a characterized boundary and incoming slip system based on the surface slip trace of the incoming and outgoing systems.

Shen et al. proposed two additional criteria to predict the dislocation response at the grain boundary [76]. They claimed that the emitted system should minimize the angles between the Burgers vector of the incoming and outgoing dislocations as well as the line of intersection made
from the slip planes of the incoming and outgoing dislocations and the grain boundary. Mathematically, this is given as the variable $M$ being minimized,

$$M = (\mathbf{I}_{in} \cdot \mathbf{I}_{out})(\mathbf{g}_{in} \cdot \mathbf{g}_{out})$$

where $\mathbf{I}$ is the line of intersection made between the incoming and outgoing system slip planes and the boundary. The second additional criterion proposed was that the resolved shear stress exerted on the outgoing system from pileup of dislocations in the incoming system should be maximized. The combined line direction/slip direction criterion was used to predict the slip plane and the resolved shear stress was used to predict the slip direction. Comparison with TEM analysis of deformed austenitic stainless steel samples showed that in the majority of cases, the proposed criteria were sufficient for predicting the correct emitted system. They were also shown to be more reliable than the criteria proposed by Livingston and Chalmers.

Lee et al. proposed an addition to the criteria given by Shen et al. They used the following three conditions [9, 77, 78]:

1) The angle between the line of intersection made with the incoming and outgoing slip planes and the grain boundary should be minimized

2) The magnitude of the Burgers vector of the residual dislocation ($|\mathbf{b}_{r}^{gb}|$) left in the boundary after the interaction should be minimized

3) The resolved shear stress acting on the emitted slip system should be maximized

They found through in situ TEM straining experiments of 310 austenitic stainless steel samples that criterion 1 was not necessarily satisfied in all cases. Criterion 2 was found to be most important, but in some cases criterion 3 was found to dictate the interaction. In these cases, the
dislocation emission could only continue temporarily before the buildup of elastic strain in the boundary forced the initial system to shut down and a different system, which did satisfy criterion 2, was activated. In cases of multiple incoming/outgoing systems, criterion 2 was still found to hold [77]. That is, the system response mainly seemed to be driven by the minimization of elastic strain buildup, which is analogous to the magnitude of the Burgers vector of the residual grain boundary dislocation.

Computational methods have added additional atomistic details to the understanding of the interactions as well as proposing or modifying the slip criteria proposed by Lee et al. [8, 79-81]. These simulations have the benefit of considering a perfectly characterized system in a controlled environment. Individual atoms can be tracked at any moment in time, providing detail and resolution not available in any experimental technique. However, computational expense limits the simulations to high strain rates over short periods of time, in small areas, and at unrealistically low temperatures. Generally only the initial response at the grain boundary from impinging dislocations can be simulated, with longer term effects not captured in the simulations. The simulations generally consider only grain boundaries in equilibrium, limiting the scope of grain boundaries that can be investigated [82, 83].

Bachurin et al. used molecular dynamics (MD) simulations to investigate various dislocation interactions with [111] tilt grain boundaries in a Ni thin film [79]. For low angle grain boundaries, they added the additional criteria that dislocations with a Burgers vector sign which increased the grain boundary misorientation upon transmission penetrated more easily than those with an opposite sign. They also observed that lead and trailing partial dislocations need not nucleate at the same point, free surfaces facilitate the nucleation of dislocations, the pinning points in a grain boundary occur at the centers of hydrostatic compressive stresses, and
the grain boundary inclination has minor influence on the observed mechanisms. In the case of high-angle tilt boundaries, they claim that the ability of the grain boundary to accommodate dislocations makes it unlikely that the incoming and outgoing glide planes will align. They also claim, considering it as a violation of the slip transfer criteria proposed by Lee et al., that high-angle boundaries can emit only leading partials and cannot nucleate the trailing, which is an odd claim in light of experimental evidence to the contrary [76].

In a study similar to Lee et al. but in a computational environment, Dewald and Curtin published a series of papers simulating dislocation interactions in Al using a coupled atomistic/discrete dislocation (CADD) model [8, 80, 81]. They proposed a modified set of the slip transfer criteria which add the following to the slip transfer criteria developed by Lee et al.:

1) The magnitude of the step of the residual dislocations left in the boundary after an emission event should be small;
2) For transmission to occur, the resolved shear stress on the grain boundary should be small;
3) For formation of a grain boundary dislocation, the primitive vectors of the grain boundary and the associated step with any grain boundary dislocation should be small;
4) The normal compressive stress on the boundary should be small;
5) If a lagging lattice Shockley partial dislocation remains near the intersection without being absorbed, the resolved shear stress acting on the leading pileup dislocation need not be high; and
6) Transmission of a leading Shockley partial dislocation necessitates the formation of an intrinsic stacking fault.
Additionally, they claim that their proposed criteria should be applied to partial dislocations only, not to the complete dislocation. They did not claim that the criteria gave fully predictive capabilities as the relative importance of each criterion varies with many factors such as details of the grain boundary structure and the location of the dislocation intersection with the grain boundary. De Koning et al. similarly found by computational studies on grain boundary/dislocation interactions in Al that the complexity of the interactions precludes the possibility of developing a simple set of rules to predict the system response to dislocation impingement on a grain boundary [84].

Associated with the study of slip transfer across grain boundaries is the nucleation of dislocations at grain boundaries and the influence of boundary features on the nucleation process. In 1963, Li postulated that ledges in grain boundaries could act as sources of edge dislocations [85], with Price later proposing a similar model for the nucleation of screw dislocation from boundary ledges [59]. The presence of ledges and their influence on dislocation behavior, specifically on the flow stress of metals, was later supported through mechanical testing and TEM investigations of deformed materials [86, 87], with direct evidence of dislocation emission from boundary ledges coming later [66, 88]. In a molecular dynamics study on the effects of ledges on dislocation nucleation, Capolungo et al. found that at 10 K, the stress required for the nucleation of a partial dislocation decreased from 6.1 GPa to 5.8 GPa when a ledge was present on the boundary plane. This nucleation is shown in Figures 2.2 and 2.3, where the partial dislocation is seen nucleating directly from the ledge.

Outstanding issues yet remaining in understanding dislocation/grain boundary interactions in FCC materials include the effects of temperature on the interaction and how criteria determining the interactions may vary when partial dislocations are considered. The
reported experimental work on dislocation/grain boundary interactions were performed at room temperature and the computational experiments were simulated at or near 0 K. Materials are known to become more ductile at higher temperatures, though this has not explicitly linked to any change in interactions at the grain boundaries. Also, while the simulations consider almost exclusively partial dislocations, experimental work has focused on perfect dislocations interacting with boundaries. More experimental work is needed to verify the results of the simulations.

Figure 2.2. Nucleation of a partial dislocation from a defect free $\Sigma 5$ boundary shown in a molecular dynamics model. The straining occurred in a simulated environment at 10 K under uniaxial tension. Atoms are colored by the centrosymmetry parameter [7].
Figure 2.3. Nucleation of a partial dislocation from a $\Sigma 5$ boundary containing a ledge shown in a molecular dynamics model. The straining occurred in a simulated environment at 10 K under uniaxial tension. Atoms are colored by the centrosymmetry parameter [7].

2.2.2 Interactions with $\Sigma 3$ grain boundaries

Due to their high coherence, high prevalence in certain systems, and unique and often desirable properties, $\Sigma 3$ boundaries have been studied more thoroughly than any other type of boundary. They have, for example, motivated the development of nanotwinned materials, which show increased strength without the loss in ductility commonly seen in nanograin materials [89]. However, this intense scrutiny has led to varying opinions on their role in deformation processes, with the boundaries themselves treated as anywhere from impenetrable barriers to dislocation motion to posing no effective barrier to slip transfer [10, 90].

*In situ* TEM straining has shown that screw dislocations can directly transmit through a coherent $\Sigma 3$ boundary when the slip planes on either side of the boundary are exactly aligned and the Burgers vector of the dislocation is fully preserved during the transmission [9]. This requires that the line direction of the screw dislocation is parallel to the direction of the line formed by the
intersections of the slip planes and the grain boundary. A significant dislocation pileup formed prior to the observed transmission, suggesting that even in cases of direct transmission the boundary still poses some barrier to dislocation motion. Poulat et al., in a weak-beam dark-field study of dislocations interacting with Σ3 boundaries in Ni, found that the boundaries pose a barrier to dislocation motion even when the glide planes of the dislocations in both crystal shared a common trace in the boundary plane [91, 92]. The impinging dislocations were fully absorbed into the boundary and interacted with intrinsic grain boundary dislocations before emitting into the neighboring grain. As no interactions were described in their papers that resulted in complete transfer of the Burgers vector across the grain boundary, it is unknown how they came to this conclusion.

Kashihara and Inoko investigated the interactions of dislocations with Σ3 boundaries using Al bicrystals in the SEM [93]. They found that the interactions obeyed the slip transfer criteria proposed by Lee et al. and that the barrier strength of the boundary was proportional to the magnitude of the Burgers vector of the residual grain boundary dislocation left after the interaction. Similar to Lee et al., they observed easy transmission of screw dislocations across the boundaries, but edge dislocations could not pass through the boundary and their absorption played an important role in inducing boundary motion.

Coupling in situ nanoindentation experiments in the TEM with atomistic simulations, Li et al. proposed a new dislocation multiplication process associated with dislocation/twin boundary interactions [94, 95]. They observed the dissociation of a perfect dislocation absorbed into a coherent twin boundary into a Frank partial disconnection, which can be thought of as the combination of a step in the boundary with an edge dislocation [96], and a twinning dislocation. The Frank partial disconnection in the boundary instigated the emission of a second twinning
dislocation into the matrix. High resolution imaging during the in situ nanoindentation allowed for the direct observation of steps forming in the boundary associated with the glide and emission of twinning dislocations.

Molecular dynamics based studies have in detail described the interactions of dislocations of various Burgers vectors with coherent Σ3 boundaries. Jin et al. simulated screw dislocations impinging on a Σ3 boundary in Al, Cu, and Ni FCC systems [97]. They found in Al that when dislocations impinged on a Σ3 boundary they split into Shockley partial dislocations. The individual partial dislocations propagated in opposite directions along the boundary shifting the boundary by one Burgers vector (Fig. 2.4). The stacking error was perfectly accommodated in the boundary, meaning that the energy barrier dictating the separation of partial dislocations did not apply. In the Cu and Ni simulations, the dislocations were observed to cut through the boundary leaving no residual dislocation, similar to what was observed experimentally by Lee et al. (Fig. 2.4). They hypothesized that the differences between $\gamma_S$, $\gamma_{US}$, and $\gamma_{UT}$ could account for the differences in observed behavior. Here $\gamma_S$ is the stacking fault energy of the material, $\gamma_{US}$ is the unstable stacking fault energy, or the energy needed to create an intrinsic stacking fault from a perfect lattice, and $\gamma_{UT}$ is the energy needed to create a twin fault along a pre-existing twin plane.
Figure 2.4. Snapshots of screw dislocations interacting with a coherent Σ3 twin boundary during simulated deformation with associated schematic. The simulated material is given in the first frame of each interaction. Simulation time is given in each image. Coloring is according to the potential energy for each atom [97].

In a similar study to Jin et al., Chassagne et al. investigated screw dislocation interactions with coherent Σ3 boundaries using molecular dynamics, but came to a slightly different conclusion [98]. They determined that the interaction at the boundary, whether the dislocations were transmitted or split into Shockley partials in the boundary, was dictated by the ratio $\gamma_s/\mu b$, where $\gamma_s$ is the stacking fault energy and $\mu$ is the shear modulus.
where \( \mu \) is the shear modulus and \( b \) is the magnitude of the lattice dislocation Burgers vector. They also found that the transmission of a dislocation through the boundary was not direct and relied on reaching a critical stress level of close to 400 MPa, below which the dislocations were only absorbed into the boundary plane.

In a separate study, Jin et al. investigated interactions of non-screw dislocations with coherent \( \Sigma 3 \) boundaries, again using molecular dynamics computer simulations [99]. As expected, these interactions became much more complex, but the reaction pathway was still dictated by the stacking fault energy of the material. They found that interactions proceeded through nucleation of partial dislocations and the formation of Lomer locks in the boundary, with the resistance to nucleation of partial dislocations given by:

\[
R = \left( \frac{\gamma_{US} - \gamma_S}{\mu b} \right) \text{ on a normal glide plane and}
\]

\[
R = \left( \frac{\gamma_{UT} - \gamma_S}{\mu b} \right) \text{ on a common twin plane.}
\]

Thus, an important factor on the interaction was the applied stress level as it determined whether or not a partial dislocation nucleated at the boundary (Fig. 2.5). They also observed that the twin boundaries can act as an effective trap for both screw and non-screw dislocations. In looking at nanotwinned materials using molecular dynamics simulations, Wu et al. observed similar complexity of non-screw interactions with coherent twin boundaries, which they hypothesized could act as an important mechanism for dislocation multiplication in nanotwinned metals, preserving their ductility [100, 101].
The lack of consensus among the computer simulations highlights the need for further experimental investigation of dislocation interactions with Σ3 boundaries. How these interactions vary with dislocation type has not been extensively explored outside of a computational environment. Also, similar to grain boundary interactions in FCC materials in general, the effects of temperature on these interactions is not well known.
Figure 2.5. MD snapshots of incoming 60° dislocation interacting with a coherent Σ3 twin boundary in Al. The applied strain varied for each interaction and is given below each set of images [99].
2.2.3 Slip transfer in Ti and other HCP systems

Slip transfer in HCP systems include additional factors of complexity due to the anisotropic nature of deformation, with the preferred slip plane being dependent on the material-specific c/a ratio. For example, the most prominent slip systems in α-Ti are the \{110\}<11\overline{2}\overline{0}\> (prismatic slip), but slip can also activate on the \{1\overline{1}\overline{0}\} planes (pyramidal slip) and the (0001) plane (basal slip). While \textbf{a}-type dislocations (\langle1100\rangle-type) are most common, there are also reports of \textbf{c+a}-type (\langle11\overline{2}\overline{3}\rangle-type) being active (see Figure 2.6 for a schematic of all commonly available slip systems in HCP systems). Though computational investigations of dislocation/grain boundary interactions in HCP systems are limited, there have been a few experimental studies.

![Figure 2.6. Common slip planes in HCP crystals with the associated families of Burgers vectors glissile on each plane.](image)

Figure 2.6. Common slip planes in HCP crystals with the associated families of Burgers vectors glissile on each plane.
Kehagias et al. investigated mechanically deformed α-Ti in the TEM with the aim of verifying the role of twinning dislocations and comparing the observed slip transfer behavior to the criteria proposed by Lee et al [102]. They found that minimization of $|\mathbf{b}_{rb}|$ and minimization of the angle of intersection between the incoming and outgoing slip planes with the grain boundary were sufficient to correctly predict the grain boundary reaction from impingement of a dislocation system. As dislocations of a single type can slip on either the prismatic or pyramidal planes in α-Ti, the resolved shear stress criterion was used to determine which was more likely. They found that, similar to the observations of Lee et al., these criteria were sufficient to correctly predict the response at a grain boundary to dislocation impingement, with minimization of $|\mathbf{b}_{rb}|$ being the most important factor. Low-angle, high-angle, and twin boundaries were examined. However, only $<$a>-type dislocations were considered and observed in the study.

In a similar study, though using in situ TEM deformation, Shirikoff et al. investigated grain boundary dislocation interactions in α-Ti 4 wt % Al [103]. Three separate interactions were observed and one, slip transfer across a random high-angle grain boundary, was characterized for the purpose of comparing the interaction to the criteria proposed by Lee et al. In the characterized interaction, $<$a>-type dislocations impinged on the boundary, resulting in a primary system of $<$a>-type dislocations emitting from the boundary, as well as more limited emission of $<$c+a>-type dislocations into the same grain and $<$a>-type dislocations being back-emitted into the same grain as the impinging dislocations. They showed that consideration of $|\mathbf{b}_{rb}|$ correctly predicted the Burgers vector of the emitted dislocation, and consideration of the resolved shear stress allowed identification of the slip plane. The main observed emitted system slipped on the (011 0), which is the most energetically favorable plane for dislocation glide in Ti. It is yet
unknown if the criteria hold true for predicting dislocation emission on the pyramidal planes as well, or if energy considerations are a more important factor. The other two systems emitted acted to further reduce the residual grain boundary dislocation in accordance with the slip transfer criteria. Only a single interaction was fully characterized due to the difficulty of collecting enough diffraction information for Burgers vector characterization during in situ deformation.

Dislocation interactions and cross-slip events can lead to the formation of subgrain boundaries made up of arrays of intersecting dislocations, as reported by Zhang et al. [104]. During in situ deformation of α-Ti in the TEM, they observed dislocation interactions with these subgrain boundaries and found that slip transfer through the boundaries depended on the effect of the crystal rotation on the slip plane of the dislocations. In cases where the crystal rotation did not cause a deviation of the slip plane, the dislocations could pass through the boundary unimpeded. If the boundary misorientation did cause a redirection of the dislocation motion, the barrier strength of the subgrain boundaries was found to be proportional to the misorientation. Slip transfer through the boundary occurred either through transmission of dislocations through climb and/or cross-slip mechanisms or through absorption of the dislocations and nucleation and emission of a new dislocation system.

Serra and Bacon investigated dislocation interactions with twin boundaries in pure α-Ti using atomistic simulations, focusing on how these interactions varied based on the dislocation slip plane, basal or prismatic, and the twin boundary habitat plane, \{1\bar{0}1\} or \{10\bar{1}2\} [105]. To simplify the analysis, only \textbf{<a>}-type screw dislocations were considered. They found that two possible interactions can occur at the boundary: transmission across the boundary via slip onto an accommodating plane or dissociation into twinning dislocations. The former interaction occurred
during interactions with the \{1012\} and could result in slip onto less energetically favorable planes such as the basal plane. The latter interaction was mainly associated with interactions with the \{1011\} twins and resulted in the formation of a step in the boundary plane. In both cases, the twin boundary acted as a barrier to dislocation motion. This was attributed to the dislocations being forced to constrict prior to cross-slip.

The computer simulation results are largely in agreement with the experimental findings of Lay and Nouet in their investigation of dislocation/twin interactions in zinc [106]. In the TEM, they observed primarily \(<a\)-type dislocations interacting with \{1012\} twins and noted three possible interactions. First, if the dislocations and slip planes aligned on either side of the boundary favorably, the dislocation could pass unimpeded through the boundary with no residual grain boundary dislocation. The second observed interaction occurred when direct transfer was not possible. The impinging dislocation underwent decomposition in the boundary, resulting in two dislocations with smaller Burgers vectors. This second interaction was considered to be especially interesting as the decomposed dislocations could potentially act as twinning dislocations responsible for further twin growth. The third interaction involved the recombination through climb of boundary dislocations with lattice dislocations that could be responsible for elimination of steps in the boundary as well as reduction of the density of dislocations in the twin interior.

In studies on grain boundary structure using molecular dynamics simulations, Wang and Beyerlein introduced a possible mechanism for the formation of deformation twins in magnesium [107, 108]. They showed that the impingement of basal dislocations on symmetric tilt boundaries can create a stress field sufficient to cause the dissociation of nearby grain boundary dislocations. Once dissociated, the dislocations could then recombine as twinning
dislocations, leading to the formation of either \{10\bar{1}1\} or \{10\bar{1}2\} twins, depending on the nature of the original boundary. The dominant, and at room temperature only, generally active slip systems in magnesium are the \{11\bar{2}0\}(0002), which provide only two independent slip systems (the third possible slip system being a combination of the other two). For this reason, twinning at the grain boundaries instead of the activation of additional slip systems is a common response.

2.2.4 Slip transfer in other systems

Grain boundary/dislocation interactions have been studied in a number of other systems besides FCC materials, including BCC [38, 109, 110], L12 compounds [111-113], intermetallics [9, 114, 115], interactions across phase boundaries [116-119], and even in ice crystals [120]. These studies generally showed that, similar to what has been seen in FCC systems, slip can transfer across the grain boundary through direct transfer or through stress induced nucleation of dislocations in the neighboring grain or phase. Two of the studies directly compared the observed slip behavior to criteria proposed by Lee et al. Of these, it was found that the criteria correctly describe the behavior of dislocation transmission for BCC Fe-4 at.%Si [38] and Ni3Al [9]. However, only a single grain boundary/dislocation interaction was used for comparison to the criteria in each study.
2.3 DISLOCATION INTERACTIONS IN $\alpha$-TI

Of equal importance to understanding dislocation/grain boundary interactions during deformation of crystalline materials is the understanding dislocation interactions in the grain interior, including interactions with other dislocation and the prevalence of slip on certain planes. Experimental observations have confirmed and computational studies support that the most prevalent slip system in $\alpha$-Ti is $<11\bar{2}0>\{1\bar{1}00\}$ [121-126]. This can vary when stress is applied to highly textured or single crystal samples oriented for favorable slip on different planes. For example, Shechtman and Brandon reported primary basal slip when the Schmidt factor associated with basal slip was approximately 4 times higher than that associated with prismatic slip during room temperature deformation [121]. Based on their experimental results, they also concluded that pyramidal slip could only function as a secondary system activated through cross-slip from the prismatic planes. The resolved shear stress needed to activate pyramidal slip was reported to be approximately 2.5 times higher than that reported for prismatic slip. $<c+a>$-type dislocations were seen only rarely and were considered to be an insignificant contribution to the overall slip behavior. The relative critically resolved shear stress (CRSS) values vary strongly with alloy type. For example, Bieler and Semiatin reported a ratio of 0.7:1:3 for the CRSS values of prism $<a>$: basal $<a>$: pyramidal $<c+a>$ slip in Ti-6Al-4V [127].

Besides orientation, the prevalence of slip on different planes has also been reported to vary as a function of temperature and concentration of solute atoms [123, 128]. Naka and LaSalmonie reported a transition temperature near 27°C where cross-slip became much more prevalent, leading to higher levels of work hardening with the increasing frequency of intersecting dislocations [125]. They postulated that variations in oxygen mobility and pinning points with temperature strongly influenced their observations, but could not confirm this as
oxygen was not detected directly in the experiments. Churchman reported seeing a lower prevalence of slip on the basal or pyramidal planes with increasing oxygen and nitrogen levels [129]. He proposed that this was due to the preferential interstitial sites occupied by the solute atoms which inhibited slip on non-prismatic planes.

TEM investigations of α-Ti samples prepared from deformed bulk material have further shown how cross-slip can lead to dislocation multiplication. In a mechanism schematically shown in Figure 2.7, Naka and LaSalmonie described a process in which sections of screw dislocations underwent double cross-slip events from prismatic to pyramidal planes, leading to pinning points forming and loop expansion occurring [125]. This led to a prevalence of edge dipoles found throughout the deformed material. The cross-slip events were only observed post mortem after ex situ deformation of bulk materials. In situ TEM straining was also conducted by the same group. Slip was again seen predominately on prismatic planes. However, glide on a large number of planes not usually associated with slip in Ti was also observed. They attributed this behavior to thin film effects.
Figure 2.7. Loop formation in $\alpha$-Ti following a double cross-slip event. P.S.P. and C.S.P. refer to the primary and conjugate slip planes, respectively [125].
In a study on cross-slip in various materials using *in situ* straining in a high voltage electron microscope, Messerschmidt and Bartsch observed similar cross-slip events in a Ti-6Al alloy [130]. Characterization of the slip plane during the interaction showed the glide and cross-slip planes to be the active on both the {0001} and {1100}, which differs somewhat from previous reports where cross-slip was mainly observed onto the pyramidal planes. This may an alloying effect as the majority of other studies were conducted on commercially pure materials. Direct comparison cannot be made as Messerschmidt and Bartsch’s data were not published; only the final results as reported here are available.

As few computational studies have focused on dislocation interactions in HCP materials, progression in this field has relied almost exclusively on experimental and theoretical work. Previous work has focused on *post mortem* characterization of dislocation interactions, and so little has been confirmed of the dynamics of dislocation interactions. For example, while the dislocation configurations after *ex situ* deformation suggest the occurrence of previous double cross-slip events, there are no published studies reporting observations of these interactions occurring during *in situ* deformation. *In situ* characterization can further the understanding of these interactions by identifying when they occur during the deformation and the dislocation behavior both before and after an event occurs.
2.4 ELECTRON TOMOGRAPHY

2.4.1 Conventional electron tomography

TEM was responsible for the first direct observations of dislocations [131, 132], and has since been crucial in furthering our understanding of dislocation character and dynamics [133]. Present needs in furthering our understanding of the mechanical behavior of materials motivate the continuous development of dislocation characterization techniques. These developments have come in part through more powerful and diverse electron microscopes such as dynamic TEM [134] and spherical aberration corrected TEM [135], stages allowing novel in situ experiments such as nanoindentation [136], and new techniques including both software and hardware advances such as orientation mapping using precession diffraction [137]. One area still needing further development is microscopy techniques which allow complete characterization of defect interactions, including the 3D spatial distribution of defects as well as time-resolved information, providing the full four-dimensional (4D) analysis. Current 4D characterization techniques in materials science do not provide the spatial or temporal resolution needed to understand dislocation interactions at the local level. 3DXRD using a synchrotron source allows for 4D characterization of a microstructure, but the large interactions volume of x-rays limits the spatial resolution to the micron level [138-140]. Zewail et al. have developed a technique known as ultrafast microscopy to couple dynamic TEM, which has a temporal resolution of picoseconds, with electron tomography to achieve 4D high resolution characterization [141, 142]. However, their technique is limited to interactions that can be instigated by a laser pulse and which revert to their original state after each pulse making it unsuitable for dislocation characterization.
Conventional electron microscopy is inherently limited to 2D projections of a 3D defect state. Direct 3D imaging of dislocations was first accomplished using stereographic pairs, and later full series of images, in X-ray topography [143, 144]. However, the large interaction volume of X-rays limits resolvable dislocation separation to approximately 10 μm, limiting the technique’s applicability. Dislocation stereography was later extended to TEM analysis, where the higher spatial resolution allows more complex dislocation interactions to be imaged and analyzed. Two images collected from the same area but at different tilt angles separated by approximately 15° can be viewed in a stereoscopic imager or used to create a red/blue anaglyph, resulting in a 3D representation of the defect structure [145, 146]. The resultant stereogram is limited to viewing from a single vantage point and requires red/blue glasses or a stereoviewer to present.

Electron tomography, first introduced in 1968 [147, 148], seeks to overcome challenges associated with stereographic pairs through the collection of a large number of images over a wide angular tilt range, generally from 120-140° with an image collected every degree. These images must satisfy the projection requirement, which states that the intensity of an image must be a monotonic function of the sample thickness and no more than one other physical characteristic of the material [11]. In order to reduce the time needed for collecting images, and thus reduce the beam damage induced during acquisition of a tilt series, techniques have been developed to automate the image acquisition process [149-151]. These innovations and the application of electron tomography in general have been limited mainly to the life sciences due to the difficulty of satisfying the projection requirement for crystalline materials, though there have been notable advances in the physical sciences by using high angle annular dark-field (HAADF) STEM imaging to minimize dynamic scattering effects [152-154].
The fundamentals of electron tomography lie in the ability to represent an image as a series of frequencies and corresponding coefficients using Fourier transforms and the associated projection theorem. Fourier transforms allows an N-dimensional image to be converted between representative frequencies and a real space image or spectrum with only the translation remaining ambiguous. The projection theorem of Fourier analysis states that the Fourier transform of a 2D projection of a 3D object is identical to the center slice of the 3D Fourier transform of the object. Thus, a series of projection images transformed into Fourier space can be used to construct the 3D Fourier transform of the original object. If images are collected at a small tilt increment over the full range of tilt, ±90°, the object can be perfectly reconstructed. However, as the TEM and stage geometry generally limit the available tilt range and images are collected at discrete tilt increments, any reconstruction from Fourier space must deal with a “missing wedge” of information, which is readily apparent when data are presented in Fourier space (Fig. 2.8). Tilting schemes such as conical tilting, using two tilt axes, can partially fill this missing wedge. However, overcoming this missing information remains a central challenge of tomographic reconstructions in electron microscopy.
Figure 2.8. Tomogram visualization of silver nanoparticles in a metal organic framework in real space (a) and Fourier space (b). Notice that individual planes, corresponding to images taken at regular intervals in the TEM, are visible in the Fourier space representation (shown enlarged in inset). Image courtesy of Stephen House.

Although various reconstruction algorithms exist [155-157], the weighted back-projection algorithm, which is used in this study, will be the main focus of discussion here. As its name suggests, back-projection algorithms rely on projecting the image back to the sample plane [155]. The back-projections are summed, resulting in higher intensity regions where the object of investigation is located. The resulting summation is convoluted with a point-spread function which describes the resultant image from the input of a single point into the imaging system, in this case the TEM. A weighting function is used to correct oversampling of regions nearest the tilt axis. For tilt series collected using a single tilt axis at regular tilt intervals, this weighting function has the analytical form:

\[ W_s(R, Y, \Gamma) = R \]
Here, \( Y \) is the distance from the origin along the tilt axis and \( R \) and \( \Gamma \) are the cylindrical coordinates in the XZ plane. That is, the weighting function varies symmetrically and linearly with distance from the tilt axis.

The spatial resolution of electron tomography is somewhat difficult to define as it does not depend on any one variable. Instead, it is the summation of multiple factors including the tilt range, which directly determines the size of the missing wedge, tilt increment, spatial resolution of the source images, and signal to noise ratio of the source images [156]. If one assumes that images are collected over the full tilt range, \( \pm 90^\circ \), the resolution in the off-tilt axis directions, \( y \) and \( z \), can be related to diameter of the object of interest \( D \) and the number of images collected over \( 180^\circ \) \( N \) by [158]:

\[
d_y = d_z = \frac{\pi D}{N}.
\]

The resolution in the direction parallel to the tilt axis is equal to the resolution of the source images.

As most practical applications of electron tomography do not access the full tilt range, an expression is needed to relate the tomogram degradation to the tilt range. This has been derived by Radermacher et al. in the form of an elongation factor \( e_{yz} \) which acts on the resolution in the direction perpendicular to the optical axis as [159]:

\[
d_z = d_y e_{yz}
\]

and can be related to the maximum tilt angle \( \alpha \) by
The elongation factor, $e_{yz}$, is plotted as a function of $\alpha$ in Figure 2.9 with the tilt range of a standard double-tilt stage indicated. The resolution degradation is shown in Figure 2.10 where a 2D image was converted into Fourier space using a Radon transformation and converting back into real space again, but limiting the information used in the reconstruction to simulate the effects seen in electron tomography. As a rule of thumb, Midgley et al. found that the resolution of large objects, $D > 100$ nm, is approximately $D/100$ [154].

Figure 2.9. Elongation factor $e_{yz}$ as a function of the tilt semi-angle. The approximate tilt range of a standard double-tilt stage is indicated by the black circle.
Figure 2.10. Schematic of resolution degradation with limited tilt range and number of images. Top two rows show image elongation from limiting the tilt range (given in upper left corner of each image) for images taken every one degree. Bottom row shows image degradation with number of images (given in upper left corner of each image) taken from a ±90º tilt range.

Additional algorithms reliant on iteratively updating the final reconstructed tomogram using the source images from an acquired tilt series have also been developed (see, for example, the algebraic reconstruction technique (ART) [160], the simultaneous algebraic reconstruction technique (SART) [161], and the simultaneous iterative reconstruction technique (SIRT) [162]). These algorithms use algebraic expressions to compare the reconstructed tomogram to the projection images acquired in the TEM to retrieve the information contained in the “missing wedge.” If the images composing a tilt series can be represented using only a few discrete gray levels, the algebraic technique can be refined using a discrete algebraic reconstruction technique (DART) to reduce noise levels and sharpen the edges of objects in the reconstructed tomogram.
While theoretically more robust and accurate than weighted back-projection algorithms, computational costs have limited the use of iterative techniques, though they are seeing more widespread use with the increase in available computational power.

2.4.2 Extension of electron tomography to dislocations

Recently, Barnard et al. showed that electron tomography could be extended to defect analysis in crystalline materials [11]. By maintaining the tilt axis used during acquisition of the tilt series parallel to a crystal plain normal, the diffraction conditions, diffraction vector (g) and excitation error (s), can be kept constant and contrast variations between images are kept to a minimum. Barnard et al. demonstrated the feasibility of this technique by reconstructing a tomogram of dislocations gliding from a crack tip in GaN. Since then, dislocation tomography has been applied to resolving a number of dislocation interactions. Higashida et al. used dislocation tomography to identify the role of dislocations in stress shielding in front of a crack tip in silicon [164-168]. Liu et al. applied dislocation tomography to resolving the complex dislocation behavior around precipitates in an Al-Mg-Sc system [169]. Resolution of the 3D structure allowed identification of dislocation cross-slip near the precipitate interface, clarifying the source of dislocation debris generated during dislocation/precipitate interactions. Tanaka et al. also studied precipitate interactions using dislocation tomography, though they looked at dislocation generation around oxide precipitates in silicon [170]. The addition of 3D characterization allowed identification of the dislocation line directions and slip plane, highlighting sessile sections of dislocation loops that could potentially act as Frank-Read sources. These details were not noticed in previous studies that relied on conventional electron
microscopy to investigate the interactions as all segments of the dislocation loop were assumed to share a common plane [171]. Kacher et al. investigated the complex structure of defect free channels formed in deformed irradiated stainless steel samples [172].

Various imaging techniques have been used during the image acquisition process, each with unique benefits and limitations. These tradeoffs include spatial resolution, defined mainly by the apparent dislocation width, signal to noise ratio, ease of use, and prevalence of unwanted diffraction effects. During the original development of dislocation tomography, it was hypothesized that if dislocation overlap was minimized and the dislocation contrast was limited to a localized region around the core, the projection requirement would be sufficiently satisfied [11]. With an apparent dislocation width of approximately 1 nm and offset from the true dislocation core also of approximately 1 nm, weak-beam dark-field (WBDF) imaging most closely satisfies these requirements [173]. Additionally, a background can be collected from the images, consisting of thickness fringes averaged over the tilt series, and subtracted from the individual images, further reducing varying diffraction contrast effects, i.e. those effects not associated with a lattice defect [11]. However, WBDF imaging requires maintaining a very precise excitation error, which becomes problematic in samples with high strains as lattice rotations can cause local deviations from the WBDF condition.

Various STEM imaging techniques have also been used for image acquisition, including bright-field (BF) [164, 167], low angle annular dark-field (ADF) [166, 174], medium angle ADF (MAADF) [175], and HAADF. Barnard et al. showed that MAADF STEM imaging provides a compromise between HAADF imaging where thermal diffuse scattering dominates the image and BF and ADF imaging where the contrast is dictated by only one or two diffracted beams. MAADF imaging allows dislocations to be clearly resolved and relaxes the need to maintain a
consistent excitation error during image acquisition as multiple diffracted beams are detected simultaneously. Additionally, dynamic scattering effects such as thickness fringes are less apparent than in BF or ADF as electrons are collected only from higher scattering angles, opening the possibility of using tomography software for automated image acquisition with user input needed only for refocusing [175]. The reduction of these additional scattering effects comes at the cost of lower imaging resolution – the apparent dislocation width is approximately 10-20 nm.

Ease of application and stronger contrast make BF imaging an appealing alternative to WBDF. However, the apparent dislocation width is approximately 10 nm, making it difficult to image dense dislocation networks, and unwanted diffraction effects are more apparent than in either WBDF or STEM images. BF TEM imaging was used in the study on dislocation interactions with irradiation induced defects by Kacher et al. [172]. In that application, sample thickness and high levels of local curvature inhibited the use of WBDF imaging.

The selection of which imaging technique to use ultimately depends on the interaction and dislocation structure to be characterized. Samples with high dislocation density where multiple dislocations overlap in a single projection generally require WBDF to resolve the structure. When large elastic strains or crystal rotations are present in an area of interest, STEM-based techniques may be required as the diffraction condition can be somewhat relaxed. BF TEM imaging provides a compromise between WBDF and STEM-based imaging as it provides a higher resolution than the STEM-based techniques and is not as sensitive to variations in diffraction conditions as WBDF imaging.

Further developments to dislocation tomography by Liu et al. have sought to reduce the number of images needed to reconstruct a useful tomogram, overcome information loss from the
invisibility criteria of dislocation imaging, and access quantitative information by including a reference frame in the tomograms [169]. Conventional tomography applications utilize images acquired over 120-140° of tilt. Double tilt stages used to characterize crystalline materials, however, are often limited to approximately 80° tilt, greatly reducing the resolution of the resultant tomogram. Liu et al. overcame challenges associated with a limited tilt range by developing a technique where the reconstructed tomogram is used as a basis for construction of a 3D dislocation model. In order to construct the 3D model, prior knowledge of dislocation behavior, specifically that dislocations must terminate at interfaces or as loops and that they are line defects, are used to aid in high fidelity manual tracing of the individual dislocations. Diffraction information collected during acquisition of the tilt series is used to place a coordinate system in the 3D dislocation model, generally in the form of a Thompson tetrahedron in FCC materials. Comparison of the Thompson tetrahedron coordinate system to reconstructed stacking-fault tetrahedra has shown that the coordinate system can be oriented to within 3° of the actual crystal orientation [169].

Liu et al. demonstrated that the construction of a 3D dislocation model can be used to overcome the g●b invisibility challenge [176]. Two tilt series can be collected at a single area using different diffraction vectors selected such that all defects are captured in the images. Each series can then be used to construct a 3D model of the interaction which can then be combined to form a single model containing all of the defects. Liu et al. used this method to produce a model of dislocations interacting with Co-rich particles in a Cu-Co system, shown in Figure 2.11 [176]. The model highlights the dislocations that would be absent from the image if only one of the diffraction vectors was used. Small half loops are seen attached to Co particles from previous dislocation interactions. It is apparent that these half loops do not undergo cross-slip events but
remain on a single plane. Also evident in the model is particle shearing from twinning dislocations propagating through the matrix. This was of interest as the particles were normally not shearable by dislocations.

![Model of dislocation/particle interactions in a Cu-Co system. The particles are represented by purple spheres, with yellow half-spheres indicating where previous shearing events took place. The model was constructed from two different tomograms reconstructed using different diffraction conditions to ensure that all defects were present [176].](image)

**Figure 2.11.** Model of dislocation/particle interactions in a Cu-Co system. The particles are represented by purple spheres, with yellow half-spheres indicating where previous shearing events took place. The model was constructed from two different tomograms reconstructed using different diffraction conditions to ensure that all defects were present [176].

In summary, previous work has demonstrated the feasibility of dislocation tomography for a variety of systems. Simple as well as complex interactions have been characterized in 3D, providing information on the spatial relations of dislocations at a resolution not achievable using conventional tomography. The additional step of using a reconstructed tomogram as the basis for
a 3D-dislocation model developed by Liu et al. further expanded the capabilities of dislocation tomography by making the interactions easier to resolve and, through the further addition of a coordinate system, quantifiable. This quantification includes the straightforward identification of dislocation line directions and slip planes, allowing full system characterization. However, while the feasibility of dislocation tomography has been well demonstrated, the practical applications are as yet limited to only a few types of interactions. There are still many areas that could benefit from the application of dislocation tomography. Additionally, situations where it is not possible to maintain a consistent diffraction condition while acquiring images for a tilt series are not currently resolvable by dislocation tomography. These situations include most dislocation/grain boundary interactions due to the crystal rotation across the boundary as well as in situ experiments that require a single tilt stage.

2.5 DISSERTATION OBJECTIVES

The objective of this dissertation is to increase understanding of dislocation interactions through the development and application of a novel technique allowing the combination of in situ TEM deformation experiments with electron tomography. The focus of the study will be on two different material systems, austenitic stainless steel and α-Ti, and on two different types of dislocation interactions, interactions with other dislocations and interactions with grain boundaries.

The investigation of austenitic stainless steel will center on dislocation/grain boundary interactions at elevated temperature. A prime objective of the study is to determine if criteria such as those developed by Lee et al. for predicting which system at a boundary will be activated
during dislocation/grain boundary interactions at room temperature [9] are applicable at elevated temperatures. Also, the experiments presented here will seek to expand the applicability of the criteria dictating dislocation/grain boundary interactions to interactions involving partial as well as perfect dislocations and exploring the variations of the response at the grain boundary from the impingement of multiple dislocations. That is, to discover how dislocation/grain boundary interactions evolve with time.

As Σ3 boundaries are of special interest to the materials engineering community, additional attention will be given to them. This will include a focus on understanding how dislocation interactions with Σ3 boundaries vary as a function of the Burgers vector of the incoming dislocations, with two broad categories being those dislocations for which the boundary plane represents a conjugate slip plane and those for which it does not.

As there have been few studies on grain boundary/dislocation interactions in HCP systems, this study will focus on verifying the applicability of the criteria developed by Lee et al. [9] for dislocation/grain boundary interactions in α-Ti at room temperature and, if necessary, developing new criteria to predict such interactions. Dislocation interactions will also be investigated in the grain interior to further understand dislocation generation and entanglement. Such interactions have been characterized after ex situ deformation [125, 177], but no published studies showing dislocation generation in α-Ti during in situ TEM deformation yet exist.

Transmission electron microscopy will be the primary tool used to investigate the dislocation interactions. This includes well established techniques such as Burgers vector characterization and in situ room and elevated temperature deformation, as well as the more recently developed technique of diffraction contrast electron tomography for defect
characterization. A methodology will be presented that allows diffraction contrast electron tomography, currently limited to post mortem investigations of material deformed ex situ, to be applied during interrupted periods of in situ TEM deformation experiments, providing quasi-4D characterization of dislocation interactions.
CHAPTER 3

EXPERIMENTAL METHODS

3.1 SAMPLE PREPARATION

A main aim of this dissertation is the development of 4D dislocation studies and so the majority of the deformation experiments were carried out in situ in the TEM. Ex situ deformation of bulk samples was also used to initiate dislocation interactions for comparison to confirm that the dislocation dynamics observed in situ were not dictated by thin film effects.

3.1.1 304 stainless steel

TEM samples were sheared from austenitic 304 stainless steel sheets to stage specified dimensions, 3 mm disks for ex situ experiments and 11.5 x 2.5 mm for in situ deformation, and ground to a thickness of approximately 150 µm. Bolt holes for connection to the in situ deformation stage were drilled 9 mm apart. The samples were then annealed at 1060°C for 30 minutes to remove deformation and increase the grain size. After annealing, the 3 mm disks were slightly kinked to introduce a low level of dislocation activity. Electron transparency was achieved using a twin jet polisher with a 6% perchloric acid, 39% butanol, and 55% methanol electrolyte with the solution kept at -15°C and an applied voltage of 30 V.
3.1.2 Titanium

Sheets of Ti were first cut from a block of commercially pure Ti using a low speed diamond saw. TEM samples were then sheared from the sheets to stage specified dimensions, 3 mm disks for *ex situ* experiments and 11.5 x 2.5 mm for *in situ* deformation, and ground to a thickness of approximately 150 µm. Bolt holes for connection to the *in situ* deformation stage were drilled 9 mm apart. The samples were then annealed at 830°C for 1 hr. A thin oxidation layer was formed during the annealing and was subsequently removed by lightly grinding the samples using silicon carbide paper. The 3 mm disks were slightly kinked to introduce a low level of dislocation activity. Electron transparency was achieved using a twin jet polisher with a 6% perchloric acid, 39% butanol, and 55% methanol electrolyte with the solution kept at -30°C and an applied voltage of 16.5 V.

3.2 INSTRUMENTATION AND CONVENTIONAL ELECTRON MICROSCOPY

Unless stated otherwise, the electron microscopy was performed using a JEOL 2010 LaB₆ microscope operating at an accelerating voltage of 200 kV. TEM stages used in this study include a Gatan model standard double-tilt stage allowing approximately ±40° α-tilt and ±30° β-tilt and a displacement controlled Gatan model deformation/heating stage that allows samples to be deformed under tension *in situ* at displacement rates up to 1 µm/s and at temperatures up to 1000°C, stage images are shown in Figure 3.1. Due to inconsistent contact between the heating element and the sample, the actual sample temperature can be as low as 150°C below the reported value. The tilt range is limited to ±35° over a single tilt axis.
Dislocation imaging and analysis was performed in two-beam dynamical BF mode. The dislocation Burgers vectors were determined using the $g \cdot b = 0$ invisibility criterion. When using the double-tilt stage, in order to find a sufficient number of $g$-vectors to characterize the dislocations a low-index zone axis was identified in diffraction space and images of the dislocations were collected using the available vectors. The sample was then tilted along a Kikuchi band until a second low-index zone axis was identified. Again, images were taken using the available $g$-vectors. Additional zone axes were similarly tilted to as needed. As the deformation stage is limited to a single axis of tilt, only those $g$-vectors which lie along the tilt line could be collected. In the majority of cases, this was sufficient to uniquely identify the dislocation Burgers vectors.

Figure 3.1. Photos of the double-tilt stage (a) and deformation/heating stage (b) with samples loaded. Images courtesy of Jamey Fenske.
3.3 *In situ* TEM Heating/Straining Experiments

*In situ* combined heating and straining experiments performed in the TEM followed one of three different paths: room temperature straining, room temperature followed by elevated temperature straining, or elevated temperature straining. All experiments were carried out using the Gatan model heat/strain stage under two-beam dynamical BF imaging conditions. Basic room temperature straining was carried out by first loading the sample into the microscope and identifying likely areas of interest. The sample was deformed at a displacement rate of 1 µm/s until dislocation motion was observed, generally at a crack nucleation from the center hole. Once dislocation motion was observed, the displacement was arrested and the dislocation interactions during foil relaxation were captured using the CCD camera at a frame rate of 10-33 frames per second. The sample was strained periodically to induce further dislocation motion. Burgers vector characterization was performed early in the interaction while the defect density was still relatively low and was repeated as new dislocation systems nucleated. Elevated temperature straining experiments were performed in an identical manner with the exception that the sample temperature was raised to a nominal temperature of 400°C prior to sample loading.

A combined room temperature/elevated temperature experiment was designed to directly compare dislocation interactions at different temperatures and identify potential thermally activated processes. Initially, a sample was strained at room temperature until dislocation activity was observed, identical to the procedure outlined for room temperature straining experiments. A grain boundary/dislocation interaction was observed for a sufficient time period to identify the initial grain boundary response. The sample was then unloaded, the temperature raised to 400°C, and the load reapplied, re-initiating dislocation motion. The same dislocation/grain boundary
interaction could then be observed with all variables kept near constant except for the environment temperature.

3.4 DIFFRACTION CONTRAST ELECTRON TOMOGRAPHY

This section outlines the procedure for constructing a 3D dislocation model from a tilt series acquired using a double-tilt stage. For strategies employed when the sample is loaded in a single tilt stage, such as a heat/strain stage, the reader is referred to section 3.5. Unless stated otherwise, two-beam dynamical BF imaging conditions were used in all cases presented here, but the procedure outlined is general to all diffraction contrast imaging conditions (DF, WBDF, STEM ADF…).

3.4.1 Acquisition, alignment, and reconstruction

Proper diffraction-contrast electron tomography generally requires a dual axis tilt stage, either a double-tilt stage with α and β-tilt capabilities or a tilt/rotate stage. A Gatan model double-tilt stage was used in this study. To obtain a tomogram, a series of images was first collected over the maximum tilt range allowed by the stage, approximately ±40° depending on the specimen z-height, while maintaining the diffraction conditions, both the g-vector used and the excitation error s, constant. This is possible by aligning the tilt axis precisely normal to a crystallographic plane. Practically this was accomplished identifying a Kikuchi band in diffraction space and tilting along that band. To minimize precession effects and for ease of acquisition, the Kikuchi band should be running near perpendicular to the α-tilt axis with β-tilt
used to correct any misalignment between the \( \alpha \)-tilt axis and the selected crystal plane normal. Images were collected at regular tilt intervals with the tilt interval selected in consideration of resolution requirements (see Figure 2.9) and time constraints. Images were refocused during the tilt series acquisition using \( z \)-height adjustments primarily as changes in the objective lens focus can affect the image magnification, introducing errors into the reconstruction algorithms.

Tomography of grain boundaries required special care as the diffraction conditions generally vary across different grain orientations. A two-beam BF condition was maintained in neighboring grains simultaneously only in special cases where the grains shared a common plane, such as is the case with the shared \{111\} between coherent \( \Sigma 3 \) boundaries. In this case, the tilt axis was aligned with the boundary plane normal. If no shared plane exists as is encountered when viewing random high angle boundaries, only one grain can be kept in a two-beam condition throughout the tilt range and further measures such as those described in section 3.5.4 were taken to reconstruct a tomogram containing the dislocations in both grains.

Once acquired, the tilt series was aligned and the tilt axis was identified in the image using EM3D software (http://em3d.stanford.edu). Fiducial markers were identified in the images by adjusting contrast gradient and threshold levels. The most common fiducial marker used was the intersection points of dislocations with the foil surfaces as they were generally easily identifiable between images. Manual identification of fiducial markers was also needed to compensate for strong contrast fluctuations due to bend contours. Once identified, the fiducial markers could be aligned such that image features translated smoothly when tilted images were superimposed on each other. Multi-beam images, such as are encountered near zone axes during image acquisition, could not be aligned and so were removed from the tilt series. Absent images
were automatically accounted for in the software. Using a weighted-back projection algorithm, the tilt series was reconstructed into a tomogram, again in EM3D.

### 3.4.2 3D dislocation model construction

Limitations in the tilt range and numbers of images collected as well as precession effects due to using two axes of tilt led to smearing and poor feature resolution in the tomogram visualizations. However, as dislocations are known to be line defects and terminate only at interfaces or as loops, the information contained in the tomogram is sufficient to construct a high fidelity 3D dislocation model. Tomograms were visualized and dislocation models were constructed with the aid of UCSF Chimera, a 3D visualization and image manipulation software developed specifically for electron tomography applications [178]. The 3D information was loaded into Chimera from EM3D and the brightness and contrast was adjusted to allow maximum resolution of the defects. Dislocations were manually traced with the assumed location being the highest density portion of the visualized dislocations. Correct placement of the traced dislocations required iterating between different viewing angles and modifying the location of the traced dislocations accordingly.

A key benefit of the 3D dislocation model is the ability to place a coordinate system in the model, corresponding to crystal lattice planes and directions and allowing straightforward identification of dislocation slip planes and line directions. These were in the form of a tetrahedron for FCC stainless steel samples, labeled according to the Thompson tetrahedron convention (Fig. 3.2), and a small hexagonal lattice for the Ti samples (Fig. 3.3). During acquisition of the tilt series, multiple zone axis diffraction patterns were also collected with
accompanying BF images. The constructed dislocation models were tilted such that they aligned with a BF image. The corresponding zone axis diffraction pattern could then be used to identify the crystal plane normals, and the coordinate system placed in the model was rotated accordingly. Generally, three independent planes were sufficient to uniquely place the coordinate system.

Figure 3.2. Unfolded version of a Thompson tetrahedron showing conventional labeling of planes and directions in an FCC lattice.
Figure 3.3. Coordinate systems used for direct identification of slip planes and line directions in 3D dislocation models. The tetrahedron, used for FCC materials is labeled according to the Thompson tetrahedron convention (Fig. 3.2). The hexagonal lattice is used for HCP materials and can be customized for varying c/a ratios.

The 3D dislocation model was also coupled with the Burgers vector analysis to provide a complete visual characterization of the system. Dislocations were color coded according to Burgers vector and when two grains were present in the tomographic volume, separate coordinate frames were added for each grain. Thus, in a single graphic information on dislocation character, slip planes, grain boundary planes, and approximate misorientation between grains is readily available. The 3D model also allows detailed study of dislocation shape and proximity to other defects as it could be viewed from any arbitrary vantage point, providing information on, for example, cross-slip behavior and loop formation.
3.5 4D ANALYSIS

Traditional TEM analysis is limited to 2D projections at a single instance in time, forcing the operator to assume certain dislocation distribution and behavior between at best a before and after snapshot of complex interactions. The preceding sections outlined approaches to overcome both the projection effect of electron micrographs and their static nature. Currently in situ deformation and tomography techniques for defect analysis are employed only separately, leaving the choice of sacrificing either the dynamic information or the 3D spatial distribution. A main aim of this work has been to combine the 3D analysis with dynamic in situ TEM experiments to provide 4D analysis of dislocation interactions. Challenges associated with combining in situ TEM deformation with electron tomography stem from the conventional assumptions of electron tomography and the limitations of the majority of in situ TEM deformation stages. The conventional approach to electron tomography generally requires images collected over 120-140° of tilt with an image collected at one degree intervals. Additionally, these images must satisfy the projection requirement. That is, the image contrast must vary linearly with only one variable besides the specimen thickness. The preceding sections showed that the projection requirement could be satisfied if the diffraction conditions were kept constant during image acquisition by aligning the stage tilt axis with a crystal plane normal. It was also shown that a high fidelity 3D model of a dislocation interaction could be constructed using prior knowledge of dislocation behavior, specifically that dislocations must terminate at interfaces or as loops.

Maintaining consistent diffraction conditions over a wide tilt range for electron tomography generally requires a dual axis tilt stage in order to keep the tilt axis aligned with a single crystallographic plane. This becomes problematic when attempting to couple electron
tomography with \textit{in situ} TEM deformation as most deformation stages for the TEM are limited to a single axis of tilt. Multiple strategies have been devised to bypass the requirement for maintaining consistent diffraction conditions and thus achieve 4D characterization of dislocation interactions. Their applicability is dependent on sample character, dislocation density, and resolution requirements. These strategies are described below, with the first sections of the results chapter dedicated to exploring the applicability of each approach through various examples. The advantages and disadvantages of each method will also be further discussed in the results section.

3.5.1 \textit{Pre-aligned specimen}

Using a pre-aligned sample is the most basic approach to 4D characterization and could have application when characterizing single crystal or bicrystal samples. During preparation, a sample can be cut along a known crystallographic plane, which is then aligned with the stage tilt axis such that when loaded into the TEM the $\alpha$-tilt axis is aligned with a crystal plane normal. In this way, only a single axis of tilt is needed to maintain consistent diffraction conditions over a wide tilt range.

3.5.2 \textit{Aligning using colloidal gold fiducial markers}

Colloidal gold fiducial markers broaden the applicability of 4D analysis beyond single and bicrystal samples. In this method, a drop of colloidal gold in deionized water is allowed to dry on the electron transparent portion of a sample. In the TEM, an area of interest is identified
which includes both a dislocation interaction of interest and a sparse distribution of colloidal gold particles. A tilt series is collected disregarding the requirement to maintain a consistent imaging condition, *i.e.* a tilt series can be collected using a single tilt deformation stage before, during, and/or after *in situ* deformation in the TEM. Once a tilt series is collected, the dispersed colloidal gold particles are used as fiducial markers, facilitating image alignment even with strong contrast fluctuations of the base crystal.

3.5.3 In situ straining combined with FIB lift-out

In polycrystalline materials where the crystal planes cannot be pre-aligned with the stage tilt axis and due to complex dislocation configurations a high level of detail is needed from the tomogram, *in situ* straining in the TEM can be coupled with *post mortem* focused ion beam (FIB) machining for subsequent tomographic investigation using a dual-axis tilt stage. Using a combined approach, the dynamic dislocation interactions are first observed during *in situ* TEM deformation, providing information such as the sequence of events leading to an observed dislocation structure. An area of interest is then identified and the sample is loaded into the FIB. The same location is then found using secondary electron imaging, the surface slip traces can facilitate identifying the area, and a section of the sample is cut from the foil using ion milling. This section is then attached to a copper grid using a platinum weld, which can be loaded into a dual-axis tilt stage. At this point, diffraction contrast electron tomography is used as previously described to obtain a tomogram of the interaction. The 3D dislocation structure can be correlated with and compared to the previously recorded dislocation dynamics.
3.5.4 Aligning with digital fiducial markers

Similar to construction of a 3D dislocation model, prior knowledge of dislocation behavior and diffraction contrast theory can be used to reconstruct a tomogram from a sparsely populated tilt series with strong contrast fluctuations. As already described, if the tilt axis is not aligned with a crystallographic plane during acquisition of a tilt series, as is most often the case when imaging a polycrystalline sample in a single tilt holder, dislocation contrast will vary strongly between images. However, in many instances the location of the dislocations can be identified manually. To aid in the alignment and reconstruction of such a tilt series, digital fiducial markers can be placed in the collected image at the ends of the dislocations, corresponding to where the dislocation intersects the free surface. Once reconstructed, the fiducial markers appear as spheres marking the intersection point in the tomogram visualization. These spheres can be connected manually to construct a 3D dislocation model which, when only straight dislocations are involved, contains all the needed information on dislocation line direction and slip plane, allowing the full characterization of a dislocation interaction.

When imaging curved dislocations, a high fidelity dislocation model can still be constructed granted the dislocations are in clear contrast in at least two images separated by a large angular tilt. As the dislocations are known to be line defects, views from only two angles are needed to uniquely identify the location in space of a point on a dislocation. In the reconstructed tomogram, the spheres from the digital fiducial markers make the slip plane readily apparent and the morphology of the dislocations can be iteratively fitted to the true dislocation shape by viewing the tomogram from different angles and fitting the 3D dislocation model to the visualized dislocation structures. This is similar in principle to the use of stereographic projections with the added advantages of more widely spaced viewing angles, increasing the
accuracy of the technique, and the digital form of the display which allows inclusion of a coordinate system and inspection of the data from any arbitrary viewing angle.

3.5.5 *Comparison to iterative reconstruction techniques*

A class of tomography reconstruction methods known as iterative reconstruction techniques has been increasing in popularity recently due to the promise of higher fidelity reconstructions at the cost of higher computational power requirements. As the name suggests, these techniques follow an iterative procedure where the tomogram, once constructed, is compared to the individual projections originally acquired in the tilt series [157]. Adjustments are then made to the tomogram to ensure that the tomogram truly reflects the projected volume, theoretically increasing the resolution and fidelity of the reconstructed tomogram. One such variant of the iterative techniques known as the SIRT method (simultaneous iterative reconstruction tomography) [162] was used to reconstruct tilt series aligned using EM3D for direct comparison to the weighted back projection technique. The version used is available as a plugin through ImageJ known as TomoJ [179]. The aligned images were loaded into TomoJ and reconstructed using five iterations as the set number for tomogram reconstruction.

3.6 GRAIN BOUNDARY CHARACTERIZATION

Identifying coherent $\Sigma 3$ twin boundaries in FCC materials is a straightforward process where the sample is tilted to a zone axis such that the twin plane normal is perpendicular to the
electron beam. The twin plane is then given by the g-vector which is parallel to the twin plane normal. This g-vector will be a mirror plane in the diffraction pattern across which the other diffraction spots are reflected. An example of a characterized twin boundary is shown in Figure 3.4.

Figure 3.4. Characterization of a twin. The diffraction pattern shows the (111) mirror plane with the associated diffraction vector at a 90° angle to the twin plane. The beam direction is given and the diffraction spots are labeled with T indicating those spots diffracting from the twinned region.

For all other grain boundaries, EBSD analysis in the SEM was used for post mortem characterization after the TEM analysis. The microscope used was a JEOL 7000F SEM operating at 20 kV. No further sample preparation beyond that done for TEM analysis was needed. HKL
software was used for the data collection and OIM TSL analysis software was used for data
display and interpretation. The corresponding area to that investigated in the TEM was found by
first taking an area scan over the entire electron transparent region. Similar grain morphologies
were identified in the scan, allowing correlation with the TEM data. Grain boundary information
from the EBSD analysis was in the form of an angle/axis pair (Fig. 3.5). The characteristic
transformation matrix \( Q \) can be constructed from the normalized rotation axis \([uvw]\) and rotation
angle \( \theta \) according to [180]:

\[
Q = \begin{bmatrix}
1 + (1 - \cos \theta)(u^2 - 1) & -w \sin \theta + uv(1 - \cos \theta) & v \sin \theta + uw(1 - \cos \theta) \\
v \sin \theta + uw(1 - \cos \theta) & 1 + (1 - \cos \theta)(y^2 - 1) & -u \sin \theta + vw(1 - \cos \theta) \\
-v \sin \theta + uw(1 - \cos \theta) & u \sin \theta + vw(1 - \cos \theta) & 1 + (1 - \cos \theta)(w^2 - 1)
\end{bmatrix}
\]

For the Ti data, an additional step must be taken before construction of the transformation matrix
to convert the vector information from 4-index notation to Cartesian 3-index notation. This was
done using the transformation:

\[
\begin{bmatrix}
h' \\
k' \\
l'
\end{bmatrix} = \begin{bmatrix}
\frac{3}{2} & 0 & 0 & 0 \\
\sqrt{3} & \sqrt{3} & 0 & 0 \\
\frac{2}{2} & 0 & 0 & c/a
\end{bmatrix} \begin{bmatrix}
h \\
k \\
l
\end{bmatrix}
\]

where \( c/a \) is specific to the material lattice constants. For Ti, a \( c/a \) value of 1.587 was used.
Figure 3.5. Characterization of a grain boundary using EBSD. The HCP lattice is represented by hexagonal prisms in each grain, with the two grains being related by a $70^\circ$ rotation about the $\langle 1010 \rangle$ axis. Black pixels are points that were not indexed, most of which were due to pattern mixing at the boundary. The color coding legend is given in the upper right hand corner.

Once constructed, the transformation matrix was used to represent all lattice information in one common reference frame, generally the crystal frame of the grain containing the incoming dislocations. This was used to represent the Burgers vectors of dislocations in neighboring grains in a common reference frame, allowing direct subtraction to calculate the Burgers vector of the
residual grain boundary dislocation, and to express the stress tensor in the coordinate system of a neighboring grain. For vectors, the coordinate transformation is:

\[ \mathbf{u}_{\text{out}} = Q^{\text{in} \rightarrow \text{out}} \mathbf{u}_{\text{in}} \]

and for second order tensors:

\[ \mathbf{M}_{\text{out}} = Q^{\text{in} \rightarrow \text{out}} \mathbf{M}_{\text{in}} Q^{\text{in} \rightarrow \text{out}}^T \]

where \( \mathbf{u} \) is any vector, \( \mathbf{M} \) is any second order tensor, and the subscripts \( \text{in} \) and \( \text{out} \) refer to two different coordinated systems related by the transformation matrix \( Q^{\text{in} \rightarrow \text{out}} \). \( T \) indicates the matrix transpose.

Special care was taken to ensure that the TEM data and the EBSD data were consistent across the grains. Generally, this was done manually by checking first that the \( hkl \) as given by the EBSD analysis corresponded to the foil normal characterized in the TEM. Additionally, it was verified that zone axes characterized in neighboring grains at similar stage tilts could be mapped onto each other using the transformation matrix constructed from the TEM data.

An alternative approach to characterization of the grain boundary misorientation that avoids the need to maintain consistency between the EBSD data and the diffraction patterns collected in the TEM was also used. In this approach, the orientations of the two individual grains were determined separately by using diffraction patterns collected in the TEM to identify the location in Kikuchi space where the stage tilt was zero. This included both translation in Kikuchi space as well as rotation. Simulations of Kikuchi space available in the OIM DC software package aided in navigating Kikuchi space as it allows free translation and rotation to any diffraction position (Fig. 3.6). The orientation was output by the software in terms of the
Euler angles \((\phi_1, \Phi, \phi_2)\). Orientation matrices, equivalent to transformation matrices for rotations between the crystal and the sample frames, were constructed using the following equation [180]:

\[
Q^{S\rightarrow C} = \begin{bmatrix}
\cos \phi_1 & \sin \phi_1 & 0 \\
-\sin \phi_1 & \cos \phi_1 & 0 \\
0 & 0 & 1
\end{bmatrix}
\begin{bmatrix}
1 & 0 & 0 \\
0 & \cos \Phi & \sin \Phi \\
0 & -\sin \Phi & \cos \Phi
\end{bmatrix}
\begin{bmatrix}
\cos \phi_2 & \sin \phi_2 & 0 \\
-\sin \phi_2 & \cos \phi_2 & 0 \\
0 & 0 & 1
\end{bmatrix}
\]

\(Q^{in\rightarrow out}\) was then constructed according to:

\[
Q^{in\rightarrow out} = Q^{S\rightarrow C}_{out} Q^{S\rightarrow CT}_{in}
\]

Generally, the two methods for grain boundary characterization were used in tandem, with one being used to verify the other. In all cases, agreement between the two methods was within 5° for determination of the misorientation angle across a grain boundary.
Figure 3.6. Screenshot showing the OIM DC software. An area of simulated Kikuchi space is shown for α-Ti in the upper left. The crystal lattice structure and the Euler angles corresponding to the location in Kikuchi space are shown in the upper right and lower left, respectively. The pole figure in the lower right displays the orientation visually as a red dot.
3.7 ESTIMATING RESOLVED SHEAR STRESS

In order to investigate the effects of the local resolved shear stress on the interaction, a simple ‘super dislocation’ model was developed. In this construct, the incoming dislocations were treated as a single dislocation at the grain boundary with the same Burgers vector and line direction as that of the incoming dislocations. The local stress state was calculated by summing the contributions from the edge and screw components of the super dislocation, given by [181]:

\[
\sigma_{edge} = \begin{bmatrix}
-D_y \frac{(3x^2 + y^2)}{(x^2 + y^2)^2} & D_x \frac{(x^2 - y^2)}{(x^2 + y^2)^2} & 0 \\
D_x \frac{(x^2 - y^2)}{(x^2 + y^2)^2} & D_y \frac{(x^2 - y^2)}{(x^2 + y^2)^2} & 0 \\
0 & 0 & \frac{2Dyv}{x^2 + y^2}
\end{bmatrix}, \quad D = \frac{Gb}{2\pi(1 - \nu)}
\]

\[
\sigma_{screw} = \begin{bmatrix}
0 & 0 & -\frac{Gb}{2\pi} \frac{y}{(x^2 + y^2)} \\
0 & 0 & \frac{Gb}{2\pi} \frac{x}{(x^2 + y^2)} \\
-\frac{Gb}{2\pi} \frac{y}{(x^2 + y^2)} & \frac{Gb}{2\pi} \frac{x}{(x^2 + y^2)} & 0
\end{bmatrix}
\]

\[
\sigma_{total} = \sigma_{edge} + \sigma_{screw}
\]

where \(\nu\) is Poisson’s ratio and \(G\) is the shear modulus. The \(z\)-direction is defined to be parallel to the dislocation line direction and the \(x\)-direction, for edge dislocations, is defined as parallel to the Burgers vector.
The total stress was then transformed into the neighboring grain reference frame using the
appropriate transformation matrix. The resolved shear stress was calculated at a position one
arbitrary unit from the boundary in the direction of dislocation motion using:

\[ \tau_{RSS} = \sigma \mathbf{m} \otimes \mathbf{n} \]

Where \( \mathbf{m} \) is the slip direction (parallel to the Burgers vector), \( \mathbf{n} \) is the slip plane normal, and \( \sigma \) is
the stress state from the super dislocation, \( \cdot \) denotes the tensor dot product and \( \otimes \) is the dyadic
product. Since only the relative resolved shear stress was needed, the calculations were
normalized by setting the maximum resolved shear stress equal to unity and scaling the resolved
shear stress on the other available slip systems accordingly. In the presentation of results, all
subscripts refer to the coordinate frame of the grain in which the vectors are given, with \( \text{in} \)
referring to the grain in which dislocations impacting the grain boundary reside and \( \text{out} \) to the
neighboring grain into which dislocations are emitted.
4.1 STRATEGIES FOR 4D ANALYSIS

This section provides examples of 4D analysis demonstrating the feasibility and applicability of the different strategies presented in section 3.5. Some of the experiments done were for feasibility testing only, and as such, analysis may be minimal.

4.1.1 Pre-aligned specimen

To demonstrate the feasibility of using a sample cut such that a crystallographic plane normal aligns with the stage tilt axis for 4D analysis, the dynamics of a grain boundary/dislocation interaction in austenitic stainless steel was coupled with a reconstructed tomogram of the interaction in a large grain polycrystalline sample. The \textit{in situ} movie shows that dislocations approach the grain boundary from both directions and are ejected from the boundary into both grains (Fig. 4.1). This lead to complex dislocation tangles forming near the grain boundary, including dislocation nodes and a high concentration of dislocations absorbed into the boundary plane. The dislocations were first ejected into grain 1 and only later into grain 2, see Figure 4.1 for grain identification. Already this highlights the importance of the data obtained through \textit{in situ} deformation as identification of dislocations impinging on and emitted from the
boundary would remain ambiguous if the investigation was limited to *post mortem* characterization of the interaction.

Grain 1 was oriented such that the (111) normal was parallel to the stage tilt axis which enabled a tilt series with minimal contrast fluctuations between images to be collected. Images were collected over a range of 55° with an image collected every degree. No crystallographic plane normal in grain 2 was aligned with the stage tilt axis, resulting in strong contrast variations between images. The tilt series was used to reconstruct a tomogram of the interaction, shown in Figures 4.2 and 4.3. As expected, the dislocations are clearly resolvable in grain 1. Details of the dislocation interactions such as the junctions formed from dislocation intersections can be identified, suggesting the possibility of using electron tomography to more accurately measure the stacking-fault energy. This is done by measuring the curvature of the nodes or from the radius of a circle within the node and currently requires a correction factor to account for projection effects [182]. Although the dislocations in grain 2 were visible only in some of the images collected in the tilt series, they are resolvable in the reconstructed tomogram. This suggests that the conventionally assumed requirements for electron tomography, that images must be collected at small tilt increments over a wide tilt range with minimal contrast fluctuations between them, do not necessarily hold true when reconstructing dislocation structures.
Figure 4.1. Dislocations interacting with a high angle grain boundary in 304 stainless steel during in situ deformation in the TEM at 400°C. Top two images show evolution of the dislocation structure with straining with the experiment time given in each image. The bottom image shows a higher resolution image of the interaction with a dislocation node selected for enlarged view. Arrows show direction of dislocation motion and grains are numbered ‘1’ and ‘2’ for reference in the text.
Figure 4.2. Select images from the tilt series collected of the interaction shown in Fig. 4.1. The tilt angle is given in each image.
Due to the analysis being limited to a single $g$-vector, traditional $g\cdot b$ techniques could not be used to characterize the dislocation Burgers vectors. The tomogram should allow identification of the slip plane, limiting the possible Burgers vectors to two, at which point dislocation simulation matching could potentially be used for Burgers vector characterization. As this experiment was done mainly to demonstrate the possibility of combining in situ TEM deformation with electron tomography, the Burgers vector characterization was not done.
4.1.2 Aligning using colloidal gold fiducial markers

Colloidal gold particles have the potential of providing fiducial markers for alignment of a tilt series that are not subject to the diffraction conditions of the underlying crystal. The feasibility of using colloidal gold particles as fiducial markers was demonstrated using a 3 mm Ti disk deformed *ex situ*. A drop of colloidal gold suspended in deionized water was placed on the sample after jet polishing and allowed to dry. In the TEM, an area of interest was identified where dislocations were seen entering into, propagating along, and exiting from a grain boundary. A tilt series was collected over an angular range of 80° using only α-tilt, which was then reconstructed into a tomogram in EM3D (Fig. 4.4a-b). As can be seen, the characterized area includes sparsely dispersed gold colloids as well as areas of dense conglomerations of gold. Only one axis of stage tilt was used to replicate the tilting capabilities of a standard TEM strain stage.

Two dislocation systems are seen exiting the grain boundary, both of which are clearly resolved in the tomogram, as is the grain boundary and extrinsic grain boundary dislocations (Fig. 4.4). The incoming dislocation system is not as clearly resolved, but is still of sufficient contrast to identify the slip plane. This is similar to what is seen in grain 2 of Fig. 4.3; that dislocations can be resolved three-dimensionally even when the tomogram is reconstructed using a sparsely populated tilt series composed of images with strongly fluctuating contrast. The tomogram suggests that the dislocations impinge at one point in the boundary and are emitted from the boundary at or near that same point, but also propagate along the boundary and exit at a separate point. That no single dislocation system is optimal is evident by the presence of at least two distinct systems being emitted into the neighboring grain. As the primary purpose of this
experiment was to test the feasibility of using colloidal gold particles as fiducial markers for 4D analysis, no further analysis was done to characterize the dislocations or grain boundary.

Figure 4.4. Tomography process using the colloidal gold technique. Example images from a tilt series are shown with the stage tilt angle indicated in each micrograph and arrows showing the assumed direction of dislocation motion. Numbered arrowheads in a) indicate
(Fig. 4.4 continued) a lone gold particle (1) as well as a dense conglomeration of gold (2). Views of the reconstructed tomogram and 3D dislocation model are also shown. The grain boundary is represented as a blue plane.

4.1.3 In situ straining combined with FIB lift-out

In situ TEM straining was combined with FIB lift-out for post mortem tomographic investigation of the dislocation structure in a study on dislocation interactions with irradiation induced defects [172]. The purpose of studying deformation of irradiated materials is to gain a fundamental understanding of the mechanisms responsible for the degradation of mechanical properties compared to unirradiated materials [183-185]. This knowledge is needed to inform and validate models as well as to direct innovative approaches to overcome degradation issues. Two areas central to this goal that were clarified in this study are the evolution of the dislocations in the cleared channel, including the formation of dislocation pileups at invisible obstacles, and the mechanism of channel widening. Understanding the details of these channels has implications beyond the mechanical response as they are also thought to have a critical role in the irradiation-assisted stress corrosion process [186].

The in situ TEM experiments were conducted at Argonne National Laboratory using a Gatan displacement controlled heat/strain stage. The samples were observed in a Hitachi 9000 TEM operated at 200 kV. Samples were heated to a nominal temperature of 400°C and irradiated in situ with 1 MeV Kr\(^+\) ions to a fluence of approximately 3x10\(^{17}\) ions m\(^{-2}\). In these electron transparent samples of stainless steel, the ion irradiation energy is such that the damage is primarily produced by secondary cascades and the resultant damage is in the form of Frank loops [187]. The samples were deformed incrementally at a maximum displacement rate of 1 \(\mu\)m s\(^{-1}\).
Results were recorded both as still frames and as video using a CCD camera at a recording rate of 10 frames per second.

*In situ* straining showed the formation of channels partially cleared of irradiation defects (Fig. 4.5). Complex dislocation interactions occurred in the partially cleared channels, leading to dislocation cross-slip and the formation of walls of dislocations bordering the channel boundaries. These walls were composed of dense tangles of dislocations not resolvable in the micrograph as well as elongated dislocations with line direction parallel to the channel direction. Dislocation sources were activated in the channel walls, emitting dislocation half-loops into the irradiated matrix. An example of half-loops extending from a channel wall and eventually coming in contact with the foil surface is highlighted in Figure 4.5a-d. Although the formation of the channels and dislocation walls was observed in real time, interpretation of the results was difficult due to the inability to resolve the spatial distribution of the tangled dislocations composing the channel boundary.

To resolve 3D distribution of the complex dislocation tangles, an area containing the microstructure of interest was identified, seen in the SEM image (Fig. 4.6a), and machined out using the FIB machining technique. The extracted sample was attached to a copper grid using a platinum weld and loaded onto a dual-axis tilt stage (Fig. 4.6b). Images were acquired in two-beam BF mode every degree over an angular range from -40° to +35° by tilting along a (200) Kikuchi band and maintaining a similar Bragg deviation parameter. A tomogram of the interaction was reconstructed, from which a 3D model of the dislocations was constructed.
Figure 4.5. Frames from a video taken during *in situ* deformation at 400°C of an irradiated stainless steel sample. The higher intensity band is a channel swept partially free of defects. Half loops emitting from the channel boundaries are indicated by arrowheads.
Figure 4.6. Secondary electron images of FIB machining process. An area was selected for FIB machining (a) and was then attached to a copper grid (b). An area of interest was identified by the slip traces visible in the SEM micrograph.

Figure 4.7 shows a TEM micrograph of the area selected for further tomographic investigation. FIB induced damage partially obscures the dislocations, though elongated dislocations running parallel to the channel direction, similar to those seen in during the in situ TEM deformation, are still visible, as well as tangles of dislocations near the channel boundaries. Figure 4.8a-c shows select views of the visualization of the tomogram constructed from the tilt series. Details of the dislocations within the channel are obscured in this image by the presence of a high density of small loops, which were not present in the original slip band prior to the FIB machining. That these features reside close to one surface is apparent in the tomogram profile (Fig. 4.8c), suggesting that the loops are from ion damage caused by the machining process. The defect cloud extends approximately 34 nm from the surface, which is in good agreement with TRIM (Transport of Ions in Matter) simulations which predicted a damage depth of 32 nm. Select views from the 3D model are shown in Figure 4.8d-f; the approximate location of the
channel-matrix boundary is indicated by the blue planes. Only the dislocations were traced to form the model; the FIB induced damage was ignored.

Figure 4.7. TEM micrograph showing the area selected for tomographic analysis. The approximate location of the channel boundaries are approximately defined by the dotted white line and examples of elongated dislocations lining the channel are indicated by arrowheads.

From the different viewing directions it can be surmised that the elongated dislocations lie on parallel but not coplanar planes outside the primary channel (highlighted in Figure 4.8d). Helical-shaped dislocations remain in the primary channel, suggesting past double cross-slip events. Double cross-slip from the boundary walls could account for the half-loops seen emerging from the channel boundaries during in situ deformation and could be an important
factor contributing to channel widening in irradiated materials. Also, the tomogram clearly shows that significant dislocation debris remains in the channel interiors after deformation. Kinks in the remaining dislocations are likely pinning sites from interactions with irradiation induced defects. In understanding deformation of irradiated materials, defect free channels are often treated as easy glide paths for the dislocations [188, 189]. The presence of pinned dislocations in the channel suggests that such an assumption is only partially accurate.
Figure 4.8. Select views of the tomogram visualization (a-c) and 3D dislocation model (d-f) of the interaction shown in Fig. 4.7. (c) displays a profile of the tomogram, highlighting the FIB machining-induced damage layer. (d) highlights in blue the elongated dislocation lining the channel boundary. A helical shaped dislocation is indicated in (e), as well as kinked dislocation in (f), likely due to pinning on an irradiation induced defect. The blue planes in (e) and (f) represent the approximate location of the defect free channel boundaries.
4.1.4 Aligning with digital fiducial markers

Two separate tests were conducted to first, demonstrate the ability to reconstruct a tomogram using a sparsely populated tilt series and second, reconstruct a tomogram from a tilt series collected using a single tilt stage with no physical fiducial markers. For the first test, an area in a 3 mm 304 stainless steel disk was located where dislocations were both entering and exiting a coherent $\Sigma 3$ grain boundary (Fig. 4.9). A tilt series of the interaction was collected by tilting along a (111) plane while maintaining a two-beam BF condition. This dataset is discussed in more depth in section 4.2.1 and is used here only as a verification test. 40 images were collected, one every other degree over a range of $80^\circ$, and were then used to reconstruct a

![Dislocations interacting with a coherent $\Sigma 3$ twin boundary in 304 stainless steel.](image-url)
tomogram of the interaction from which a 3D dislocation model was constructed (Fig. 4.10). The tilt series was reprocessed by placing markers at each end of the dislocations (Fig. 4.11a) and the TEM micrograph was deleted from the image, leaving only the markers (Fig. 4.11b). The markers were aligned and reconstructed using EM3D (Fig. 4.11c), and the spheres in the resultant tomogram were connected to recreate the dislocation configuration of the material (4.11d). The reconstruction process was repeated for both the original micrographs and the fiducial images using progressively fewer images for the reconstruction. The resulting tomograms are compared in Figure 4.12, illustrating the resolution degradation as the number of projections used in the reconstruction decreases. Assuming the angle between the dislocations from the original tomogram to be most accurate, the variation in measured angle can then be used as a quantitative measure of the quality of reconstruction (Table 4.1). As can be seen, the fiducial method suffers much less from reconstruction degradation than the micrographic reconstruction, with as few as 10 projections providing sufficient resolution to determine line direction to within 12° of the measured line direction determined using all 40 projections.
Figure 4.10. 3D dislocation model (a) and visualization of the tomogram (b) of the dislocation/grain boundary interaction shown in Fig. 4.9.
Figure 4.11. Digital fiducial method for constructing a 3D dislocation model. In a TEM micrograph (a) the dislocations are identified and marked with a circular fiducial marker at either end (b). The underlying micrograph is then deleted (c). This is done for each image in a tilt series, which is then used to reconstruct a tomogram (d). The fiducial markers, spheres in the tomogram, are connected to construct a 3D dislocation model (e).
Figure 4.12. Comparison of traditional diffraction contrast electron tomography methods (left) and the digital fiducial method (right) when a decreasing number of images is used in the reconstruction. The number of images is given in the left column.
Table 4.1. Measured angle between slip systems in the dislocation models constructed using a decreasing number of images.

<table>
<thead>
<tr>
<th>Number of images</th>
<th>No image processing</th>
<th>Fiducial method</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>71°</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>60°</td>
<td>67°</td>
</tr>
<tr>
<td>10</td>
<td>Lines could not be identified</td>
<td>59°</td>
</tr>
</tbody>
</table>

To explore the possibility of using the fiducial marker technique to reconstruct tilt series composed of images with strongly varying contrast, as is generally encountered when using a single-tilt holder, a stainless steel sample was loaded into the TEM and deformed lightly at room temperature using the heat/strain stage. An area of dislocations impinging on a grain boundary was identified (Fig. 4.13). Perfect dislocations were seen entering the grain boundaries, resulting in pairs of partial dislocations being emitted from the boundary and a large buildup of elastic strain in the boundary itself, evident by the strong bend contours. Further straining of the sample did not result in dislocation activity. However, the direction of dislocation motion is readily apparent by the presence of slip traces. There are also unrelated dislocations near the grain boundary originating from a different interaction.
A series of images of the interaction was collected at one degree intervals over a tilt range of 57°. As the tilt axis was not aligned with a crystallographic plane, the diffractions conditions varied strongly between images in the tilt series. Digital fiducial markers were manually placed at the ends of dislocations, which were then used to align and reconstruct the images in EM3D. In this case, the underlying micrograph was not deleted as it allowed identification of the boundary plane in the reconstructed tomogram. The visualization of the tomogram as well as the 3D dislocation model are displayed in Fig. 4.14. The fiducial markers, spheres in the reconstructed tomogram, are clearly visible, allowing the correct placement of the dislocations in the 3D model and straightforward identification of the slip planes and line directions of the
dislocations. As this experiment was done only for feasibility purposes, the dislocation analysis was not done.

![Reconstructed tomogram and related 3D dislocation model](image)

**Figure 4.14.** Reconstructed tomogram and related 3D dislocation model of the grain boundary/dislocation interaction shown in Fig. 4.13. The grain boundary is represented as a blue plane in the model.

### 4.1.5 Comparison to iterative reconstruction techniques

Two tilt series were reconstructed using a SIRT algorithm, one shown in Figure 4.2 as an example of a traditional diffraction-contrast tilt series, and the series used to construct the
tomogram shown in Figure 4.14 to explore the quality of reconstruction from tilt series aligned using the digital fiducial marker technique.

The tomogram shown in Figure 4.15 is from the same interaction as in Figure 4.1. The same images were used with changes only in the boundaries of the images due to loading differences between the two software programs used. The only variable was the reconstruction algorithm as the SIRT algorithm was used to reconstruct the tomogram shown in Figure 4.15. As can be seen in the tomogram visualization, the dislocations that were kept in contrast using two-beam BF imaging reconstructed well, comparable to the quality of the dislocations visible in the tomogram reconstructed using a weighted back projection algorithm (Figure 4.15c-d). The resolution, however, degrades quickly near the boundary plane. In the opposite grain, where the dislocations were not kept in two-beam BF contrast during acquisition of the tilt series, no features are visible in the tomogram visualization. The boundary plane itself is poorly identifiable and has a wavy instead of planar face (Figure 4.15 a). This may be due to contrast from bend contours visible in the tilt series images (Figure 4.2).
Figure 4.15. Views from a tomogram visualization reconstructed using a SIRT algorithm. The tomogram was reconstructed using the same tilt series as shown in Figure 4.2. (c) and (d) show enlarged images of the dislocations from approximately the same viewing angle as is shown in (a) and (b), respectively. Anomalous contrast near the edges, indicated by arrowheads in (a), is an artifact resulting from loading pre-aligned images into the TomoJ software.

The SIRT algorithm was also applied to the dataset reconstructed using the fiducial marker method shown in Figure 4.14 to reconstruct a tomogram (Fig. 4.16). Although the grain boundary plane is well resolved in the tomogram, the location of the fiducial markers is no longer clear. Even when the tomogram is viewed directly down the through thickness direction, Figure 4.16a, the location of the markers cannot be identified with the precision needed to construct a 3D model of the interaction. A second tomogram was reconstructed using the SIRT
algorithm from a different dataset, also collected using the Gatan heat/strain stage and aligned using the fiducial marker method. As can be seen, the fiducial markers are easily identifiable, even from a viewing angle perpendicular to the through thickness direction (Figure 4.17a-b). The grain boundary plane is also clearly resolved (Figure 4.17c), arguably with greater clarity than provided by the tomogram reconstructed using a weighted back projection. The location of the partial dislocations emitted into the neighboring grain, however, can only be vaguely determined, far below the resolution needed to accurately identify the plane on which they reside (Figure 4.17d). This is in contrast to the weighted back projection reconstruction in which the stacking fault fringes can be resolved at select discrete orientations, allowing the accurate identification of the plane on which they reside (Fig. 4.40).

![Figure 4.16. Views from a tomogram visualization reconstructed using a SIRT algorithm. The tomogram was reconstructed using the same tilt series as shown in Fig. 4.14.](image-url)
Figure 4.17. Views from a tomogram visualization reconstructed using a SIRT algorithm. The tomogram was reconstructed using the same tilt series as shown in Fig. 4.40.

4.1.6 Summary of 4D analysis techniques

Four different approaches to 3D analysis were presented here; orienting the sample such that a crystallographic plane normal is aligned with the stage tilt axis prior to loading the stage into the TEM, use of gold fiducial markers for alignment, combining in situ TEM deformation with post mortem FIB machining, and placing digital fiducial markers to aid in aligning images in a tilt series. These approaches will be briefly compared with their benefits and shortcomings listed below.
Using a sample that has a crystallographic plane normal aligned with the stage tilt axis prior to loading the stage into the TEM allows a high quality tomogram to be reconstructed from a collected tilt series. As only a single diffraction condition is used during image acquisition, contrast fluctuations between images should be minimal in comparison to techniques reliant on external fiducial markers such as manually placed digital markers or colloidal gold particles. The primary shortcoming of using a pre-aligned sample is that it can only be used for specially designed experiments, mainly those involving single or bicrystal samples. Also, characterization of the dislocations is difficult as only a single \( g \)-vector is available for \( g \cdot b \) analysis.

Application of gold fiducial markers to a sample prior to TEM investigation allows a tilt series to be aligned from an arbitrarily oriented grain as the gold particles can be used as fiducial markers. While contrast fluctuations between images due to varying diffraction conditions do reduce the quality of the reconstructed tomogram, it was shown that a usable tomogram can still be produced. This approach is much more versatile than using a pre-aligned sample, expanding 4D analysis to polycrystalline materials. As the colloidal gold is applied to the sample suspended in a water droplet, this technique cannot be applied to materials which readily oxidize in the presence of water. A second drawback is that areas of interest, where a dislocation/grain boundary interaction is occurring for example, may be obscured by dense conglomerations of gold particles or may have an insufficient number of nearby particles for image alignment.

When combining post mortem FIB machining with in situ TEM deformation, the interaction of interest can be characterized using a double tilt stage, allowing \( g \cdot b \) analysis and collection of a high quality tomogram. However, this approach is destructive in nature and can only be applied after an interaction is complete.
The digital fiducial marker method is not dependent on physical features in the sample other than the dislocations themselves, making it more versatile than using colloidal gold particles. As multiple \( \mathbf{g} \)-vectors are used during the acquisition of the tilt series, Burgers vector characterization using \( \mathbf{g} \cdot \mathbf{b} = 0 \) analysis is also possible. The circular fiducial markers are easily recognizable by most software packages, and their perfectly even contrast makes reconstruction a straightforward process, reducing the number of images needed for a successful reconstruction to as few as 10 as opposed to the 40 or more usually used in diffraction contrast electron tomography. This versatility and ease of use makes the digital fiducial marker method ideal for the interactions investigated in this study and so it was used exclusively in the interactions described below.

4.2 DISLOCATION/GRAIN BOUNDARY INTERACTIONS IN STAINLESS STEEL

4.2.1 Ex situ deformation

By deforming a sample \textit{ex situ}, the slip transfer behavior as occurs during bulk deformation can be analyzed. The first characterized interaction involved three separate but identical slip systems impinging on a coherent \( \Sigma 3 \) twin boundary in a deformed 304 stainless steel disk (Fig. 4.18) [190]. A series of images of this interaction was collected by tilting along the parallel \{111\} planes on either side of the boundary and collecting images in BF mode every 2° over a tilt range of \( \pm 40° \) (Fig. 4.19). A 3D dislocation model of the interaction was constructed with a coordinate system placed in each grain relative to its orientation (Fig. 4.20). The two coordinate systems are related by a \( 109.5° \) rotation about the [011] axis.
Figure 4.18. Bright-field micrograph of dislocations interacting with a coherent $\Sigma 3$ twin boundary in 304 stainless steel. Arrows indicate the assumed direction of dislocation motion and system labeling is for reference in the text.
Figure 4.19. (a) Four images taken from the tilt series used to reconstruct the tomogram of the interaction shown in Fig. 4.18. The tilt angle is given in each image. (b) Selected view of the tomogram visualization.
Interestingly, whereas the incoming dislocations in all three systems are identical, two different reactions occur at the boundary. The incoming dislocations reside on the δ-plane and have a Burgers vector of \( \mathbf{b} = \pm a/2[011]_{\text{in}} \). The line direction is parallel to \([11 \bar{0}]_{\text{in}}\), making them mixed character dislocations. The emitted dislocations in system 1 have a Burgers vector \( \mathbf{b} = \pm a/2[011]_{\text{out}} \), reside on the β-plane, and have a line direction parallel to \([211]_{\text{out}}\), making them pure edge dislocations. The reaction in system 2 resulted in two emitted slip systems, labeled \( a \) and \( b \) in Figure 4.18. The dislocations that comprised system \( a \) were found to reside on the γ-plane with a Burgers vector \( \mathbf{b} = \pm a/2[11 \bar{0}]_{\text{out}} \) and a line direction parallel to \([121]_{\text{out}}\), making them mixed dislocations. \( \mathbf{g} \cdot \mathbf{b} \) analysis reduced the Burgers vectors possible for system \( b \) to either \( \mathbf{b} = \pm a/2[11 \bar{0}]_{\text{out}} \) or \( \mathbf{b} = \pm a/2[101]_{\text{out}} \). The tomogram showed these dislocations to reside on the δ-plane, leaving \( \mathbf{b} = \pm a/2[11 \bar{0}]_{\text{out}} \) as the only possible Burgers vector. Due to insufficient contrast for reliable reconstruction, the line direction of these dislocations could not be determined. Consistent with a Σ3 grain boundary, the tomogram shows the boundary plane to be \((111)_{\text{in}}// (111)_{\text{out}}\).
Figure 4.20. Dislocation model constructed from tomogram shown in Fig. 4.19. Thompson tetrahedra define the coordinate system for each grain, the boundary location is given by the blue plane, and dislocations are colored according to their Burgers vector.

The Burgers vector of the residual grain boundary dislocation from and the resolved shear stress acting on all available slip systems in the outgoing grain was calculated. The data are displayed graphically in Figure 4.21. As can be seen, four of the twelve available systems leave, in terms of $|b_r^{gb}|$, identical dislocations in the grain boundary. The two slip systems activated in system 2 had identical Burgers vectors and minimized the Burgers vector of the residual grain boundary dislocation. The system with the higher resolved shear stress, $b = \pm a/2[1\overline{1}0]_{0ut}$ on the $\gamma$-plane, appeared to be more active from the TEM micrographs. Of the other two systems that
left equal dislocations in the boundary, the $\pm[101]_{\text{out}}(111)_{\text{out}}$ had only a slightly lower resolved shear stress than the main emitted system. It may be that this was sufficient to activate the observed system; that is, the resolved shear stress acted as a deciding factor between two otherwise equally optimal systems. The slip system activated in system 1 had the highest resolved shear stress acting on it. According to the criteria developed by Lee et al. [9], the system should have only limited activity before a different system, most likely that seen in the system 2 interaction, would activate. As the deformation was performed ex situ, this assumption could not be verified.
Figure 4.21. Resolved shear stress and magnitude of the Burgers vector of the residual grain boundary dislocation associated with each potential emitted slip system from the interaction shown in Fig. 4.18. Values are normalized with the highest value set to unity to facilitate direct comparison. The observed emitted systems are indicated consistent with the labeling given in Fig. 4.18.

A second interaction involved dislocations impinging on a twin boundary, new dislocations being ejected from the boundary into the twinned crystal and their interaction with the other twin boundary [191]. This sample was deformed ex situ. The overall interaction is captured in the BF images at two different sample tilts and imaging conditions presented in Figure 4.22; diffraction conditions used were A) $\mathbf{g} = (1 \overline{1} 1)_{in}$ and B) $\mathbf{g} = (2 \overline{2} 0)_{o.o}$ with the excitation error slightly positive. The twin plane was found to be $(1 1 1)_{in}$. Three identical but
distinct slip systems impact one of the twin boundaries - these interactions are referenced as system 1, system 2, and system 3 in Figure 4.5. As seen in the image, these incoming dislocations generate different responses from the twin boundary, and these are labeled as 1a and 1b for those primarily associated with system 1; 2a, 2b, 2c, and 2d for system 2 reactions; and as 3a, 3b, 3c, 3d and 3e for system 3 reactions. The second image of the same region at a different sample tilt and diffraction vector shows a large number of extrinsic grain boundary dislocations (Fig. 4.22B), labeled 1b and 3e in Figure 4.22A. The curvature of these dislocations suggests that they originate from an unrelated dislocation system impinging on the grain boundary, though from the still shot of the interaction this cannot be known with certainty. The Burgers vector of the incoming dislocations shown does not allow cross-slip onto the boundary plane, but the dislocations could be a byproduct of the residual dislocations left after the absorption and emission of dislocations, especially when considering the complexity of the system 3 interaction.
Figure 4.22. Images using two different diffraction conditions of dislocations interacting with twin boundaries. The $g$-vectors used for the two-beam imaging is shown in the upper corner of each image and were indexed to be (A) $g = (1\ 1\ 1)_\text{in}$ and (B) $g = (2\ 2\ 0)_\text{out}$. The assumed dislocation propagation direction is indicated by the arrows. Labels are for reference in the text.
Images of the twin boundary region were acquired over an angular range of -35° to +39° in α-tilt and -22° to 1° in β-tilt with an image collected every 2°, a tomogram reconstructed from the resulting images, and a 3D-dislocation model formed from the tomogram. The region involving reaction products 3a and 3b was not reconstructed due to the complexity of the structure. Also, many of the extrinsic grain boundary dislocations could not be identified in the tomogram, and so were not included in the model. Examples from the reconstructed tomogram and 3D-dislocation model of the interactions as viewed from different vantage directions are shown in Figure 4.23; Thompson tetrahedra were added to the original and twinned crystal region for reference and the dislocations were color-coded according to their Burgers vector. Figure 4.23b shows an enlarged view of the dislocations seen end on in the tomogram. As a consequence of the large range of β-tilt used when acquiring the tilt series, precession effects in the form of streaking are apparent in the image. However, the 3D location of the dislocations in the tomogram is still readily identifiable.
Figure 4.23. Visualization of the tomogram and different views of the 3D dislocation model of the interaction shown in Fig. 4.22. The green planes indicate the twin boundaries’ location and the red plane represents a faulted region between Shockley partial dislocations. Thompson tetrahedra, specific to each region, are included and the dislocations are color coded according to their Burgers vectors.

The Burgers vectors of the dislocations, with the exception of the overlapping partial dislocations associated with system 3, were determined using the invisibility condition. A
summary of the interaction is given in Table 4.2. The incoming dislocations are on the slip system \( \pm \frac{a}{2}[110]_{in}(111)_{in} \), and have a line direction parallel to [112]_{in}, making them pure edge dislocations; the slip plane and dislocation line direction are determined readily by inspection of the 3D model, see Figure 4.23 for identification of the slip plane. System 1 generates dislocations with a Burgers vector \( \mathbf{b} = \pm \frac{a}{2}[101]_{out} \) that are emitted into the twinned region on two parallel planes, (111)_{out}; this is again obvious from Figure 4.23. The emitted dislocations on the parallel planes have two different line directions, [121]_{out} and near [132]_{out}, making them edge and mixed character dislocations, respectively. There is also very limited emission, only the lead partial dislocation can be seen, emerging from the boundary back into the original grain. The Burgers vector could not be determined due to the proximity of these dislocation to the grain boundary.

System 2 causes emission of dislocations with a Burgers vector \( \mathbf{b} = \pm \frac{a}{2}[101]_{out} \) into the twinned region, 2a, but only on a single plane, the (111)_{out}. In this case, all the dislocations have a line direction [121]_{out} and so are all pure edge dislocations. This reaction is coupled with a more limited emission of Shockley partial dislocations back into the original grain (2c). The partial dislocations reside on the (111)_{in}, the lead partial dislocation has a Burgers vector \( \mathbf{b} = \pm \frac{a}{6}[112]_{in} \) and the trailing one has a Burgers vector \( \mathbf{b} = \pm \frac{a}{6}[211]_{in} \). There is also an additional dislocation system active in system 2 in which the dislocations are perfect dislocations, but with half of the dislocation split into Shockley partial pairs with a lead Burgers vector of \( \mathbf{b} = \pm \frac{a}{6}[211]_{out} \) and a trailing Burgers vector of \( \mathbf{b} = \pm \frac{a}{6}[121]_{out} \) (2b).

Incoming system 3 dislocations generated a complex response from the twin boundary with dislocations being emitted into the twin and back into the original grain. The dislocation structure ejected into the twinned region consisted of emission on a plane parallel to systems 1a
and 2a (3a), as well as an additional system of partial dislocations with the attendant ribbon of stacking fault that appeared to originate from a wider region of the twin boundary (3b). Two dislocation systems were emitted back into the original grain; those in system 3c reside on the (111)$_{in}$ plane and have Burgers vectors $\mathbf{b} = \pm \frac{a}{6}[112]_{in}$ and $\mathbf{b} = \pm \frac{a}{6}[211]_{in}$ which combine to yield $\mathbf{b} = \pm \frac{a}{2}[101]_{in}$, and those in system 3d which reside on the (111)$_{in}$ plane and have a Burgers vector of $\mathbf{b} = \pm \frac{a}{2}[011]_{in}$. Also evident in this region, as small black / white dots, are dislocations in the twin boundary itself, seen more clearly in Figure 4.22B.

As the dislocations generated from the interaction with the upper twin boundary traverse the twinned region and intersect the other twin boundary, they cause different responses. System 1a appears to cause emission of an isolated dislocation, although there is significant contrast along the boundary between the points of intersection of systems 1a and 2a with the twin boundary. System 2a on intersecting the lower twin boundary generates system 2d, which consists of the emission of lead partial dislocations with trailing extended faulted regions. The trailing partial dislocation was not ejected from the grain boundary. As evident from the 3D model, the ejected partial dislocations reside on the (111)$_{in}$ plane, which is identical to the incoming plane impacting the upper twin boundary. Assuming these emitted dislocations have the same Burgers vector as the incoming dislocations in system 2, the emitted partial dislocations should have a Burgers vector of either $\mathbf{b} = \pm \frac{a}{6}[2 11]_{in}$ or $\mathbf{b} = \pm \frac{a}{6}[121]_{in}$; this was not verified in the experiment.

Subtraction of the Burgers vectors of the incoming and outgoing dislocations, after expressing them in a common reference frame, yields the following information: For system 2, the emitted dislocations, which would create dislocations in the twin boundary with the smallest Burgers vectors, are $\mathbf{b} = \pm \frac{a}{2}[101]_{out}$ and $\mathbf{b} = \pm \frac{a}{2}[110]_{out}$. This is consistent with the analysis for
system 2a, which has the emitted dislocation having a Burgers vector of $b = \pm \frac{a}{2}[101]_{\text{out}}$, or, after the appropriate coordinate transformation, $b = \pm \frac{a}{6}[4\;1\;1]_{\text{in}}$. However, the interaction of system 2 with the twin boundary generates emission of more than one set of dislocations from it. In considering the interaction and the generation of system (the ± sign is omitted to simplify the analysis), $b^r_{gb}$ generated by activation of system 2a is:

$$\frac{a}{2}[110]_{\text{in}} \rightarrow \frac{a}{6}[4\;1\;1]_{\text{in}} \rightarrow \frac{a}{6}[121]_{\text{in}}$$

As the twin resides on $(111)_{\text{in}}$, this first interaction leaves a partial dislocation glissile on the boundary plane. Activation of slip system 2c, could result in the following reaction for emission of the leading partial dislocation:

$$3\frac{a}{6}[121]_{\text{in}} \rightarrow \frac{a}{6}[1\;1\;2]_{\text{in}} \rightarrow \frac{a}{6}[271]_{\text{in}},$$

and with emission of the trailing partial dislocation, $b = \pm \frac{a}{6}[2\;1\;1]_{\text{in}}$ yields

$$\frac{a}{6}[271]_{\text{in}} \rightarrow \frac{a}{6}[2\;1\;1]_{\text{in}} \rightarrow a[0\;1\;0]_{\text{in}}.$$

This reduces $|b^r_{gb}|$ after 3 absorption and emission events from 1.22a to a magnitude of 1a. As the deformation was performed ex situ, the number of absorbed dislocations associated with emission in system 2c could not be verified. In this interaction, it is only after the second partial dislocation is emitted that $|b^r_{gb}|$ is reduced; consideration of the lead partial dislocation only results in an increase of $|b^r_{gb}|$. A significant residual grain boundary dislocation remains after slip transfer, which could explain the complexity of system 3 as more dislocation types are emitted in an effort to further reduce the elastic strain in the boundary.
System 2b appears to play no role in reducing the buildup of elastic strain in the boundary, and appears to contradict the slip transmission criteria. Their presence, however, could be explained by the extrinsic grain boundary dislocations exiting the interface at the same location as the system 2 interaction, possibly provoked by the elastic strain buildup surrounding the interaction. Inspection of the tomogram supports this as the system 2b dislocations (the yellow dislocations in Figure 4.23) originate from the extrinsic grain boundary dislocations leaving the boundary.

In contrast to the emitted partial dislocations in system 2c, the partial dislocations in system 2d leave an elongated faulted region as the trailing partial dislocation remains in the grain boundary. The plane of the partial dislocation can be identified in system 2d as the (111)$_{in}$, but the Burgers vector of the individual dislocations could not be resolved. The possible Burgers vectors were limited to a combination of $b = \pm \frac{a}{6}[112]$, $b = \pm \frac{a}{6}[12\ 1]$, and $b = \pm \frac{a}{6}[2\ 11]$. It is reasonable to assume the emitted partials have the same combined Burgers vector as the incoming dislocation, even more so as it is the system which results in the minimal residual Burgers vector. This suggests an interaction sequence of:

$$\frac{a}{6}[4\ 11]_{in} -- \frac{a}{6}[2\ 11]_{in} = \frac{a}{6}[2\ 02]_{in}$$

$$\frac{a}{6}[2\ 02]_{in} -- \frac{a}{6}[12\ 1]_{in} = \frac{a}{6}[12\ 3]_{in}.$$

This sequence is opposite to that described in system 2, where the lead partial dislocation reduces the $|b_{r^{gb}}|$, but emission of the trailing partial dislocation increases it, which could account for the emission of just the leading partial dislocation with the attendant ribbon of stacking fault. Similar effects are seen during the in situ experiments described below.
Table 4.2. Summary of the dislocation interactions shown in Figure 4.6.

<table>
<thead>
<tr>
<th>System</th>
<th>Emission into twinned region</th>
<th>Back emission into original grain</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Slip system character</td>
<td>Slip system character</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Incoming system</td>
<td>$\pm\frac{a}{2}[110]<em>{in}(111)</em>{in}$</td>
<td>Edge</td>
</tr>
<tr>
<td>System 1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1a</td>
<td>$\pm\frac{a}{2}[101]<em>{out}(111)</em>{out}$</td>
<td>Edge</td>
</tr>
<tr>
<td>1a</td>
<td>$\pm\frac{a}{2}[101]<em>{out}(111)</em>{out}$</td>
<td>Mixed</td>
</tr>
<tr>
<td></td>
<td>1 Unidentified</td>
<td></td>
</tr>
<tr>
<td>System 2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2a</td>
<td>$\pm\frac{a}{2}[101]<em>{out}(111)</em>{out}$</td>
<td>Edge</td>
</tr>
<tr>
<td>2b</td>
<td>$\pm\frac{a}{6}[211]<em>{out}(111)</em>{in}$</td>
<td></td>
</tr>
<tr>
<td>2b</td>
<td>$\pm\frac{a}{6}[211]<em>{out}(111)</em>{in}$</td>
<td></td>
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Using the 3D model created of the interaction to identify the line directions, and thus the character, of the incoming dislocations, the local resolved shear stress imposed on the outgoing
systems from the incoming dislocation system can be calculated and the results are presented graphically in Figure 4.24. The normalized value of \( |\mathbf{b}_{r}^{gb}| \) is also included. There were four systems which left dislocations in the grain boundary with Burgers vectors of identical magnitude. Of these, the \( \pm \frac{a}{2}[110]_{out}(111)_{out} \) had the highest resolved shear stress acting on it, but it was the \( \pm \frac{a}{2}[101]_{out}(111)_{out} \) which was emitted into the twinned region. In this case, the resolved shear stress model appears to be an oversimplification as it does not take into account the narrow twinned region or the extrinsic grain boundary dislocations.
Figure 4.24. Resolved shear stress and magnitude of the Burgers vector of the residual grain boundary dislocation associated with each potential emitted slip system from the interaction shown in Fig. 4.22. Values are normalized with the highest value set to unity to facilitate direct comparison.

4.2.2 Variable temperature experiment

Initial in situ TEM straining experiments were designed to investigate the effects of temperature on dislocation interactions with a grain boundary. To enable identification of the role of thermal effects, dislocations were observed to interact with a grain boundary at room temperature, the load relaxed, the temperature increased and the sample reloaded. The first
interaction involved partial dislocations ejected from a crack tip piling-up and interacting with a Σ3 coherent twin boundary. The interaction at room temperature is shown in the micrographs presented in Figure 4.25; the grain boundary normal is perpendicular to the beam direction and the arrow indicates its location. The image contrast in Figure 4.25a is complex as close to the twin boundary the expected periodicity in the contrast from three or more overlapping stacking faults is not obeyed, although it is obeyed away from the boundary. Therefore, from the images acquired during the in situ deformation experiment it cannot be determined if the contrast arises from overlapping stacking faults or a thin twin [192, 193]. Although the dislocation pile-up is extensive and there is evidence of strain contrast in the adjoining grain, no dislocations are ejected from the twin boundary into it. From the in situ experiments, it is seen that many dislocations enter the twin boundary and move along it. This is captured in Figure 4.25a-b with the dark contrast features in the twin boundary indicating glissile dislocations; examples are marked by arrowheads. The dislocations in the boundary only propagate in one direction. That the dislocations move along the boundary plane before any dislocation emission takes place suggests that this is a cross-slip event and is not the propagation of residual grain boundary dislocations from an absorption and emission reaction. If the incoming partial dislocations were associated with a twin, the strain accommodation at the boundary, as noted by Mahajan et al., would require activation of two systems out of the boundary as well as an additional system glissile in the boundary plane [194, 195]. This interaction sequence is shown in Figure 4.26 where twinning dislocations with Burgers vector \( \mathbf{b} = \frac{\mathbf{a}}{6}[1 0 1] \) are seen impinging on a coherent twin boundary. This results in the emission of perfect dislocations with Burgers vector \( \mathbf{b} = \frac{\mathbf{a}}{6}[1 1 4] \) into the neighboring grain and the back emission of dislocations with Burgers vector \( \mathbf{b} = \frac{\mathbf{a}}{6}[1 2 1] \) into the original grain. Residual dislocations with Burgers vector \( \mathbf{b} = \frac{\mathbf{a}}{6}[1 2 1] \)
remain in and glide along the boundary after the interaction. Such a sequence of events was not observed experimentally, suggesting that the partial dislocations are associated with stacking faults and not twins and requires that the partial dislocations recombined either prior to or on entry into the twin.

Figure 4.25. Dislocations impinging on a coherent $\Sigma 3$ boundaries at room temperature.

Figure 4.26. Mechanism proposed by Mahajan et al. for a twin interaction with a coherent twin boundary (C.T.B.) [194]. (a) shows the twinning partial dislocations impinging on the boundary and (b) shows their dissociation into three different dislocation systems. All Burgers vectors are given in the coordinate system of the incoming grain.
Reloading the sample at 400°C caused activity in the original system and activation of another dislocation system as shown in Figure 4.27. On interacting with the twin boundary, the main reaction was for the approaching dislocations to be incorporated in and become mobile along the twin boundary. This incorporation into the boundary occurred at a low dislocation pileup density, with as few as two dislocations being present near the boundary before incorporation as opposed to the dense pileup formed at room temperature. Despite the increase in dislocation activity no dislocations are emitted from the twin boundary initially. However, as shown by the appearance of surface slip traces, Figure 4.27c, transmission of slip eventually occurs. During the observation period, two parallel sets of slip traces were seen exiting the boundary, indicating multiple nucleation sites on the boundary.

Diffraction analysis of the interaction showed the incoming partial dislocations reside on the (111)$_{in}$ and the boundary to be on the (111)$_{in}$. As the dislocations appear to be glissile on both, the Burgers vector of the incoming dislocations is deduced to be $\mathbf{b} = \pm \frac{a}{2}[011]_{in}$. Two different dislocation systems were seen emitting from the boundary. Both were perfect but one, the more dominant system, had a Burgers vector $\mathbf{b} = \pm \frac{a}{2}[101]_{out}$, and the other had a Burgers vector $\mathbf{b} = \pm \frac{a}{2}[011]_{out}$ with the coordinate systems of the two grains related by a 60° rotation about the [111]$_{in}$ axis. The dominant emitted system was more prevalent by more than a factor of ten, but the less dominant system was seen emitted first. When expressed in the incoming grain coordinate system, the dominant emitted system has Burgers vector $\mathbf{b} = \pm \frac{a}{2}[011]_{in}$, identical to the Burgers vector of the incoming dislocations. In this case there should be no residual dislocation left in the boundary from the interaction and so the interaction follows the criteria proposed by Lee et al. [9, 77]. The secondary emitted system leaves a significant residual
dislocation in the boundary, and so, in accordance to the slip transfer criteria, it becomes less active and the system which leaves the minimal residual dislocation becomes dominant. The line direction of the dislocations was not recovered, and so the influence of the local stress state could not be determined. The interaction is therefore qualitatively independent of temperature, but the dislocation density in the pile up was lower than at room temperature. These results suggest that the twin boundary poses a lesser barrier to dislocation emission at elevated temperature and higher levels of complexity are reached at an earlier stage of deformation than during room temperature deformation.

![Image](image_url)

*Figure 4.27.* Dislocations impinging on a coherent Σ3 boundary at 400°C. This interaction is the same as shown in Fig. 4.25 at a later time and at elevated temperature.
4.2.3 Interactions with coherent $\Sigma 3$ twin boundaries

Due to the high prevalence of coherent $\Sigma 3$ twin boundaries in 304 stainless steel, a variety of dislocation interactions could be explored. As shown in previous sections, dislocations impinging on $\Sigma 3$ boundaries can be absorbed into the boundary (Fig. 4.22), cross-slip onto the boundary plane (Fig. 4.27), and/or be emitted into the neighboring grain. Further in situ deformation experiments at elevated temperatures were conducted to explore the range of these dislocation interactions.

A common response to dislocations impinging on a twin boundary was for the dislocations to enter the twin and become mobile along the boundary plane. An example of this is shown in Figure 4.28 in which a sample has been strained in situ at 400°C. The incoming dislocations have Burgers vector $b = \pm \frac{1}{2}[110]_{in}$ and reside on the $(1\bar{1}1\bar{1})_{in}$. The boundary is a $\Sigma 3$ twin boundary residing on $(1\bar{1}1\bar{1})_{in}$. The dislocation contrast shown in the adjacent grain is due to an unrelated dislocation system. The dislocations readily cross-slip onto the boundary plane, propagate along the boundary in one direction, and remain in the boundary far from the initial interaction point. This interaction is similar to the initial boundary response shown in Fig. 4.27.
A similar cross-slip reaction, though during *in situ* straining at room temperature and with a controlled purity 21Cr15Ni austenitic stainless steel, is shown in Figure 4.29. Again, dislocations impinging on a Σ3 boundary are seen to readily cross-slip onto the boundary. Between images a and b, 5.8 seconds passed, in which time 7 dislocations cross-sliped onto the boundary. The dislocations propagate in one direction only, to the right of the impingement point, though some are seen to reside to the left as well. These do not propagate but remain immobile in the boundary plane. The mobile dislocations remained in the boundary and propagated along the entire length of the grain. Slip traces exiting the boundary plane, seen in
Figure 4.29c which was taken at a later time than a and b, show that slip transmission does eventually occur. The emitted dislocations were not characterized.

Figure 4.29. Dislocations cross-slipping and transmitting through a coherent $\Sigma 3$ twin boundary during *in situ* straining at room temperature. (a) and (b) are frames taken from a movie taken of the interaction, with the time elapsed given in (b). (c) was taken at a later point in the interaction. Arrows indicate direction of dislocation motion.
Figures 4.30 and 4.31 illustrate the effects of the Burgers vector of the incoming dislocations on interactions at \( \Sigma 3 \) boundaries. Two different dislocation systems are shown impinging on a \( \Sigma 3 \) twin boundary during \textit{in situ} straining at 400°C. Perfect dislocations with a Burgers vector of \( \mathbf{b} = \pm \frac{a}{2}[110]_{in} \) glissile on \((111)_{in} \) and \( \mathbf{b} = \pm \frac{a}{2}[101]_{in} \) on \((111)_{in} \), impinged on a \( \Sigma 3 \) twin boundary which resides on \((111)_{in} \). The spacing between dislocations leading up to the boundary is large in comparison with the interaction shown in Figure 4.25. The Burgers vector of the dislocations visible in Figure 4.30b allows the dislocations to cross-slip onto the boundary plane, and movement of dislocations on it is evident (Fig. 4.31a-b). Interestingly, the dislocations are observed to propagate in both directions along the twin boundary. The dislocations visible in Figure 4.30a are unable to cross-slip onto the boundary plane, and every instance of impingement results in the immediate, to within 1/10th of a second, emission of a dislocation into the neighboring grain (Fig. 4.31c). Tracking individual dislocations shows that dislocations visible in Figure 4.30b remain perfect as they enter the boundary. Those dislocations which impinge to the left of the main interaction point propagate to the left along the boundary (Fig. 4.31b) and those which impinge on the boundary to the right of the main interaction zone propagate to the right (Fig. 4.31a).
Figure 4.30. Two different dislocation systems impinging on a coherent $\Sigma 3$ twin boundary during \textit{in situ} deformation at 400°C. Diffraction vectors are given for each image.
Figure 4.31. Different responses of a coherent $\Sigma 3$ twin boundary to dislocation impingement. Dislocations shown in Fig. 4.30b that impinge to the right of the main impingement point enter the boundary and slip to the right along it (a). Dislocations shown in Fig 4.30b that impinge to the left of the main impingement point enter the boundary and slip to the left along it (b). Dislocations shown in Fig 4.30a enter the boundary and quickly transmit through (c). The two frames shown in (c) are separated by 0.1 seconds.
A final dislocation/twin boundary interaction is shown in Figure 4.32. Here, a system of perfect dislocations was seen impinging on a coherent \( \Sigma 3 \) twin boundary during \textit{in situ} deformation at 400°C. Although the dislocation activity at the impingement point was not captured, the \textit{in situ} deformation allowed the direction of dislocation motion to be confirmed. The incoming dislocations instigated the emission of partial dislocations into the neighboring grain. The lead partial did not extend far into the grain before the trailing partial dislocation was also emitted, as is evident in Figure 4.32b where both the lead and the trailing partial dislocations are indicated (labeled L and T, respectively). Two systems were also emitted back into the original grain, one seemingly overlapping the incoming dislocations, labeled 1 in Figure 4.32b, and the other propagating in a different plane, labeled 2 in 4.32a. Additional systems active near the boundary include a twin or series of partial dislocations impinging on the boundary, labeled 3 in Figure 4.32a, as well as scattered perfect dislocations propagating towards the boundary, labeled 4 in Figure 4.32a. These other systems seem to have only a minor impact on how the interaction progressed.
Figure 4.32. Dislocations impinging on a coherent Σ3 grain boundary during in situ straining at 400°C. The associated diffraction patterns are given in each image. The direction of dislocation motion is indicated by arrows. Labeling is for reference in the text.
The twin boundary shown in the interaction resided on the $(11\bar{1})_{in}$. The main dislocation system impinging on the boundary was found to be composed of perfect dislocations with Burgers vector $b = \pm \frac{a}{2}[101]_{in}$. The emitted partial dislocations glided on $(111)_{out}$, with the leading and trailing partial dislocations having Burgers vector $b = \pm \frac{a}{6}[12\bar{1}]_{out}$ and $b = \pm \frac{a}{6}[12\bar{1}]_{out}$, respectively, resulting in a combined Burgers vector of $\pm \frac{a}{2}[01\bar{1}]_{out}$. The system of perfect dislocations emitted back into the original grain, visible in Figure 4.32a, was found to have Burgers vector $b = \pm \frac{a}{2}[101]_{in}$, identical to that of the incoming dislocations. The available diffraction information limited the possible Burgers vectors of the back-emitted dislocations visible in Figure 4.32a to either $b = \pm \frac{a}{2}[110]_{in}$ or $b = \pm \frac{a}{2}[101]_{in}$.

Figure 4.33 and 4.34 display the normalized value of $|b_{r}^{gb}|$ when considering the emission of perfect and partial dislocations, respectively. The observed emitted system is indicated in each graph, assuming that the sign of the Burgers vector corresponds to the system that best minimizes $|b_{r}^{gb}|$. As can be seen, the emitted system minimizes $|b_{r}^{gb}|$ only when both the leading and trailing partials dislocations are considered. This is not the case when only the lead partial is considered as there is one system, $[12\bar{1}]_{out}(11\bar{1})_{out}$, which results in a lower value of $|b_{r}^{gb}|$. The evolution of the residual grain boundary dislocation, considering only the absorption of the main incoming system and the emission of partial dislocations, proceeds as follows (note that the sign of the incoming dislocation system is assumed):

Beginning with the emission of the lead partial dislocation after a single incoming dislocation has been absorbed:
\( \frac{a}{2}[1\ 0\ 1]_i - \frac{a}{18}[5\ 5\ 2]_i = \frac{a}{18}[4\ 5\ 7]_i \)

Followed by the emission of the trailing partial dislocation:

\( \frac{a}{18}[4\ 5\ 7]_i - \frac{a}{18}[7\ 2\ 1]_i = \frac{a}{6}[1\ 12]_i \)

This shows that after the complete emission of the dislocation, a single partial dislocation remains in the boundary. This partial dislocation is glissile in the twin plane, but no evidence of dislocation motion along the boundary is evident in the images. The emission of the trailing partial dislocation reduces \( b_r \) further. This is similar to the reaction seen in Figure 4.22 for system 2c and opposite to that seen in system 2d.
Figure 4.33. Normalized magnitude of the Burgers vector of the residual grain boundary dislocation associated with each available system for emission from the interaction shown in Fig. 4.32. Only perfect dislocations are considered. The Burgers vector representing the combined partial dislocations observed in the interaction is indicated by an arrow.
Figure 4.34. Normalized magnitude of the Burgers vector of the residual grain boundary dislocation associated with each available system for emission from the interaction shown in Fig. 4.32. Only partial dislocations are considered. The observed leading and trailing dislocations are indicated by arrows with ‘L’ and ‘T,’ respectively.

4.2.4 Interactions with high angle grain boundaries

For interactions involving high-angle grain boundaries, the response of the grain boundary to an incoming dislocation system can vary with number of dislocation interactions. Figure 4.35 shows an incoming dislocation system impinging on a grain boundary during an in situ straining experiment carried out at a nominal temperature of 400°C. The incoming dislocations are all part of the same system, but reside on multiple parallel planes with a single
dislocation of different type being tangled with the incoming dislocation system. The interaction results in two emitted dislocation systems on non-parallel planes into the neighboring grain, the first being the limited emission of perfect dislocations, which had already left the viewing area shown in Figure 4.35, and the second being an extensive emission of partial dislocations on multiple parallel planes. The buildup of elastic strain in the boundary, evidenced by the strain contours surrounding the interaction, also results in cross-slip from the main incoming dislocation system before impingement on the boundary. The cross-slipped dislocations propagate on a plane approximately parallel to the boundary plane, and these can cross-slip back towards the boundary. This behavior is seen more clearly in Figure 4.36a. These dislocations are easily incorporated into and emitted from the boundary, with a single absorbed dislocation instigating the emission of a lead Shockley partial dislocation into the neighboring grain with no pileup of dislocations forming before absorption (Fig. 4.36b). Also of note is the local disruption of the grain boundary structure evident at the main point of dislocation impingement (Fig. 4.36c). This local disruption increased as the interaction progressed, eventually leading to the formation of a sharp bend or break in the boundary (resulting in the formation of a discontinuous step of approximately 50 nm shown in Fig. 4.37). This local disruption could have important implications in irradiation assisted stress corrosion cracking as it has been hypothesized that grain boundary deformation could be responsible for disrupting surface oxide layers, exposing new material to a corrosive environment [186, 196].
Figure 4.35. Frames taken from a video showing dislocations impinging on a high angle grain boundary during *in situ* deformation of 304 stainless steel at 400°C. Perfect dislocations are seen incoming on multiple parallel plains, resulting in the emission of Shockley partial dislocations as well as cross-slip prior to impingement. The experiment time is given in each frame. Arrows indicate the direction of dislocation motion.
Figure 4.36. Dislocations seen cross-slipping to avoid a high strain buildup at a dislocation interaction site, and cross-slipping again, resulting in impingement on the grain boundary. (a) was taken at an earlier stage of the deformation than (b) and (c). Arrows indicate direction of dislocation motion.
Figure 4.37. Grain boundary/dislocation interaction shown in Figure 4.35 at a later time. The intersection of the boundary plane with the foil surface is extended across the interaction to show the formation of a step in the boundary, measured to be approximately 50 nm.

Application of the $g \cdot b$ invisibility criterion shows the main incoming dislocations have a Burgers vector $b = \pm \frac{a}{2}[011]_i$. Additionally, their character can be deduced to be screw in nature due to their ability to cross-slip in front of the boundary. The single dislocation tangled in the incoming dislocation system had a Burgers vector $b = \pm \frac{a}{2}[101]_i$. The limited emission of perfect dislocations had a Burgers vector $b = \pm \frac{a}{2}[110]_o$ and the dislocation system involving partials dislocations had a Burgers vectors $b = \pm \frac{a}{6}[112]_o$ for the leading partials, and $b = \pm \frac{a}{6}[211]_o$ for the trailing partial dislocations. The partial dislocations reside on the $(111)_o$. 

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EBSD was used to characterize the grain boundary and it was found to be a $15^\circ$ rotation about the $[3\ 27\ 11]_{in}$ direction.

The optimal dislocation system emitted, in terms of minimizing the Burgers vector of the residual dislocation, would be $b = \pm \frac{a}{2}[011]_{out}$. The Burgers vector of the perfect dislocations emitted, $b = \pm \frac{a}{2}[110]_{out}$, left a significant residual dislocation after the interaction and so, in agreement with the slip transmission criteria, was limited to the emission of only a few dislocations. Emission of either $b = \pm \frac{a}{6}[112]_{out}$ or $b = \pm \frac{a}{6}[121]_{out}$ lead partial dislocations would result in creating the minimal residual grain boundary dislocation. A tomogram of the interaction could not be reconstructed, and so it is unknown whether or not the resolved shear stress was the determining factor between the two available systems. The analysis does confirm however, that emission of the $b = \pm \frac{a}{6}[112]_{out}$ lead partial dislocation, the system observed in the interaction, is one of the optimal systems to be emitted. Emission of the trailing partial, however, leads to a significant increase in the magnitude of the Burgers vector of the residual dislocation. Similar to system 2d in Figure 4.22, this explains the presence of the extended faulted region originating at the grain boundary. In Figure 4.22, the complete dislocation (when both leading and trailing partial dislocations are taken into account) generated the dislocation with the smallest residual Burgers vector compared to the other possible reactions. This is not the case in the interaction shown in Figure 4.35, which is contrary to the prediction of the slip transmission criteria. After further straining, a new system activated and became the dominant system (Fig. 4.38). Characterization of this new system (shown in Fig. 4.38a) showed the dislocations to have a Burgers vector $b = \pm \frac{a}{2}[011]_{out}$, which is the system that ultimately creates the residual grain boundary dislocation with the minimal residual Burgers vector.
Using the fiducial method described in the experimental procedure section, a 3D model of the interaction can be created from a series of images collected using the single-tilt deformation stage. This allows for the combination of time-resolved information with the full 3D characterization of the system. Figure 4.39 shows the interaction of incoming partial dislocations with a random high-angle grain boundary, found to be a 36° rotation about the [1122]ₐ island. Images of the interaction were collected at 1° intervals over a tilt range from -29° to +28° and reconstructed into a tomogram, from which a 3D model of the interaction was made. Figure 4.40 displays select micrographs from the tilt series, views of the reconstructed tomogram, and views of the 3D dislocation model. The micrographs have been aligned and fiducial markers included in the image. As the images were collected using a single-tilt TEM holder, the diffraction conditions varied strongly as the sample was tilted. The visualization of the tomogram is shown at a single vantage point but at different intensity levels. During construction of the model, different intensity levels were used to highlight planar defects (Fig. 4.40c) or the fiducial markers to identify the dislocations (Fig. 4.40d).
The incoming dislocations were found to reside in the $\pm \frac{a}{2}[101]_{in}(111)_{in}$ slip system and have a line direction near parallel to [101]$_{in}$ as seen in the model of the interaction. One system was back-emitted into the incoming grain. These dislocations were also found to have Burgers vector $\mathbf{b} = \pm \frac{a}{2}[101]_{in}$ but resided on (111)$_{in}$. The dislocations were absorbed and emitted into the neighboring grain as partial dislocations with an extended faulted region trailing the lead partial. Emission of a lead partial dislocations occurred within 0.1 s of absorption into the boundary, see Figure 4.39b-c. The partial dislocations were found to have a combined Burgers vector $\mathbf{b} = \pm \frac{a}{2}[101]_{out}$ and to reside on the (111)$_{out}$ plane. The Burgers vectors of the individual partial dislocations are $\mathbf{b} = \pm \frac{a}{6}[211]_{out}$ and $\mathbf{b} = \pm \frac{a}{6}[112]_{out}$, however, the information was insufficient to determine which was the lead partial dislocation. An additional system was emitted into the neighboring grain and remained near the boundary, preventing reliable characterization.
Figure 4.39. Dislocations impinging on a high angle grain boundary during *in situ* deformation at 400°C. Arrowhead indicates a dislocation impinging on the grain boundary and emitting into the neighboring grain within 0.1 seconds.

Analysis of the Burgers vector the residual grain boundary dislocation shows that when considering only perfect dislocations, those with a Burgers vector $\mathbf{b} = \pm \frac{a}{2}[101]_{out}$ or $\mathbf{b} = \pm \frac{a}{2}[11$
When partial dislocations are taken into account, \( \mathbf{b} = \pm \frac{a}{6}[211]_{\text{out}} \) leaves the dislocation with the minimum Burgers vector. The trailing partial dislocation, \( \mathbf{b} = \pm \frac{a}{6}[112]_{\text{out}} \), significantly increases \( \left| \mathbf{p}^{gb} \right| \), again explaining the extended faulted region. The combination of the emitted partial dislocations results in the optimal emitted perfect dislocation in terms of minimizing \( \left| \mathbf{p}^{gb} \right| \), \( \mathbf{b} = \pm \frac{a}{2}[101]_{\text{out}} \), so no new dominant system emerged. Figure 4.41 displays a comparison of the relative resolved shear stress on the available partial dislocation systems as well as the normalized magnitudes of the Burgers vectors of the residual boundary dislocations associated with each system. As can be seen, the highest resolved shear stress acts on the partial dislocation with Burgers vector \( \mathbf{b} = \pm \frac{a}{6}[211]_{\text{out}} \). The nearest two optimal systems, \( \mathbf{b} = \pm \frac{a}{6}[211]_{\text{out}(111)} \) and \( \mathbf{b} = \pm \frac{a}{6}[211]_{\text{out}(111)} \), have much lower levels of resolved shear stress acting on them, suggesting that the resolved shear stress can act as a deciding factor between possible dislocation systems that leave residual dislocations with similar Burgers vectors. The trailing partial dislocation emitted from the grain boundary has a significantly smaller resolved shear stress, giving an additional possible reason for the presence of the faulted region extending from the grain boundary.
Figure 4.40. Construction of the 3D dislocation model of the interaction shown in Fig. 4.39. (a) and (b) show aligned micrographs from the tilt series with fiducial markers included. Stage tilt angle is given in each image. (c) and (d) show the reconstructed tomogram from a single vantage point but at different intensity levels to highlight different features. (e) and (f) show views of the 3D dislocation model with the coordinate systems included. The green plane represents the grain boundary and the blue plane represents the faulted region between Shockley partial dislocations. The dislocations are color coded according to Burgers vector.
Figure 4.41. Resolved shear stress and magnitude of the Burgers vector of the residual grain boundary dislocation associated with each potential emitted slip system from the interaction shown in Fig. 4.39. Values are normalized with the highest value set to unity to facilitate direct comparison.

As the interaction progressed, other secondary systems were activated (Fig. 4.42), reflecting the cumulative effect of the residual grain boundary dislocation buildup as dislocations continued to be absorbed and emitted at the grain boundary. These secondary systems were limited in their activity, emitting only a few dislocations compared to the many absorbed and emitted dislocations in the dominant system.
Figure 4.42. Dislocation/grain boundary interaction shown in Fig. 4.39 but at a later stage in the interaction. Arrows indicate the direction of dislocation motion.

Inspection of the same interaction at the grain level, instead of a single interaction, shows how different dislocation systems interact and lead to complex dislocation tangles observed in deformed materials. Figure 4.43 shows the grain into which the dislocations were emitted in
Figure 4.39 both at an early stage of the interaction (a) and at a later point of the interaction when the main dislocation system is nearing the point of crack formation (b). As can be seen in Fig. 4.43a, the initial grain boundary response to dislocation impingement was the emission of lead Shockley partial dislocations with extended trailing faulted regions. Systems were emitted at various locations along the boundary, not necessarily correlated to an impinging dislocation system, with the majority of the dislocation activity concentrated around the region marked ‘A’ which corresponds to the interaction shown in Figure 4.37. It is also apparent in the image that the emitted systems are not coplanar. That is, multiple systems are emitted from the boundary near each other, but offset by some distance (magnified in Figure 4.43a). This suggests that instead of a point on the boundary from which dislocations are emitted, there exists an activated volume from which multiple parallel but not coplanar systems are emitted.

Upon further deformation, the dislocation activity increased throughout the grain, but then became more concentrated around the initial dominant area. Regions of interest are numbered in Figure 4.43 and shown at higher magnification in Figures 4.44 and 4.45. Region 1 emphasizes that the emitted dislocation systems need not correspond to an impinging dislocation system. Dislocation activity at a different point on the boundary can instigate dislocation emission, possibly through dislocation glide in the boundary plane. Region 2 shows the resultant dislocation debris from a previously active system. Figure 4.44a shows region 2 to have previously been the area of partial dislocation glide. Once passed, a residue of dislocation segments populated the edges of the system. That their slip plane is near perpendicular to the foil surface is apparent by the short length of the dislocations. The contrast from the dislocations, black and white circular lobes, suggests that these are screw dislocations viewed end on [197]. To the left of where the dislocations previously passed, the contrast shows the white lobes above
the black lobes. This is reversed on the opposite side of the dislocation pass, suggesting that sign of the Burgers vector is reversed [198]. Region 3 highlights a dislocation gliding on a parallel plane to the dominant emitted systems, but in the opposite direction, evidenced by the trailing slip traces. This is similar to what is shown in Figure 4.42 and demonstrates a mechanism for concentrating plasticity during deformation. The back emitted dislocations, when emitted on a parallel plane, impinge on the boundary at the same location as the dislocations coming from the neighboring grain, thus further increasing the stress at the boundary. This is similar to the behavior seen in Figure 4.4 where dislocations are seen approaching the grain boundary from both directions. Region 4 highlights a dislocation node, created from intersecting dislocations. These nodes can create barriers to dislocation motion, leading to work hardening during deformation. Region 5 shows the standard grain boundary response late in the deformation process. Dislocations transfer across the boundary onto multiple planes in the neighboring grain. There is also significant back emission on different planes, reflecting the complicated stress state at the impingement point.
Figure 4.43. Dislocation evolution in a single grain at an early stage of deformation (a) and later in the deformation (b). The dislocation interaction indicated by ‘A’ in (a) is the same shown in Fig. 4.39. A magnified view of the systems emitted from the boundary is included as an inset in (a). Regions of interest are numbered 1-5 in (b) and shown at higher magnification in Figures 4.44 and 4.45.
Figure 4.43 (cont)
Figure 4.44. Regions 1-4 shown in Fig. 4.43. Regions who 1) dislocations emitted from a dislocation where no dislocations are seen impinging in the neighboring grain, 2) residual dislocation debris from a previously active dislocation system, 3) a dislocation back emitted into the grain on a parallel plane to the impinging dislocations, and 4) a dislocation junction created from intersecting dislocations on non-parallel glide planes. Arrows indicate direction of dislocation motion.
Figure 4.45. Region 5 from Fig. 4.43 showing the dominant dislocation system impinging on a coherent $\Sigma 3$ twin boundary. The interaction results in dislocations transmitting through the boundary as well as multiple slip systems being back-emitted into the grain. Arrows indicated direction of dislocation motion.

4.2.5 Effects of boundary features

As discussed in the background, boundary features such as ledges can lead to dislocation emission and can affect the development of dislocation/grain boundary interactions. Such
behavior was seen experimentally during \textit{in situ} deformation in the TEM. Figure 4.46 shows an area of interaction where an incoming system of perfect dislocations impinged on a coherent $\Sigma 3$ twin boundary. Near the impingement point, visible at the bottom of Figure 4.46 and more clearly in Figure 4.47, the boundary contained a large ledge. Dislocation activity was observed \textit{in situ} in the TEM during straining at a temperature of 400°C. Three different systems were seen emitting from the boundary, one near where the incoming dislocations were impinging on the boundary and two near the ledge in the boundary which involved dislocations emitting into both grains. Initially, only perfect dislocations were emitted from the boundary. There were also other dislocation systems seen impinging on the boundary, indicated in Figure 4.46, but these appeared to have only minor roles in the early stages of the interaction. As the interaction progressed, perfect dislocations continued to be emitted from near the impingement point of the incoming dislocations, but the activity near the boundary ledge increased as well (Fig. 4.47). The dislocations emitting near the boundary ledge consisted of partial dislocations being emitted back into the grain where the incoming dislocations originated as well as extensive emission of perfect dislocations into the neighboring grain. With further deformation, the activity at the boundary greatly increased, with multiple dislocation systems being emitted rapidly from the boundary. The emitted dislocation systems were composed of both perfect and partial dislocations and were seen propagating both quickly and slowly from the boundary.

Diffraction analysis of the interaction showed the boundary, excluding the ledge, to be a coherent $\Sigma 3$ twin boundary lying on (1 11)$_{in}$. The dislocations in the incoming system were found to have a Burgers vector of $b = \pm \frac{a}{2} [101]_{in}$. The emitted systems were not characterized due to the complexity of the interaction. The initial system is similar to that seen in Figure 4.22, which resulted in a glissile partial dislocation remaining in the boundary after emission of a
dislocation system into the neighboring grain. Although not confirmed in the video due to high stress concentrations near the boundary, the activity near the ledge could be a result of the boundary ledge inhibiting glide of the partial dislocations in the boundary plane.

Figure 4.46. BF image showing the early stage of a dislocation/twin boundary interaction during \textit{in situ} straining at 400°C. Arrows indicate the direction of dislocation motion. A large ledge in the boundary is indicated by an arrowhead.
Figure 4.47. Frames taken from a video showing a dislocation/twin boundary interaction during *in situ* straining at 400°C. The direction of dislocation motion is indicated in Fig. 4.46, which shows the same interaction at an earlier stage.

The activity of dislocation systems impinging on the boundary from other sources than the main system seen in Figure 4.48 also increased in activity during later stages of the deformation. These dislocations did not enter in a single slip band as the original system did. Rather, they impinged as individual events on the boundary plane. Each impinging dislocation was associated with the immediate ejection of a leading partial dislocation into the neighboring grain (Fig. 4.48f-h). This is similar to what was seen during the high temperature deformation shown in previous examples. That is, at elevated temperature the barrier strength of the boundary
is significantly lowered, allowing slip transmission before the formation of large dislocation pileups.
Figure 4.48. Same interaction as is shown in Fig. 4.46, but at a later stage of the deformation. Arrowheads in (f-h) indicate locations where an outside dislocation system, evident only by the surface slip traces, impinged on the twin boundary, causing the emission of a partial dislocation into the neighboring grain.
Though not as complex as the previous example, Figure 4.49 provides additional evidence of the effects of grain boundary features on dislocation nucleation. Partial dislocations on a single plane were seen impinging on a grain boundary during *in situ* straining at room temperature. Some evidence of dislocation activity in the boundary plane is evident (indicated in Figure 4.49). These grain boundary dislocations were found only to reside to the right of where the partial dislocation was impinging, suggesting that they originate from where the partial dislocation impinged. Although only one system is seen entering the boundary, partial dislocations were emitted from the boundary at various points. The lead partial dislocation did not extend far into the boundary before emission of the trailing partial dislocation, suggesting a relatively low energy barrier to emission in comparison to the interactions shown in Figures 4.26 and 4.39. The available diffraction information was insufficient to characterize the Burgers vectors of the dislocations, but image contrast suggests that all of the emitted partial dislocations are of the same type and resided on parallel slip planes.
Frames taken from the *in situ* video, Figure 4.50, show that dislocation activity was concentrated both at the point where the incoming dislocation system impinged on the boundary as well as at the sharp bend in the boundary plane. It is interesting to note that whereas only a single partial dislocation was seen entering the boundary during the interaction, multiple partial dislocations were emitted from the boundary plane over the course of the interaction. Also apparent from the *in situ* video is the much slower rate of activity in the room temperature interaction in comparison to the dislocation/twin boundary interaction shown in Figure 4.46.
Figure 4.50. Frames from a video taken during *in situ* straining of 304 stainless steel at room temperature. The same interaction is shown at a later time in Fig. 4.49. (g) and (h) show enlarged views of the areas boxed in (a) and (f), respectively. Arrows denote the direction of dislocation motion and the experiment run time is given in each image.
An enlarged view of the before and after state of the boundary region between the point where the partial dislocation is impinging and the curved portion of the boundary is shown in Figure 4.50g-h. In the initial boundary state, Figure 4.50g, multiple extrinsic grain boundary dislocations are visible. After emission of multiple partial dislocations from the boundary, the population of the extrinsic grain boundary dislocations is significantly reduced. Figure 4.51 shows an enlarged view of a single emission event from the boundary. The images were collected 0.2 s apart at a period between the 4.50d and e. As can be seen, previous to emission the partial dislocation was present as an extrinsic grain boundary dislocation.

A possible scenario explaining the high concentration of the dislocation activity near the curved region of the boundary is that the curved region of the boundary impedes the propagation of the dislocations on the boundary plane. As dislocations enter the boundary, either through cross-slip or as a residual dislocation after slip transmission, they would pile up where the boundary curvature begins. This would lead to a high stress concentration, forcing dislocation emission from the boundary. Though the initial stages of dislocations entering the boundary was not seen during the in situ deformation, the later dislocation activity supports such a sequence of events.
Figure 4.51. Single emission of a lead partial dislocation from a grain boundary. The bottom row shows magnified images of the areas boxed in the top row. The two images are separated by 0.2 s and were collected at a time between Fig. 5.50d and e. A single dislocation is tracked with arrowheads.

Two additional examples of boundary features leading to increased dislocation emission are shown in Figure 4.52 and 4.53. Both images are from datasets collected to investigate the effects of irradiation damage on dislocation activity and were irradiated to a fluence of $3 \times 10^{13}$ ions cm$^{-2}$ using krypton ions \textit{in situ} in the TEM [172]. The materials used in the study were 21Cr32Ni and 13Cr15Ni austenitic stainless steel alloys, Figures 4.52 and 4.53 respectively. The sample shown in Figure 4.52 was irradiated prior to \textit{in situ} straining in the TEM while the other
sample was strained *ex situ* prior to thinning to electron transparency and *in situ* ion irradiation. As such, Figure 4.53 represents an example of concentrated dislocation activity near a boundary ledge during bulk deformation. The *in situ* straining was conducted at a temperature of 400°C.

In Figure 4.52, three separate dislocation systems, marked with arrows, were seen impinging on a single grain boundary heavily populated with ledges. Each incoming dislocation system had a corresponding dislocation system emitting from a boundary which aligned closely with the impingement point. That is, the impact point and emission point of the incoming and outgoing dislocation systems were the same. However, there were an additional four systems emitted from the boundary that did not correlate directly with any one incoming system. Each of these additional systems emitted directly from a ledge in the boundary. The slip bands of the emitted systems which correlated directly to an incoming system have slightly higher intensity in the image, suggesting a higher level of dislocation activity then the emitted systems not correlated with incoming dislocations.
Similarly, dislocation activity was concentrated about the dislocation ledge in the interaction shown in Figure 4.53. A single system composed of perfect dislocations impinged on the boundary plane near the ledge. In response, no dislocations were transmitted into the neighboring grain, but two separate systems were emitted back into the same grain. Both were composed of perfect dislocations and both originated in the boundary ledge. The main emitted system is especially interesting in that there were many more dislocations emitted into the grain than were absorbed into the boundary, suggesting that the ledge itself can act as a dislocation source. This is similar in idea to the mechanism for dislocation nucleation at grain boundaries suggested by Li [85] and Price and Hirth [59]. Studies by Was et al. on the degradation of material properties due to irradiation have identified grain boundary sliding as a significant
contribution to irradiation assisted stress corrosion cracking [186, 196]. Boundary sliding can disrupt the surface oxide layer, exposing more material to the corrosive environment. In the model proposed by Price and Hirth, the emission of screw dislocations from grain boundary ledges is compensated by grain boundary sliding, which, in accordance with the work by Was et al., could contribute to stress corrosion cracking.

Figure 4.53. Dislocations emitting from a boundary ledge in deformed irradiated stainless steel. Arrows denote the direction of dislocation motion.
4.3 INTERACTIONS IN α-TI

4.3.1 Dislocation/grain boundary interactions during ex situ deformation

To investigate dislocation/grain boundary interactions during deformation of bulk materials, deformation was initially performed *ex situ*. Figure 4.54 shows a single dislocation system impinging on a high angle grain boundary and being absorbed into the boundary plane. Contrast in the boundary plane suggests the presence of multiple grain boundary dislocations of at least two types. Potential sources of these dislocations include previously glissile dislocations absorbed from the matrix, residual grain boundary dislocations created as a byproduct of slip transfer across the boundary, or extrinsic grain boundary dislocations interacting with secondary dislocations in the boundary compensating for deviations from a low energy configuration [199]. As the deformation was performed *ex situ*, the dislocation source can only be hypothesized. Dislocations were emitted into the neighboring grain as a complex tangle composed of two different dislocation types. The two different systems are clearly resolvable when imaged under different diffraction conditions, Figure 4.54b-c, and it is also clear that one system, shown in Figure 4.34b, is significantly more active than the other.
Figure 4.54. Dislocation/grain boundary interaction in Ti shown using three different diffraction conditions. Dislocation motion was induced by *ex situ* deformation. Example grain boundary dislocations are indicated by arrowheads in (b).
**Figure 4.55.** Normalized magnitude of the Burgers vector of the residual grain dislocation for all potential emitted dislocations from the interaction shown in Fig. 4.54. The Burgers vectors of the observed emitted dislocations are indicated by arrows.

Geometric analysis of the system showed the incoming dislocations to be \(<c + a>-\text{type with a Burgers vector } b = \pm \frac{a}{3}[1213]_{in}\). The outgoing dislocations were both \(<a>-\text{type with dislocations in the more dominant system, shown in Figure 4.54b, having Burgers vector } b = \pm \frac{a}{3}[1120]_{out}\), and in the less dominant system, shown in Figure 4.54c, having Burgers vector $b = \pm \frac{a}{3}[2110]_{out}$. Grain boundary characterization showed the two grains to be related by a $40^\circ$ rotation.
about the \([1213]_{in}\) axis. The Burgers vector of each potential residual grain boundary dislocation, taking into account all \(<\mathbf{a}>, <\mathbf{c}>,\) and \(<\mathbf{c} + \mathbf{a}>\)-type dislocations, is shown graphically in Figure 4.55. As can be seen, the optimal dislocation type for the system to emit in terms of residual grain boundary dislocation is the \(\mathbf{b} = \frac{a}{3}[1210]_{out}\), which was not seen experimentally. To more accurately reflect the dislocation state seen in the interaction, the Burgers vector of the residual grain boundary dislocation was calculated for the emission of dislocations with Burgers vectors \(\mathbf{b} = \frac{a}{3}[2 \ 110]_{out}\) and \(\mathbf{b} = \pm \frac{a}{3}[112 \ 0]_{out}\) at a 1:2 ratio. In that combination, the magnitude of the Burgers vector of the residual grain boundary dislocation is significantly smaller than that left when only dislocations with a Burgers vector of \(\mathbf{b} = \frac{a}{3}[1210]_{out}\), suggesting that minimization of the Burgers vector of the residual dislocation is still the dominant factor in determining which dislocation system is emitted.

A second dislocation/grain boundary interaction, shown in Figure 4.56, similarly shows dual dislocation systems emitting from a single incoming dislocation system. One tangled system is seen impinging on the boundary plane. Dislocation loops (Fig. 4.56d) and dipoles (Fig. 4.56c) are present in the dislocation tangles leading up to the boundary. There also appears to be a half-loop emitting from the boundary plane at the impingement point (Fig. 4.56d). Two different dislocation systems were seen exiting the boundary into the neighboring grain, each shown individually in Figure 4.56a-b. One of them, shown in Figure 4.56a, is seen to have either emitted earlier from the boundary or separated fully from the boundary plane at a lower stress level. The other system emitted only partially from the boundary as half-loops extending into the grain matrix. There were also a number of scattered dislocations around the interaction point that were thought not to be related to the interaction itself, as well as an unrelated dislocation array impinging on the boundary plane near the interaction point.
Figure 4.56. Dislocations interacting with a grain boundary after ex situ deformation. The arrow indicates the assumed direction of dislocation motion. A dislocation dipole and loop are indicated by arrowheads in (c) and (d), respectively. An enlarged view of the emitting dislocations is given in (b). The diffraction pattern associated with each image is given in each panel.

Diffraction analysis of the system showed the incoming dislocations to have Burgers vector \( \mathbf{b} = \pm \frac{a}{3}[1210]\). The outgoing dislocations shown in Figure 4.56a had Burgers vector \( \mathbf{b} = \pm \frac{a}{3}[2110] \), and the others, shown in Figure 4.56b, had Burgers vector \( \mathbf{b} = \pm \frac{a}{3}[1210] \). The two grains were related by a 32° rotation about the [1 5 6] axis. All other dislocations present in Figure 4.56 were also <a>-type. Comparison of \( |b^\text{gb}| \) for each dislocation Burgers vector available to be emitted into the neighboring grain is shown in Figure 4.57. Both \( \mathbf{b} = \pm \frac{a}{3}[1210] \)
and $\mathbf{b} = \pm \frac{a}{3}[1120]_{out}$ leave small values of $|\mathbf{\rho}_{\mathbf{r}}^{\mathbf{gb}}|$, with $\mathbf{b} = \pm \frac{a}{3}[1210]_{out}$ leaving the slightly smaller of the two. However, in the interaction, dislocations with Burgers vector $\mathbf{b} = \pm \frac{a}{3}[1120]_{out}$ were not seen exiting the boundary plane, but those with Burgers vector $\mathbf{b} = \pm \frac{a}{3}[2110]_{out}$ were. A possible interaction at the boundary could be the emission of dislocations with Burgers vectors $\mathbf{b} = \frac{a}{3}[2110]_{out}$ and $\mathbf{b} = \frac{a}{3}[1210]_{out}$. This has the combined effect of emitting a dislocation with Burgers vector $\mathbf{b} = \pm \frac{a}{3}[1120]_{out}$, which would be the optimal system to emit in terms of minimizing $|\mathbf{\rho}_{\mathbf{r}}^{\mathbf{gb}}|$. An alternative explanation is that the dislocations with Burgers vector $\mathbf{b} = \pm \frac{a}{3}[2110]_{out}$ were emitted for only a short period of time before shutting down due to a high level of resolved shear stress acting on them, after which the system composed of dislocations with Burgers vector $\mathbf{b} = \pm \frac{a}{3}[1210]_{out}$ activated. Both interactions satisfy the slip transfer criteria set forth by Lee et al., though as the deformation was performed ex situ and the signs of the Burgers vectors were not determined, neither scenario could be verified.
Figure 4.5.7. Normalized magnitude of the Burgers vector of the residual grain dislocation for all potential emitted dislocations from the interaction shown in Figure 4.56. The Burgers vectors of the observed emitted dislocations are indicated by arrows, with a referring to the system shown in Fig. 4.56a and b referring to the system shown in Fig. 4.56b.

Figure 4.58 displays a third dislocation/grain boundary interaction, again formed through *ex situ* deformation and inspected *post mortem* using the double-tilt holder. A single dislocation system was seen impinging on a grain boundary. Similar to what is seen in Figure 4.54, multiple dislocations are visible in the boundary plane. In the neighboring grain, three distinct dislocation systems were seen to be active, shown using different diffraction conditions and labeled 1, 2, and
3 in Figure 4.58. System 1 is visible in b and d, system 2 is visible in b and c, and system 3 is visible in c and d. Systems 1 and 2 appear to be emitting from the boundary as a result of the impinging dislocation system. The third system glides on a plane near parallel to the boundary plane and does not appear to be directly associated with the grain boundary/dislocation interaction. Small loops are also evident near the grain boundary where the incoming dislocations are impinging and the curvature of the dislocations before entry into the plane suggests interaction with a small loop or defect cluster.

Figure 4.58. Images of a dislocation/grain boundary interaction in Ti shown using different diffraction conditions with the associated diffraction pattern. (a) shows the grain on the left of the boundary in two beam condition while (c-d) are imaged with the grain on the right of the boundary in two beam condition. An arrow indicates the direction of slip transfer across the boundary. A small loop and a curved portion of the dislocation approaching the boundary is indicated in (a). Example grain boundary dislocations are indicated in (b).
(Fig. 4.58 continued) Three distinct slip systems to the right of the boundary are labeled for reference in the text.

Diffraction analysis of the interaction narrowed the possibilities of the potential incoming system to be composed of $\langle a \rangle$-type dislocations with Burgers vector of either $\mathbf{b} = \pm \frac{a}{\sqrt{3}}[11\bar{2} 0]_{in}$ or $\mathbf{b} = \pm \frac{a}{\sqrt{3}}[12 \bar{1} 0]_{in}$. The outgoing systems were also composed of $\langle a \rangle$-type dislocations, with system 1 having Burgers vector $\mathbf{b} = \pm \frac{a}{\sqrt{3}}[11\bar{2} 0]_{out}$, system 2 having a Burgers vector $\mathbf{b} = \pm \frac{a}{\sqrt{3}}[12 \bar{1} 0]_{out}$, and system 3 having Burgers vector $\mathbf{b} = \pm \frac{a}{\sqrt{3}}[2\ 1\ 10]_{out}$. The grain boundary misorientation was found to be a $27^\circ$ rotation about the $[1\ 5\ 6\ 2]_{in}$ axis.

![Figure 4.59. Example images from a tilt series of the dislocation/grain boundary interaction shown in Fig. 4.58. The tilt axis is aligned such that the left side of the boundary is kept under 2-beam condition throughout the tilt.](image)

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A tilt series of the interaction was collected by aligning the tilt axis with the \([01\overline{1}1]_m\) direction and collecting an image every 2° over a tilt range of 82° (Figure 4.59). This kept the grain containing the incoming dislocation system under a 2-beam BF condition, but the dislocations in the neighboring grain could not be resolved due to strong contrast fluctuations. The acquired images were reconstructed into a tomogram in which a real space 3D coordinate system was oriented to match the lattice vectors (Fig. 4.60). Due to large available tilt range and the simplicity of the interaction, construction of a 3D dislocation model was not necessary to clearly resolve the interaction.
Figure 4.60. Visualization of the tomogram of the interaction show in Fig. 4.58. (c) shows the slip plane of the dislocations and (d) shows the line direction.

Identification of the slip planes using the tomographic reconstruction allowed the complete characterization of the incoming slip system. As is readily evident in the tomogram visualization (Fig. 4.60), the dislocations reside on the (1 010)$_m$ prismatic plane and have line direction near parallel to [12 10]$_m$. The possible Burgers vectors of the incoming dislocation were already narrowed to two possibilities and the added information allows the definitive characterization of the incoming dislocations as having Burgers vector $\mathbf{b} = \frac{a}{3}[12 10]_m$, making them pure screw in nature. Combined with the grain boundary characterization, the information on the resultant $|\mathbf{b}_{r^{gb}}|$ becomes available and is plotted in Figure 4.61 with systems 1 and 2.
labeled. System 2 is the optimal system in terms of minimizing $|\rho_{s}\rho_{s}^{*}|$, but system 1 increased the elastic strain energy density.

As the incoming dislocations were fully characterized, the resolved shear stress on the emitted systems could be estimated. This information is shown graphically in Figure 4.62. Although the slip planes of the emitted dislocations were not identified, the dislocation systems with the same Burgers vector as the observed systems can be compared. As can be seen, of the $<a>$-type dislocations, those with Burgers vector $b = \pm \frac{\alpha}{3}[12\ 10]_{out}$, the same Burgers vector as the system 2 dislocations, have the highest resolved shear stress. Two-$<c+a>$ type systems have equally high levels of resolved shear stress, but were not seen active in the interaction. Dislocations with Burgers vector $b = \pm \frac{\alpha}{3}[2\ 110]_{out}$ had significantly lower values of resolved shear stress acting on them, but were still seen to be active in the interaction.
Figure 4.61. Normalized magnitude of the Burgers vector of the residual grain dislocation for all potential emitted dislocations from the interaction shown in Fig. 4.58. The Burgers vectors of the observed emitted dislocations are indicated by arrows.
During *ex situ* deformation of Ti, low angle grain boundaries were commonly formed throughout the material and acted as barriers to dislocation motion. An example interaction between dislocations and a low angle grain boundary is shown in Figure 4.63. The impinging dislocations do not form a traditional pileup, but appear as a tangled mass before the boundary. Nearer to the boundary, the density of the tangles increases, indicating that the low angle...
boundary does inhibit dislocation glide. Dislocations appear to have transferred into the neighboring grain, but again, this did not occur on any single plane nor did the dislocations follow any single direction of motion. Diffraction analysis of the interaction showed that the dislocations on both sides of the boundary had equivalent Burgers vectors. The misorientation across the boundary, as estimated by the diffraction patterns collected and the tilt differential between equivalent two-beam conditions on either side of the boundary, was below 1°. Considering the small magnitude of the misorientation across the boundary, to minimize \( b_s^s b \) the Burgers vector should not change as the dislocations transfer across. This does, however, require some small residual dislocation to remain in the boundary after slip in order for the Burgers vector to be fully conserved.
Figure 4.63. Dislocations interacting with a low angle grain boundary after ex situ deformation. The arrow indicates the assumed direction of dislocation motion.

4.3.2 Dislocation/grain boundary interactions during in situ deformation

Grain boundary/dislocation interactions were observed at room temperature during in situ straining in the TEM. In the first interaction, shown in Figure 4.64, dislocations emitted from a crack tip were seen impinging on a high angle grain boundary. The dislocations did not progress
in defined slip bands, but instead advanced as individuals. The stress level in the neighboring grain was seen to increase with the number of impinging dislocations, evident by the localized contrast near the boundary. As the interaction progressed, dislocation interactions before impingement on the boundary were seen to increasingly dictate dislocation behavior. Cross-slip led to the formation of helical-shaped dislocations as well as wavy slip traces on the surfaces, both of which are indicated in Figure 4.65b. The increasing stress state at the boundary eventually led to dislocation emission into the neighboring grain, evidenced by the slip traces visible in Figure 4.66b-d. The dislocations were emitted on parallel planes, but were not coplanar, and propagated rapidly from the boundary. This sporadic emission of dislocations continued even as the majority of the slip became concentrated in one location, shown in Figure 4.67, which led to a high level of local disruption in the boundary. Similar to what was seen during deformation of stainless steel (Fig. 4.37), the emitted dislocations caused the formation of a step or discontinuity in the boundary with a height of approximately 70 nm. Except for a few sparsely distributed dislocations, the boundary structure on either side of the interaction remained undisturbed, suggesting the strain accommodation in the boundary was highly localized.
Figure 4.64. Frames from a video showing dislocations impinging on a grain boundary in Ti. The stress buildup in the neighboring grain is indicated by an arrowhead and experiment time is recorded in each image.
Figure 4.65. Same interaction as that shown in Fig. 4.64, but at a later time. Cross-slip near the boundary is evident from wavy slip traces, indicated by arrowhead 1, and helical shaped dislocations, indicated by arrowhead 2.
Figure 4.66. Same interaction as is shown in Figures 4.64 and 4.65, but at a later time. Slip traces in the neighboring grain indicate dislocation emission from the boundary.
Figure 4.67. Local disruption of a grain boundary in Ti from slip transfer. The grain boundary is the same as is shown in Fig. 4.64, but at a different location. A step was formed in the boundary and is highlighted by using white bars to extend the boundary/foil surface intersection line across the interaction zone. An example dislocation in the boundary plane is indicated by an arrowhead.

Diffraction analysis of the interaction, done at a point removed from the main interaction to avoid the highest stress levels, showed the incoming dislocations to have Burgers vector \( \mathbf{b} = \pm[1120]_{in} \). The emitted dislocations had Burgers vector \( \mathbf{b} = \pm[1120]_{out} \). The grain boundary was found to be a 46° rotation about the \([8\ 20\ 12\ 5]\) axis. Calculation of \( |\mathbf{b}_r^{gb}| \) for all available dislocation types emitted into the neighboring grain is shown graphically in Figure 4.68. As can be seen, the Burgers vector of the dislocations emitted does minimize \( |\mathbf{b}_r^{gb}| \).
Figure 4.68. Magnitude of the Burgers vector of the residual grain boundary dislocation associated with each potential emitted slip system from the interaction shown in Fig. 4.64. Values are normalized with the highest value set to unity to facilitate direct comparison. The observed emitted system is indicated by an arrow.

Figure 4.69 displays a second grain boundary/dislocation interaction in Ti, again observed during in situ straining at room temperature in the TEM. Unlike the previous example, dislocations approach the grain boundary in defined slip bands and form pileups at the boundary. A significant number of dislocations were absorbed into the boundary plane before any slip
transmitted across the plane. Tangled dislocations were seen further from the boundary. These came from a different grain boundary source not in the viewing area. Unlike the previous example, the emitted dislocations emerged slowly from the boundary and only after a significant period of time did they fully detach and propagate from the boundary plane.
Figure 4.69. Dislocations transmitting across a grain boundary during *in situ* room temperature straining of $\alpha$-Ti. The two images are of the same grain boundary but were collected at different times and using different diffraction conditions. The arrow denotes the direction of dislocation motion.
Analysis of the system showed the incoming dislocations to have Burgers vector $\mathbf{b} = \pm [2 110]_{in}$ and the outgoing dislocations to have Burgers vector $\mathbf{b} = \pm [2 110]_{out}$. The two grains were related by a $9^\circ$ rotation about the $[5 2 7 2]_{in}$ axis. $|\mathbf{b}_r^{gb}|$ for all available systems is displayed in Figure 4.70. As can be seen, the interaction does minimize $|\mathbf{b}_r^{gb}|$.

Figure 4.70. Magnitude of the Burgers vector of the residual grain boundary dislocation associated with each potential emitted slip system from the interaction shown in Fig. 4.69. Values are normalized with the highest value set to unity to facilitate direct comparison.
(Fig. 4.70 continued) The Burgers vector of the observed emitted system is indicated by an arrow.

Coupling of dislocation dynamics in Ti with electron tomography was first achieved by deforming a 3 mm Ti disk \textit{ex situ} but after thinning to electron transparency and loading the sample immediately into the TEM. By quickly loading the sample into the TEM chamber, the sample could be observed before the cessation of dislocation motion. That the observed dislocation and twin activity was due to newly nucleated dislocations was confirmed by observations made prior to deformation which showed few dislocations in the Ti matrix. Figure 4.71 shows frames from the video collected in which dislocations transfer across a coherent twin boundary. The boundary posed almost no barrier to the dislocation motion with no pileups forming before transmission occurs. The dislocations did cross-slip onto a different plane in the neighboring grain, causing a slight change in direction of motion. The easy transfer of slip is shown more clearly in Figure 4.72 where it can be seen by the absence of strain contours present in Figures 4.64-4.67, no significant dislocation buildup is needed before slip transfer.
Figure 4.71. Frames from a video taken during \textit{in situ} deformation showing dislocations transmitting across a twin boundary in $\alpha$-Ti. Experiment elapsed is given in each image. The arrow indicates the direction of dislocation motion and the arrowhead tracks a single dislocation as it transmits across the boundary.
Figure 4.72. Dislocations transmitting across a twin boundary in $\alpha$-Ti showing low levels of stress at the dislocation/boundary interaction point.

Analysis of the surrounding area shows that the induced deformation caused the formation of a twin which extended approximately 15 $\mu$m into the grain (see Figure 4.73 for a secondary electron image collected in an SEM as well as a montage of TEM micrographs showing the twin). The observed dislocation activity was instigated by a high level of stress in the sample located around a bend in the foil, evident by the curved path of the dislocations leading away from the site of dislocation nucleation (Fig. 4.74). This is different from previous experiments where dislocation activity was seen mainly as a result of emission in front of a propagating crack tip.
Figure 4.73. Twin extending into a grain in $\alpha$-Ti imaged using bright-field TEM (a) and secondary electron imaging in an SEM (b). A nearby grain boundary is indicated by an arrowhead in (b) and the approximate location of the interaction shown in Figures 4.71 and 4.72 is indicated by an arrow.
Figure 4.74. Dislocations nucleating from a twin boundary in α-Ti. Dislocations are shown nucleating and propagating in a twinned region (a) as well as back into the matrix (b). Direction of motion is indicated by arrows. The curved path of the dislocations in (b) indicates a bent foil.
The Burgers vector of the dislocations and the twin boundary type were found using diffraction analysis. Both the incoming and outgoing dislocations were $<a>$-type with Burgers vector $\mathbf{b}_\text{in} = \pm \frac{a}{3}[2\ 1\ 10]$ and $\mathbf{b}_\text{out} = \pm \frac{a}{3}[1\ 1\ 2\ 0]$, respectively. The twin boundary over which the slip transfer observed resided on the $(0\ 1\ 1\ 2)_\text{in}$ plane and had a misorientation of $108.5^\circ$ about the $[2\ 0\ 2\ 1]_\text{in}$ axis. Rotating the coordinate system used to express the emitted dislocations into the coordinate system of the incoming dislocations gives $\mathbf{b}_\text{out} = \mathbf{b}_\text{in} = \pm \frac{a}{3}[2\ 1\ 10]$, which allows the dislocations to transfer across the boundary without leaving a residual dislocation.

As shown with the characterization of coherent $\Sigma 3$ boundaries in stainless steel (Fig. 4.20), dislocation activity on both sides of a twin boundary can be captured simultaneously by aligning the tilt axis with the normal of the plane shared by the twinned and matrix regions. In the current example, the twin boundary lies on the $(0\ 1\ 1\ 2)_\text{in}$, which should allow similar characterization. However, as the incoming dislocations had Burgers vector $\mathbf{b}_\text{in} = \pm \frac{a}{3}[2\ 1\ 10]$, alignment of the tilt axis with the twin plane normal resulted in the incoming dislocations being invisible. To overcome this issue and achieve 3D characterization of the system, two separate tilt series were acquired, each maintaining a different side of the boundary in a dynamical two-beam condition. This approach is similar to that developed by Liu et al. when characterizing dislocations in a single grain but with different Burgers vectors [176]. For the emission side of the boundary, images were acquired at $2^\circ$ intervals over a range of $62^\circ$ while maintaining the tilt axis parallel to the $[0\ 1\ 1\ 1]_\text{out}$ direction. Images were acquired in the twinned region, the region which contained the incoming dislocations, over a range of $56^\circ$ while maintaining the tilt axis parallel to the $[1\ 0\ 1\ 1]_\text{in}$ direction, again with images collected every other degree.
Figure 4.75. Tomogram visualizations (a-b) and 3D-model (c-d) of the dislocation/twin boundary interaction shown in Fig. 4.72. The two columns are two separate tomograms reconstructed from tilt series acquired on either side of the twin boundary.

Once collected, the images were used to reconstruct tomograms in the same manner as described for previous 3D characterizations (Figure 4.75a-b). A dislocation model was constructed from each tomogram, focusing only on those dislocations which were kept in contrast during image acquisition (Figure 4.75c-d). Once constructed, the two models were combined using the UCSF Chimera program for visualization and model manipulation (Fig. 4.76). Faint contrast from dislocations not kept in contrast during image acquisition was still visible in both tomograms, allowing precise alignment of the two models in relation to each other. Coordinate systems were placed on each side of the boundary separately to reflect the lattice rotation.
Figure 4.76. 3D-dislocation model of the interaction shown in Fig. 5.72. The model was constructed by combining the two models shown in Fig. 4.75c-d.

Inspection of the 3D model shows the incoming dislocations reside on the (01 11)\textsubscript{in} and have a line direction parallel to [21 1 0]\textsubscript{in}, making them pure screw in nature. The outgoing dislocation resided on the (11 00)\textsubscript{out} and have a line direction parallel to [1 1 20], making them also screw in nature. The model shows that the line directions of the dislocations on either side of the boundary are parallel to each other. According to Lim and Raj [200] and Lee et al. [9], continuous slip across a grain boundary requires the lines of intersection between the incoming and outgoing slip systems with the boundary to be parallel, the dislocation line directions and
Burgers vectors to be parallel, and the dislocation line direction to be parallel to the line of intersection of the slip plane with the boundary. As seen by the information provided by the diffraction analysis and 3D-dislocation models, the three conditions are satisfied, which is in agreement with the observed flow of dislocations across the boundary plane.

4.3.3 Dislocation/dislocation interactions

_In situ_ straining of Ti foils allows dislocation/dislocation interactions to be recorded in real time. Figure 4.77 shows a double cross-slip event leading to dislocation generation in α-Ti strained _in situ_ at room temperature (also shown schematically in Fig. 4.78). The dislocation indicated in Figure 4.77a was seen gliding on a single plane originally. An unknown obstacle forced half of the dislocation to undergo a double cross-slip event (Fig. 4.77b). The applied stress caused the expansion of this cross-slipped portion, eventually leading it to come in contact with both foil surfaces, resulting in three distinct dislocation segments. That the dislocations reside on different planes is evident by the displacement of their slip planes, as indicated in Figure 4.77g. Figure 4.77h labels the planes on which the ends of the dislocations reside as 1, 1’, 2, and 2’, the numbers referring to the end of the dislocation and the ‘ referring to the displaced plane as opposed to the original glide plane. Considering the lower end of the dislocations, labeled 1, the middle and back dislocations reside on the displaced while the front dislocation resides on the original glide plane. At the upper end of the dislocation, the front and back dislocations reside on the original plane while the middle dislocation resides on the displaced plane. For this to be possible, the back dislocation must still have a jogged segment, inhibiting easy glide and potentially leading to later cross-slip events.
Figure 4.77. Frames taken from a video during *in situ* straining of α-Ti at room temperature showing dislocation generation through double cross-slip. Arrows show the direction of motion. The dislocation of interest is indicated in the first panel. Numbering in the final panel is for reference in the text. (g) highlights the slip traces of two of the dislocation segments, showing that they reside on different slip planes. The experiment time is given in each panel in seconds.
The double cross-slip interaction is shown schematically in Figure 4.78. The dislocation initially is gliding on a single plane, labeled plane 1 in Figure 4.78 (Fig. 4.78 - 1, Fig. 4.77a). An unseen obstacle causes a portion of the dislocation to undergo a double cross-slip event. This forces the dislocation to glide on two parallel planes with a sessile portion connecting the two (Fig. 4.78 - 2, Fig. 4.77b). As the dislocation continues to glide through the matrix, the portion of the dislocation on plane 1 expands towards the foil surface (Fig. 4.78 - 4, Fig. 4.77c). The expanding section eventually contacts the foil surface, breaking the dislocation into two separate dislocations; a dislocation gliding on plane 1, and a half-loop expanding from the foil surface with segments on both plane 1 and plane 2 (Fig. 4.78 - 5, Fig. 4.77d). The half-loop expands towards the opposite foil surface (Fig. 4.78 - 6, Fig. 4.77e-f), eventually making contact and splitting into two separate dislocations. The end result of the interaction is three distinct dislocations, one gliding on plane 1 in the same direction as the original dislocation, a second gliding on plane 2, also in the same direction as the original dislocation, and a third gliding in the opposite direction with portions glissile on both planes and connected by a sessile segment or series of jogs (Fig. 4.78 - 7, Fig. 4.77g-h).
A 3 mm Ti disk was deformed ex situ after jet-polishing to electron transparency to induce similar dislocation interactions to those shown in Figure 4.56. The interaction is shown in Figure 4.79. Two different dislocation systems intersect, with loops and helical portions similar to those seen in Figure 4.77 evident in one of the dislocation systems, visible in Figure 4.79b. Diffraction analysis of the dislocations showed that dislocations visible in Figure 4.79b had a Burgers vector of $b = \pm a/3[1210]$. The other dislocations, visible in Figure 4.79c, had a Burgers
vector of $b = \pm^{2}/3[112 \ 0]$. A tilt series of the interaction was obtained in the TEM using a double-tilt holder and collecting an image every degree over a tilt range of -40 to 42° while maintaining the tilt axis parallel the (101 1) plane normal (Fig. 4.80a-b). A tomogram of the interaction was then reconstructed (Fig. 4.80c-d), which was used as the basis for constructing a 3D model of the interaction (Fig. 4.81). Due to the selected diffraction condition, only those dislocations visible in Figure 4.79b were included in the tomogram.

Using the 3D-dislocation model of the interaction, it can be seen that the majority of the dislocations have a line direction parallel to [12 10] (Fig. 4.81c). The primary glide plane is the (101 1) (Fig. 4.81d), though slip is also evident on the basal and second order pyramidal plane (101 2) (Fig. 4.81b). The two dislocation segments of the helical shaped dislocation are connected by a cross-slipped segment on (101 0) (Fig. 4.81c). Small dislocation loops, represented as green spheres in the model, are seen evenly distributed throughout the matrix.
Figure 4.79. Dislocation interactions in $\alpha$-Ti shown using three different diffraction conditions. A the same helical portion of a dislocation is indicated in (a) and (b) as well as a dislocation kink in (b).
Figure 4.80. Tilt series (a-b) and reconstructed tomogram visualization (c-d) of the dislocation interaction shown in Fig. 4.79. Stage tilt angle and diffraction vector is given in the images from the tilt series.
Figure 4.81. 3D-dislocation model of interaction shown in Fig. 4.79. The helical shaped dislocation is indicated in (c).

A different area of the same sample shows the prevalence of the formation of the helical shaped dislocations (Fig. 4.82). In Figure 4.82a, dislocations are seen by their curvature to be traveling in both directions, similar to the result of the interaction shown during *in situ* deformation in Figure 4.77h. There were no visible obstacles to the dislocation motion visible in the matrix, ruling out dislocation/dislocation intersections as the sole cause of the cross-slip behavior.
Figure 4.82. Dislocation structure of ex situ deformed Ti. The deformation was induced after thinning to electron transparency. Arrows indicate direction of dislocation motion, and arrowheads indicate cusps and helical sections of dislocations similar to what was seen during in situ deformation (Fig. 4.77).
Dislocation arrays were commonly found throughout the Ti samples investigated. These arrays acted as weak barriers to dislocation motion and could be an important source of work hardening. Glissile dislocations interacting with such an array during in situ straining at room temperature are shown in Figure 4.83. The glissile dislocation bowed slightly as it intersected the immobile dislocation (Fig. 4.83b). As the glissile dislocation passed through the dislocation array, small loops and half loops separated from the dislocation remained in the matrix as dislocation debris (the remnant dislocation debris from the passage of the dislocation is indicated in Figure 4.83f). The mechanism for generating the half-loops is assumed to be similar to that shown in Figure 4.77, but with the double cross-slip event occurring near the foil surface. Generation of full loops in the matrix interior would require that only the middle portion of the dislocation underwent a double cross-slip event. Trailing glissile dislocations interacted similarly with the dislocation array, again showing slight bowing indicative of the barrier strength of the dislocations (Fig. 4.83e). \( \mathbf{gb} \) analysis of the interaction showed the gliding dislocations to have Burgers vector \( \mathbf{b} = \pm \frac{a}{3}[11\bar{2}0] \). The immobile dislocations had Burgers vector \( \mathbf{b} = \pm \frac{a}{3}[2\ 110] \).
Figure 4.83. Glissile dislocations interacting with a dislocation array in α-Ti during \textit{in situ} straining at room temperature. The arrow in (a) indicates the direction of dislocation motion. Dislocation debris created by the gliding dislocation is indicated by arrowheads in (f). The experiment time is given in each panel in seconds.

To gain a greater understanding of the interaction, a tomogram of the event was also created. As the observations were made during \textit{in situ} straining, only one axis of stage tilt was
available, necessitating the use of the fiducial marker method. Figure 4.84 shows an image of the interaction, as well as example images from the tilt series with the digital markers placed for alignment and reconstruction purposes. The reconstructed tomogram and dislocation model, Figure 4.85, show the main gliding dislocations, colored green in the model, to slip on and near (11 00) and have a line direction near parallel to [112 0], making them pure screw dislocations gliding on the prismatic plane. The cross-slipped segments glided on (11 01), or the pyramidal plane. It should be noted, however, that the glide planes were not precise. That is, the dislocations appear in the tomogram to deviate somewhat from their slip planes. This behavior is similar to what was seen by Naka and LaSalmonie during in situ straining of Ti and was attributed to thin film effects [125]. Some of the dislocations also exhibited the reverse behavior; the dislocations were glissile on a pyramidal plane and cross-slipped onto a prismatic plane. The immobile dislocations, colored red in the model, resided on (12 10), making them sessile in agreement with observations from the in situ straining experiments. The green dislocations interspersed among the red dislocations are also immobile and reside on a non-slip plane. Other features of note in the tomogram include numerous small dislocation loops interspersed evenly throughout the foil (represented as green spheres in the model), half-loops connected to the foil surface and, and kinks forming on the gliding dislocations.

Full characterization of the dislocations enables identification of the changes to the gliding dislocations from intersection with the sessile dislocations. The intersection of the two dislocations would result in a jog forming on the gliding dislocations with line direction parallel to the [2 110] direction. These jogs would still be glissile on the basal plane, but would require climb and the emission of vacancies for glide on the prismatic or pyramidal planes, thus impeding further propagation of the dislocations.
Figure 4.84. Glissile dislocations interacting with a dislocation array in α-Ti during *in situ* straining at room temperature and example images taken from a tilt series of the interaction. Frames from a video taken at an earlier stage of the interaction are shown in Figure 4.83.
Figure 4.85. Tomogram visualization and 3D-dislocation model of interaction shown in Fig. 4.84 shown from different vantage points. Kinks in the dislocations are indicated in (d) as well as an enlarged view of a dislocation half-loop attached to the foil surface. Small loops are represented by green spheres in the model.
Annihilation of colliding dislocations was also observed during *in situ* straining of the Ti. An example of this is shown in Figure 4.86 which was taken during *in situ* straining at room temperature with the observation area being the same as is shown in Figure 4.83, but at a later stage of the interaction. The initial and final state of the interaction is also shown schematically in Figure 4.87. A dislocation half-loop was seen expanding towards the foil surface (Fig. 4.86a). This half-loop was generated in the same manner as is shown in Figure 4.77 (panel f of Figure 4.77 shows the dislocation at approximately the same stage of the interaction). Upon coming in contact with the opposite foil surface, the dislocation separated into two segments, each traveling in opposite directions (Fig. 4.86c). The segment traveling to the right immediately came in contact with a dislocation traveling in the opposite direction. Both were \(\langle a\rangle\)-type dislocations with identical Burgers vectors but opposite line directions. That they were gliding on two parallel planes is evident by extending the surface slip traces in Figure 4.86c. Once they came in close proximity to each other, both remained immobile for approximately one minute, although further strain was applied to the sample during that time and other dislocations were seen to continue gliding through the matrix. Between images d and e, it was seen that both dislocations very quickly, within \(1/10^{th}\) of a second, annihilated, leaving’ half-loops at either surface (shown enlarged in Figure 4.86f). This would require the middle sections of the dislocations to undergo a cross-slip or climb event, leaving the half loops behind to connect the planes. Similar half-loops are seen in the 3D-dislocation model of the previous interaction (Fig. 4.85). These half-loops were glissile on their resident planes and continued to propagate slowly and in opposite directions. They were also seen to become smaller, that is, they partially escaped through the foil surface, though neither disappeared entirely. It is interesting to note that the same interaction shown in Figure 4.77 which led to dislocation generation can also
lead to dislocation annihilation as it creates dislocations with line and propagation direction opposite to the majority of the glissile dislocations.

Figure 4.86. Annihilation of dislocations in α-Ti during in situ straining at room temperature. The arrowheads in (a) indicate the interacting dislocations. The slip traces of
(Fig. 4.86 continued) the dislocations with the foil surface are extended in (c), showing that they are gliding on parallel planes. Arrowheads in (e) indicate dislocation half-loops remaining after the annihilation. An enlarged view of one of the half-loops is shown as an inset in (f).

Figure 4.87. Schematic of the initial (1) and final (2) state of the interaction shown in Figure 4.86. Arrows indicate the direction of dislocation motion and arrowheads show the line direction of the two dislocations.
CHAPTER 5

DISCUSSION

5.1 DISLOCATION/GRAIN BOUNDARY INTERACTIONS IN STAINLESS STEEL

5.1.1 Comparison to slip transfer criteria

In agreement with the slip transfer criteria developed by Lee et al. [9, 77], it was found that the magnitude of the Burgers vector of the residual grain boundary dislocation was the most important factor dictating which slip systems were activated in response to the impingement of dislocations on a grain boundary. There were no cases where the resolved shear stress acting on the emitted dislocation system was insufficient for propagation of the dislocations from the boundary.

There were a number of situations in which the emission of dislocations with two different Burgers vectors into the neighboring grain would result in grain boundary dislocations with identical or similar Burgers vectors (Fig. 4.18, 4.22, 4.32). In these cases and for simple interactions, estimates of the stress state near the boundary showed that the system with the higher resolved shear stress was activated, suggesting that the resolved shear stress can act as a deciding factor in determining the activated slip system. For more complex interactions, such as that shown in Figure 4.22, a more sophisticated model of the local stress state is needed.
the Burgers vector of the emitted dislocations can be uniquely predicted by considering $|b_r^{eb}|$, two systems yet remain which equally satisfy the Burgers vector minimization criterion as there are two conjugate planes for each Burgers vector. This issue, and the need for a shear stress criterion, is amplified in cases of dislocation impingement on coherent $\Sigma 3$ twin boundaries. This situation is shown in Figures 4.21 and 4.24 in which consideration of the Burgers vector alone leaves four different candidate slip systems for emission from the boundary. In these cases, the correct system can be uniquely predicted when considering the resolved shear stress acting on the available systems after the systems have been narrowed to those which minimize $|b_r^{eb}|$.

The angle made between the line of intersection of the incoming and outgoing slip planes on the boundary plane was not investigated, but was not necessary to uniquely predict the emitted slip system. These criteria held true independent of temperature up to 400°C, boundary type, and dislocation type as they were investigated for pure edge, pure screw, and mixed character dislocations as well as for both perfect and partial dislocations. The criteria were also found to hold true in ex situ deformation of bulk materials and in situ deformation of thin films.

The original papers published by Lee et al. did not investigate the applicability of the slip transmission criteria to the emission of partial dislocations [9, 77]. In this study, it was shown that the lead dislocation of a pair of Shockley partial dislocations follows the slip transmission criteria. An increase in the $|b_r^{eb}|$ by the trailing partial dislocation results in the delayed emission of the trailing partial dislocation and a faulted region extending from the grain boundary. If the combined effect of the Shockley partial dislocations does not minimize $|b_r^{eb}|$, the system activated initially becomes less active and another system which minimizes $|b_r^{eb}|$ becomes active.
That is, for the initial grain boundary response, only the lead partial dislocation need be considered, but the long-term response is governed by the combined effect of the emission of the leading and trailing partial dislocations. Dewald et al. similarly concluded from computational molecular dynamics simulations that only the lead partial dislocation in an interaction should be considered and attributed extended faulted regions to the difficulty of nucleating a trailing partial dislocation [8, 80, 81]. As only the initial stages of the grain boundary/dislocation interaction were simulated, they did not observe a transition in the response at the boundary to include the effect of the trailing partial dislocation.

The role of the resolved shear stress is somewhat reduced in application to partial dislocations as they do not have two conjugate planes to select from. Characterization of the Burgers vector of a partial dislocation alone is sufficient to uniquely predict the activated slip system. In cases where two different potential partial dislocation systems leave similar residual boundary dislocations, the resolved shear stress criterion appears to have a similar effect as it does on perfect dislocations; that is, it can act as a differentiating factor between two systems, with the system with the higher resolved shear stress being more likely to activate. However, as the residual grain boundary dislocation criterion was insufficient to uniquely predict the emitted slip system for only one of the described interactions involving partial dislocations, shown in Figure 4.35, the role of the resolved shear stress on partial dislocations cannot be stated conclusively here.

Additional slip transmission criteria were proposed by Dewald et al., including the minimization of the boundary step created by the slip transmission and minimization of the shear and compressive stresses on the boundary, stating that they were needed in order to accurately predict the critical stress needed for dislocation nucleation at the boundary [8, 80, 81]. Their
criteria, however, were applied only to the early stages of slip transfer and to low-sigma grain boundaries, specifically Σ3, Σ9, and Σ11. The analysis shown here highlights the variations the interaction can experience with extended deformation, achieving temporal scope that is not available to atomistic simulations. For example, the progression of different system emissions from the boundary shown in Figure 4.35-4.37 would not likely be captured in an atomistic simulation. Variations with time include the reduction of local order in the grain boundary, as is evident in Figure 4.36c, and the increase in elastic strain energy density due to imperfect transfer of the Burgers vector across the boundary. These effects may have an important effect on the boundary stress state, potentially altering the criteria proposed by Dewald et al.

Additional criteria proposed by Bachurin et al. emphasized the importance of the sign of the Burgers vector of the incoming dislocations on the interaction [79]. As the sign of the Burgers vector was not characterized, their criteria could not be verified directly though given the complexity and diversity of the interactions occurring at grain boundaries, it is reasonable to assume that factors beyond those considered in this study play an important role. Bachurin et al. also noted that they observed no nucleation of trailing partial dislocations from high angle grain boundaries; only the lead partial emitted. This was found not to be in agreement with the experimental results (Fig. 4.35-4.37).

5.1.2 Σ3 twin boundaries

The present study shows that for screw dislocations interacting with Σ3 grain boundaries, the nature of the interaction depends on the relationship between the slip plane and the twin plane; i.e. is the twin plane the conjugate plane of the slip plane. If it is, the dislocations cross-
slip onto and propagate along it. Molecular dynamics simulations suggest upon impingement of screw dislocations on a coherent twin boundary, one of two reactions can occur. The dislocations can split into twin Shockley partial dislocations and propagate in opposite directions along the boundary plane or they can be transferred into the neighboring grain via a cross slip mechanism, with the reaction pathway being determined by the differences between the unstable stacking fault energy, the stacking fault energy, and the energy barrier to create a faulted region on a twin plane [201]. This differs somewhat from the interactions shown in Figures 4.27-4.29 in which the dislocations on entering the twin boundary retained their lattice Burgers vector and moved in one direction only. At a later period in the interaction, after multiple dislocations had cross-slipted onto the twin plane, the reaction changed to the emission of dislocations from the boundary. In the computational studies, the splitting of the dislocation into partial dislocations glissile on the twin plane caused twin boundary glide with the passage of each dislocation resulting in the displacement of the boundary by one Burgers vector. The glide of perfect dislocations in the boundary should result in the formation of a step in the boundary at the point of impingement. However, the interactions observed in this study evolved from dislocation cross-slip onto and propagation along the twin plane to transmission across the twin boundary before a noticeable step was created.

Jin et al. simulated both copper and aluminum with 60° mixed character dislocations impinging on a twin boundary which was not a conjugate slip plane [99]. The resulting interactions were found to be dependent of both the material and the sign of the Burgers vector of the impinging dislocations, which are given parenthetically after each interaction description:
1) The dislocation transmits through the boundary as two separate partial dislocations, leaving an additional partial dislocation glissile on the boundary plane which propagates from the impingement point (copper with a positive Burgers vector).

2) Only a lead partial dislocation is emitted from the boundary and a sessile dislocation with a Burgers vector \( b = \frac{1}{3}[001] \) and a mismatch of \( \frac{1}{6}[111] \) is left in the boundary. This defect was referred to by Jin et al. as an “i-type” twin lock or “i-lock” as it contains one row of additional interstitial atoms (copper with a negative Burgers vector).

3) The dislocation is absorbed into the boundary and a partial dislocation, glissile in the boundary plane, propagates away from the interaction. The remaining partial dislocation remains sessile in the boundary plane (aluminum with a positive Burgers vector and a low applied stress).

4) The dislocation transmits through the boundary and a single partial dislocation propagates away from the interaction point (aluminum with a positive Burgers vector and a high applied stress).

5) The dislocation is absorbed into the boundary, two partial dislocations propagate away from the interaction point in the boundary plane, and a dislocation with Burgers vector \( b = \frac{1}{6}[001] + \frac{1}{18}[111] \) remains at the interaction point. This defect was referred to Jin et al. as a “v-lock” as it contains a row of vacancies (aluminum with a negative Burgers vector).

Interactions in nickel were also simulated, resulting in either an i-lock or v-lock remaining in the boundary depending on the sign of the Burgers vector of the incoming dislocation. An additional interaction proposed from molecular dynamics simulations by Wu et al. showed that the transfer of a lead Shockley partial dislocation can cause the emission of two partial dislocations into the
emission side of the boundary as well as a single partial dislocation back into the grain from which the incoming dislocations came [101].

Three separate interactions involving non-screw dislocations with twin boundaries that did not allow cross-slip were observed in stainless steel (2 in Figure 4.22 and one additional interaction in Figure 4.32). In the initial interaction seen in Figure 4.22, the dislocation transfer across the boundary resulted in the emission of perfect dislocations of two different types into the neighboring grain (system 1), the back-emission of partial dislocations, both lead and trailing, into the original grain (system 2), and the emission of lead partial dislocations as well as two systems of perfect dislocations into the original grain (system 3). Evidence of partial dislocations in the boundary plane was obscured by the presence of extrinsic dislocations of unknown origin. Qualitatively, the interaction as seen in system 1 is similar to simulation results 1 and 4 by Jin et al. Systems 2 and 3 reach a higher level of complexity seen by any of the simulations. This may be due to the presence of extrinsic dislocations interacting with glissile partial dislocations in the boundary plane. Alternatively, it may be due to the temporal limitations of molecular dynamics simulations.

The second interaction for direct comparison is also seen in Figure 4.22, but after the system 2 dislocations had traversed the twinned region (system 2d). The observed interaction should be similar in character to the original interaction from the incoming dislocations as both involve pure edge dislocations impinging on a coherent twin boundary. However, the response of system 2d at the boundary was the emission of a single partial dislocation, qualitatively similar to simulation result 2 by Jin et al., as opposed to the more complex response seen in the previously discussed interaction. This may be due to the complex stress state likely to exist in the narrow
twinned region or to the fact that more dislocations had traversed the upper twin boundary in comparison to the lower twin boundary.

The third interaction for comparison is shown in Figure 4.32 which occurred during \textit{in situ} straining at 400°C. The line direction of the incoming dislocations was not determined, leaving the dislocation character ambiguous. The result at the boundary to the impinging dislocations was the emission of partial dislocations, both the lead and the trailing, as well as the back emission of perfect dislocations with the same Burgers vector as the incoming dislocations. Analysis of the residual grain boundary dislocation showed that after the interaction, a partial dislocation glissile on the boundary plane should remain. No such feature was seen gliding in boundary, but that may be due to insufficient spatial resolution during the experiment. Again though, the complexity of the interaction seen in the experiment, in terms of number of activated systems, was higher than that seen in the simulations by Jin \textit{et al.} This may be due to differences between the simulation and the experiments, such as only a single incoming dislocation being simulated interacting with the twin boundary, the difference in strain rate (deemed “irrelevant” in the experimental section of Jin \textit{et al.}’s paper as non-linear effects on deformation behavior were ignored), environment temperature (0 K in the simulation), boundary conditions, and material investigated, specifically the stacking fault energy of the materials investigated (given in Table 5.1). The stacking fault energy could be especially important as it was considered by Jin \textit{et al.} to be a key parameter in dictating the evolution of the interactions. As can be seen from the values given in Table 5.1, stainless steel has a significantly lower stacking fault energy than either copper or aluminum, though of the two, copper is closer.
Table 5.1 Stacking fault energies as recorded in [202] and [203].

<table>
<thead>
<tr>
<th>Metal</th>
<th>Stacking Fault Energy [202] (mJ/m$^2$)</th>
<th>Stacking Fault Energy [203] (mJ/m$^2$)</th>
</tr>
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<tbody>
<tr>
<td>Stainless steel</td>
<td>&lt; 10</td>
<td>--</td>
</tr>
<tr>
<td>Copper</td>
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<td>78</td>
</tr>
<tr>
<td>Aluminum</td>
<td>~250</td>
<td>166</td>
</tr>
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5.2 DISLOCATION INTERACTIONS IN $\alpha$-TI

5.2.1 Dislocation/grain boundary interactions

Comparison of the observed dislocation/grain boundary interactions in $\alpha$-Ti to the slip transfer criteria proposed by Lee et al. presents a unique challenge as different combinations of dislocations can result in identical values of $\gamma_{gb}$ after slip transfer. For example, the emission of an $<a>$-type dislocation with Burgers vector $\mathbf{b} = a/3 [112 \overline{0}]$ from a boundary is equivalent in terms of $|\gamma_{gb}|$ to simultaneously emitting two dislocations with Burgers vectors $\mathbf{b} = a/3 [1 \overline{2} 1 0]$ and $\mathbf{b} = a/3 [21 1 0]$. Experimentally, only one of the observed interactions induced by ex situ deformation suggested that emitting the two equivalent dislocations rather than a single dislocation was preferred (Fig. 4.54). Such behavior was not seen during the in situ deformation experiments.
Unlike the observations in stainless steel, dislocation/grain boundary interactions in Ti were highly dependent on whether the sample was deformed before or after thinning to electron transparency. In the bulk deformed samples, one incoming dislocation system caused the nucleation of one emitting slip system only in the example in which the dislocations impinged on a low angle grain boundary (Fig. 4.63). In all other examples shown, two or more dislocation systems were emitted simultaneously from the boundary, often as dense tangles instead of the sequential initiation of emitted dislocation systems more commonly encountered in stainless steel. The slip bands themselves were often ill defined, making it difficult to unambiguously identify the dislocation systems associated with the interaction. Also, it was not always clear in the interactions that minimization of \( |b_r^{gb}| \) was the main factor determining which system was emitted into the neighboring grain. Of the four characterized interactions after bulk deformation, two appeared to be driven by the minimization of \( |b_r^{gb}| \) (Figures 4.54 and 4.63). One interaction may have been driven by minimization of \( |b_r^{gb}| \) but required characterization of the sign of the Burgers vector to remove ambiguity (Fig. 4.56), and one interaction seemed only partially driven by the minimization of \( |b_r^{gb}| \) (Fig. 4.58). That is, the impinging dislocations caused the nucleation of two systems into the neighboring grain, one of which reduced the value of \( |b_r^{gb}| \) while the other caused a large increase in \( |b_r^{gb}| \). As the samples were deformed \textit{ex situ} it is unknown if the interactions which did not minimize \( |b_r^{gb}| \) were only short term responses due to high levels of resolved shear stress. The local stress state could only be estimated for one of the observed interactions (Fig. 4.58-4.62) and did not appear to have a role in how the interaction progressed.
In contrast, the dislocation/grain boundary interactions observed during \textit{in situ} deformation all resulted in the initial emission of only one dislocation system which was found to be the optimal system in terms of minimizing $|\mathbf{b}_{r}^{gb}|$. This changed at later stages of the deformation when multiple tangled dislocation systems were seen emitting from a single grain boundary source (Fig. 4.67). The behavior of the emitted dislocations varied from, once nucleated, quickly propagating from the boundary source (Fig. 4.66) to where the dislocation slowly separated from the boundary as a half-loop extending into the grain matrix (Fig. 4.69). While nucleation of a dislocation system at a boundary was almost always preceded by the impingement of multiple dislocations, only rarely did a dislocation pileup form.

Two factors that could account for the difference in behavior between bulk samples and thin films are that in the latter the stress state and the proximity of the free surfaces permit slip on systems not generally active at room temperature. Dislocation slip at room temperature is generally confined to the prismatic planes during bulk deformation. However, slip on both the prismatic and pyramidal planes was commonly observed in the interactions occurring at room temperature during the \textit{in situ} straining. This increase in the number of available slip systems could potentially simplify the dislocation/grain boundary interactions as slip could more easily be mediated at the boundary. Though the activation of additional slip systems can also be instigated by elevating the temperature, the thin film effect appeared somewhat different. Naka and LaSalmonie reported that cross-slip onto the pyramidal planes is activated at temperatures slightly above room temperature [125], but pyramidal slip was never seen as a primary slip plane [121]. This differs from what was seen during \textit{in situ} deformation as dislocations could nucleate from boundary sources onto a pyramidal plane and act as the primary slip plane (Fig. 4.50).
The difference in the stress state could also simplify the dislocation interactions. The bulk samples were kinked slightly to introduce dislocation activity, creating a complex loading condition of both tension and compression with no convenient location for dislocation nucleation. The *in situ* deformation, in contrast, was introduced through simple tension, albeit with a non-circular hole and wedge profile at the center of the sample which complicates the stress state. The hole also provides a site for dislocation nucleation as most observed dislocation systems propagated from the front of progressing cracks emanating from the hole. This resulted in the dislocations generally travelling in the same direction, leading to fewer dislocation tangles and less complex interactions.

Only one of the observed dislocation interactions involved $\langle c + a \rangle$-type dislocations (Fig. 4.54), confirming reports in other studies that deformation is mainly accommodated by $\langle a \rangle$-type dislocations [128, 129]. The result was the simultaneous emission of two different dislocation systems composed of $\langle a \rangle$-type dislocations. Their combined effect minimized $|\beta_{rb}|$ more effectively than the emission of any single dislocation system.

Only a single paper on simulations of dislocation/grain boundary interactions was available for comparison to the experimental results. Serra and Bacon investigated the interactions of screw dislocations with both $\{101\overline{1}\}$ and $\{1012\}$ twin boundaries using atomistic simulations [105]. They found, in agreement with the experimental results shown in Figure 4.71-4.73, that screw dislocations can transfer across $\{1012\}$ twin boundaries through a cross-slip mechanism. The transfer across the boundary required $-0.013$ applied strain. While a direct quantitative comparison with experiment is not possible here, the barrier to slip is in agreement with the experimental results as dislocations were seen trapped in the boundary plane.
prior to transferring into the twinned region. In the majority of the simulations, the dislocations were seen to slip from a prismatic to a basal plane while crossing the boundary. In the experiment, the dislocations initially glided on a pyramidal plane and cross-slipped onto a prismatic plane when crossing the boundary. Such a discrepancy could be attributed to thin film effects or the differences in boundary conditions between the simulations and experiment.

5.2.2 Dislocation/dislocation interactions

A central feature of dislocation motion during deformation of α-Ti was the prevalence of double cross-slip events, the majority of which occurred between the pyramidal and prismatic planes. Similar double cross-slip activity has been inferred after post mortem deformation, mainly in the work by Naka et al. [125, 130, 177]. They observed that even when single crystal samples were oriented for prismatic glide, double cross-slip between the prismatic and first order pyramidal planes was observed. The occurrence of double cross-slip events was heterogeneously distributed along the length of <a>-type screw dislocations, likely reflecting a distribution of barriers to dislocation motion. These barriers could not be resolved during TEM characterization. Naka et al. proposed a model in which the core of <a>-type screw dislocations was spread between both the prismatic and the pyramidal planes, promoting cross-slip of dislocation segments onto the pyramidal planes. As the prismatic plane is the most energetically favorable for glide in α-Ti, further applied stress would cause the dislocation segment on the pyramidal plane to cross-slip back onto the prismatic plane. This cross-slip could either return the dislocation to its original glide plane or result in the dislocation being split between two parallel prismatic planes with a connecting sessile segment, similar to what is seen in Figure 4.77.
Investigations of the core structure of \(<a>\)-type screw dislocations in \(\alpha\)-Ti have confirmed limited core spreading, though the exact structure is still being debated [204–206]. As mentioned above, Naka et al. have proposed that the core spreading must exist between the prismatic and pyramidal planes based on the observed dislocation behavior [177, 204]. Neeraj et al. found that \(\alpha\)-Ti displays asymmetric behavior between deformation in tension and compression [205]. This asymmetry was attributed to the behavior of \(<a>\)-type dislocations, which were found to readily cross-slip during tensile deformation. High resolution TEM micrographs of the \(<a>\)-type dislocations showed slight core spreading onto the pyramidal plane, suggesting a similar cross-slip mechanism to that proposed by Naka et al. Both Naka et al. and Neeraj et al. suggested that the presence of solute atoms such as oxygen and aluminum may affect the dislocation core structure and thus promote further double cross-slip events. Girshick et al. investigated the core structure of \(<a>\)-type screw dislocations in Ti using atomistic simulations [206]. Similar to the conclusions of Naka et al., they observed non-planar core spreading, though this spreading occurred mainly between the prismatic and basal planes. The results presented in this study support that, based on dislocation activity, the dislocation core should be spread between the pyramidal and prismatic planes. Also similar to the studies by Naka et al., the occurrence of double cross-slip events was heterogeneously distributed along the length of screw dislocations, suggesting the influence of an unknown lattice defect.

During \textit{in situ} deformation, dislocation cross-slip was seen to result in both small loops fully enclosed in the thin films as well as larger half-loops connected to the foil surface. Obstacle size and barrier strength could account for the different dislocation reactions as a smaller barrier could cause more localized cross-slip, resulting in the formation of a small dislocation loop (Fig. 4.83). Stronger barriers to dislocation motion could lead to the formation of the larger half-loops,
such as is seen in Figure 4.77. The *in situ* deformation also showed that the dislocations can repeatedly generate dislocation loops and half-loops during glide (Fig. 4.83), creating dislocation debris throughout the matrix and impeding the glide of trailing dislocations.

### 5.3 DIFFRACTION CONTRAST ELECTRON TOMOGRAPHY

Past applications of electron tomography have focused on the morphological resolution of 3D objects in space [153-155]. For this reason, when considering the accuracy of electron tomography and the fidelity of reconstructions, factors such as elongation have played a central role. This has led to the assumption that for the successful application of electron tomography, images must be acquired over a large tilt range, 120-140°, with an image collected every degree of tilt and contrast varying linearly between images. Diffraction contrast electron tomography, when applied to the study of dislocation interactions, requires a very different approach as now the morphology of the objects of investigation is well known. That is, the dislocations themselves are already known to be line defects either self-terminating as a loop, at an interface, or at an intersection with another dislocation. The issue then becomes not the determination of the shape of an object, but that object’s location and orientation in a given volume. As such, image elongation and the need to reduce the missing wedge of information plays a less significant role in determining the usefulness and fidelity of the reconstruction as the dislocation image position can still be identified even when feature elongation in the tomogram is severe. The accurate determination of the position of the dislocation image allows for the construction of a high fidelity 3D-model of a dislocation interaction, mitigating any negative feature elongation effects.
Theoretically, only two projections are needed to uniquely define the location of a line segment in a volume. Similarly, if a boundary is known to reside on a single plane, only two projections would be needed to uniquely define its location. Bend contours, variations in diffraction conditions, and background noise require additional images to achieve a satisfactory reconstruction; here satisfactory is defined as sufficient to construct a high fidelity 3D-dislocation model of the interaction. With this information, it can be seen that an image series aligned using the fiducial marker method fulfills the requirements for the reconstruction of a satisfactory tomogram as long as the characterized area can be imaged in two-beam BF mode at a minimum of two ranges of tilt over an appropriate angular range.

As shown in section 4.1.5, iterative reconstruction techniques, usually associated with higher fidelity and lower noise, are not able to provide the same tomogram resolution as weighted back projection algorithms for resolving dislocations. This is most likely due to more stringent requirements to adhere to the projection requirement, the requirement that contrast fluctuates linearly as a function of thickness and no more than one other physical property, when reconstructing a tomogram using iterative techniques. Each image in a tilt series is used to correct data in the tomogram, which can confound the reconstruction in tilt series composed of images with strongly varying contrast [157, 162]. Weighted back projection algorithms do not impose such a high level of interdependence between images composing a tilt series. This allows tomogram features to be clearly resolved at a few select angles even when the majority of images in the tilt series have poor resolution (see, for example, Figure 4.40). As discussed, this is sufficient for the construction of a model of the interaction as the defects under consideration have well characterized morphologies and behaviors. The result of these factors is that satisfactory tomograms can be reconstructed from datasets that by traditional tomography
standards would be considered unsuitable, expanding the applicability of electron tomography to volumes spanning two different grains and to image sets acquired during in situ deformation experiments.
CHAPTER 6

CONCLUSIONS

Dislocation/grain boundary and dislocation/dislocation interactions have been investigated using in situ deformation experiments as well as ex situ deformation followed by post mortem analysis in the TEM. Electron tomography was utilized to retrieve the 3D dislocation state in both the ex situ characterization experiments as well as at interrupted points during in situ deformation. Grain boundary/dislocation interactions in austenitic stainless steel were investigated at both room and elevated temperature and behavior in the two environments was compared. Dislocation interactions in Ti were investigated at room temperature with a focus on dislocation/grain boundary interactions as well as dislocation/dislocation interactions and behavior during dislocation generation.

It was found that dislocation/grain boundary interactions in stainless steel agree well with the criteria set forth by Lee et al. at elevated as well as room temperature [9, 77]. That is, reduction of the elastic strain energy through minimizing \( |b_r| \) was the main factor in determining which system nucleated at a boundary given the impingement of an incoming dislocation system. In simple interactions, the local resolved shear stress, as estimated by approximating the incoming dislocation system as a single dislocation at the boundary with the Burgers vector and line direction of the incoming dislocations, was found to be a significant factor in determining the plane on which dislocations were emitted from the boundary. This
approximation was found to be an oversimplification in more complex interactions involving multiple dislocation systems. Increasing the temperature during \textit{in situ} deformation was shown to lower the barrier strength of grain boundaries and increase the complexity of the interactions. Interaction at elevated temperature were still, however, predominately governed by the minimization of $|b_r^g|$. 

Dislocation interactions with coherent twin boundaries were found to be highly dependent on the relation between the Burgers vector of the incoming dislocations and the twin plane. When the twin plane was a conjugate slip plane for the incoming system, the initial response upon impingement was for the dislocations to cross-slip onto and become mobile in the boundary plane without splitting into partial dislocations. When the Burgers vector of the incoming dislocations did not allow slip on the boundary plane, the dislocations transferred through the boundary, with the emitted dislocation system being determined by minimization of $|b_r^g|$. 

In $\alpha$-Ti, dislocation/grain boundary reactions varied strongly depending on whether the deformation was performed \textit{ex situ} on a bulk sample or during \textit{in situ} straining. Dislocation/grain boundary interactions after \textit{ex situ} deformation were more complex with multiple dislocation systems emitted simultaneously from the boundary plane. Due to the complexity of the interactions, it could not be confirmed for all cases that minimization of $|b_r^g|$ was the prominent factor dictating the interaction evolution. Dislocation interactions observed during \textit{in situ} deformation were simpler, with the initial grain boundary response being to emit only a single dislocation system. In all cases, the emitted system was found to be that which minimized $|b_r^g|$ in agreement with the criteria set forth by Lee \textit{et al.} [9].
Dislocation generation through double cross-slip mechanisms, previously suggested from post mortem observations, was observed in real time during in situ deformation of α-Ti. Single gliding dislocations were seen undergoing multiple sequential double cross-slip events, resulting in the emission of loops and half-loops. It is assumed, however, that thin film effects significantly influenced the interactions as dislocations were commonly observed gliding on planes not usually active at room temperature.

Full characterization of the interactions, including 3D and time resolved, in both stainless steel and Ti was possible using a novel approach to facilitate the combination of dynamic experiments conducted using a single tilt heating/straining stage with periodic 3D snapshots of interrupted interactions. Preprocessing of micrographs collected at regular intervals over a wide tilt range through manual placement of digital fiducial markers made alignment of the images for tomographic reconstruction possible. Strong contrast fluctuations still existed in the aligned images, but it was found that in many cases the resultant resolution degradation of the reconstructed tomogram could be compensated for by constructing a 3D model of the interaction using prior knowledge of dislocation behavior. A coordinate system, once properly oriented in the model, provided information on the line directions, slip plane, and spatial relations of the dislocations for full system characterization. This was found to aid in understanding the dislocation interactions and allowed more direct comparisons to results of computer simulations of dislocation interactions.
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APPENDIX A

MATLAB CODE

A.1 Residual Burgers vector FCC

Finding the Burgers vector associated with each outgoing system if given an incoming dislocation system and the grain boundary character. Works for FCC crystal lattices.

% permutations to check for a given incoming Dislocation and grain boundary characterizations, what will be the Burgers vector of the residual grain boundary dislocation associated with each outgoing system

clear all
close all

% load orientations of two grains either as the Euler angles of the two grains or as an axis/angle pair defining the boundary

% for axis/angle pairs
axs = [0 1 1]; % input the rotation axis
angle = 38.94; % input the rotation angle in degrees

% for inputing Euler angles in radians
% gmat1 = euler2gmat(3.2114, 0.6126, 0.4032);
% gmat2 = euler2gmat(0.6301, 0.7365, 0.2391);
% g2 = gmat1*gmat2';
g1 = eye(3);

% load incoming dislocation's Burgers vector
bin = g1'*[0; 1; 1];

% load all potential burgers vectors
% 1 for perfects, two for partials
dislo = 1;
if dislo == 1
    b = cell([1 12]);
b{1} = [1 1 0]';
b{2} = [1 -1 0]';
b{3} = [1 0 1]';
b{4} = [1 0 -1]';
b{5} = [0 1 1]';
b{6} = [0 1 -1]';
for i = 1:6
    b{6+i} = -b{i};
end
else
    b = cell([1 24]);
b{1} = [1 2 -1]';
b{2} = [1 -1 2]';
b{3} = [2 -1 -1]';
b{4} = [1 -1 -2]';
b{5} = [1 2 1]';
b{6} = [-2 -1 1]';
b{7} = [1 1 2]';
b{8} = [1 -2 -1]';
b{9} = [-2 1 -1]';
b{10} = [-1 1 -2]';
b{11} = [-1 -2 1]';
b{12} = [2 1 1]';
for i = 1:12
    b{12+i} = -b{i};
end
end

% rotate into sample frame
for i = 1:length(b)
    b{i} = g2'*b{i};
end

% check all residuals (magnitude)
bres = zeros(1,length(b));
for i = 1:length(b)
    bres(i) = norm(bin-b{i});
end

% display results in terms of magnitude of the Burgers vector. The a/2 % factor is not included
disp(bres)
plot(bres)

Associated functions not standard in Matlab

function g=AA2GMat(axis,angle) %angle needs to be input in degrees
angle=angle*pi/180;
ca=cos(angle);sa=sin(angle);
u=axis(1)/norm(axis); v=axis(2)/norm(axis); w=axis(3)/norm(axis); %normalize this or you'll be sorry
x=u; y=v; z=w;
g(1,1)=1+(1-ca)*(x*x-1);
g(1,2)=-z*sa+(1-ca)*x*y;
g(1,3)=y*sa+(1-ca)*x*z;
\[ g(2,1) = z + sa \times (l - ca) \times x \times y; \]
\[ g(2,2) = 1 + (l - ca) \times (y \times y - 1); \]
\[ g(2,3) = -x + sa \times (l - ca) \times y \times z; \]
\[ g(3,1) = -y + sa \times (l - ca) \times x \times z; \]
\[ g(3,2) = x + sa \times (l - ca) \times y \times z; \]
\[ g(3,3) = 1 + (l - ca) \times (z \times z - 1); \]

**function** \[ g = euler2gmat(phi1, PHI, phi2) \]
% euler2gmat - creates a g-matrix according to bunge [180] for phi1, PHI, phi2 in % radians

\% cp1 = cos(phi1);
sp1 = sin(phi1);

\% cp2 = cos(phi2);
sp2 = sin(phi2);
cP = cos(PHI);
sP = sin(PHI);
g=zeros(3,3);
g(1,1) = cp1 \times cp2 - sp1 \times sp2 \times cP;
g(1,2) = sp1 \times cp2 + cp1 \times sp2 \times cP;
g(1,3) = sp2 \times sP;
g(2,1) = -cp1 \times sp2 - sp1 \times cp2 \times cP;
g(2,2) = -sp1 \times sp2 + cp1 \times cp2 \times cP;
g(2,3) = cp2 \times sP;
g(3,1) = sp1 \times sP;
g(3,2) = -cp1 \times sP;
g(3,3) = cP;

**A.2 Residual Burgers vector HCP**

**Finding the Burgers vector associated with each outgoing system if given an incoming dislocation system and the grain boundary character. Works for HCP crystal lattices.**

% Code for measuring the residual Burgers vector for all possible emitted % dislocations in an HCP material.

clear all
close all

% input lattice parameters and c/a ratio
a = 2.950;
as = a*3/2; \% done for converting to 3 index notation later
c = 4.683;
caRat = c/a;
lambda = sqrt(2/3)*caRat;

% Input the Burgers vector of the incoming dislocation in 4 index notation
bin4 = [1 -2 1 0];
% Input rotation across the grain boundary in degrees
theta = 108.5;
rotPlane4 = [-2 0 2 -1];

% quick check of the inputted vectors
if bin4(1)+bin4(2)+bin4(3) ~= 0 || rotPlane4(1)+rotPlane4(2)+rotPlane4(3) ~= 0
    disp('you put in a bad vector')
    return
end

% convert into three index notation
S = [1 -0.5 0; 0 sqrt(3)/2 0; 0 0 caRat];
Sinv = inv(S);
bin3 = [2*bin4(1)+bin4(2) bin4(1)+2*bin4(2) bin4(4)];
bin = S*bin3';
rotPlane = S*rotPlane3';

% % calculate the rotation matrix either using the axis/angle pair
% % characteristic of the grain boundary or by inputing the Euler angles of
% % the two grains. g1 is the incoming grain, g2 is the outgoing

% % Using the axis/angle pair
% g1 = eye(3);
% g2 = AA2GMat(rotPlane,theta);

% % Using the Euler angles of the individual grains
% g1 = euler2gmat(0*pi/180, 93*pi/180, 91*pi/180);

g2 = euler2gmat(355*pi/180, 118*pi/180, 81*pi/180);
gr = g2*g1';

% load all potential Burgers vectors for the outgoing dislocations
b = cell([1 20]);
b4{1} = [1 1 -2 0];
b4{2} = [-1 2 -1 0];
b4{3} = [-2 1 1 0];
b4{4} = [1 1 -2 3];
b4{5} = [-1 2 -1 3];
b4{6} = [-2 1 1 3];
b4{7} = [1 1 -2 -3];
b4{8} = [-1 2 -1 -3];
b4{9} = [-2 1 1 -3];
b4{10} = [0 0 0 3];
for i = 1:10
    b4{i+10} = -b4{i};
end

% % look at combinations
% for i = 1:20
%     for j = 1:20
%         b4(20*i+j) = (b4(i)+b4{j});
%     end
% end
% end

% convert the Burgers vectors into 3 index notation
for i = 1:length(b4)
    b3 = [2*b4{i}(1)+b4{i}(2); b4{i}(1)+2*b4{i}(2); b4{i}(4)];
    b{i} = S*b3;
end

% rotate them into the incoming grain reference frame
for i = 1:length(b)
    b{i} = gr'*b{i};
end

% check all residuals (magnitude)
bres = zeros(1,length(b));
for i = 1:length(b)
    bres(i) = norm(bin-b{i});
%    bres(i) = findangd(bin,b{i});
end

% Display and plot the magnitudes of the Burgers vectors of the residual
% grain boundary dislocations
disp(bres)

figure, plot(bres)

A.3 Resolved shear stress calculation FCC

Calculating the resolved shear stress acting on all the potential emitted dislocation systems from
a dislocation pileup of a single type of dislocation. Works for FCC crystal lattice.

% Estimate the local stress on an adjacent grain caused from incoming
% dislocations and estimate the resolved shear stresses on the slip planes

clear all
close all

% load material constants
G = 77.2; % in GPa
nu = 0.3; % poisson's ratio
a = 0.285; % in nm

% input orientations
g1 = eye(3);
g2 = AA2GMat([[-3 -27 -11],15];
% g2 = g1;
Qg1g2 = g2;
% load the incoming burgers vector, line direction, and slip plane (in grain 1 frame)
bin = a/2*[0 1 1];
lin = [0 1 1]/sqrt(2);
pin = [1 1 -1]/sqrt(3);
b = norm(bin);

% load boundary plane (in grain one frame)
% plane = [1 1 1]/sqrt(3);

% rough calculation of dislocation type
ang = findang(bin,lin);
bscrew = abs(b*cos(ang));
bedge = abs(b*sin(ang));

% x axis for the dislocation frame is parallel to the edge component of the
burgers vector, defined
% by the cross product of the line direction with the slip plane normal
be = cross(pin,lin);
be = be/norm(be);
plane = be;

% y axis is defined by the plane normal and z axis is defined by the line
% direction. We can use that to calculate the dislocation to incoming grain
% transformation matrix
Qdg1 = [be'/norm(be) pin'/norm(pin) lin'/norm(lin)];

% calculate stress tensor from grain 1 (incoming dislocations) in
% dislocation frame
% rotate plane normal into the dislocation frame to get x and y for stress
% field around edge dislocatoin (assumes that the dislocation line
% direction lies in the boundary plane)
planed = Qdg1'*plane';

alpha = [-1;1;-1]/sqrt(3);
beta = [1;-1;-1]/sqrt(3);
gamma = [-1;-1;1]/sqrt(3);
delta = [1;1;1]/sqrt(3);

% for each plane, get the plane normal projected direction and rotate into
% the
% dislocation frame
% to get the projected vector, project the grain boundary plane normal onto
% the plane of interest and normalize
% use the projected vector to calculate the stress tensor at one unit
% vector distance from the boundary
% for alpha

% Find the projected vector
mager = dot(plane',alpha);
projer = plane' - alpha*mager;
projer_alpha = projer/norm(projer);
projer_alphad = Qdg1'*Qg1g2'*projer_alpha;

xy = projer_alphad-[0; 0; projer_alphad(3)];
xy = xy/norm(xy);

% x = projer_alphad(1);
% y = projer_alphad(2);
% Calculate the stress tensor for the incoming dislocations. Equations come
% from the Hull and Bacon book [181]. This needs to be done for
% each available plane (alpha, beta, gamma, delta in for a FCC lattice)
sscrewa = [0 0 -G*bscrew/(2*pi)*y/(x^2+y^2);...
0 0 G*bscrew/(2*pi)*x/(x^2+y^2);...
-G*bscrew/(2*pi)*y/(x^2+y^2) G*bscrew/(2*pi)*x/(x^2+y^2) 0];

D = G*bedge/(2*pi*(1-nu));

sedgea = [-D*y*(3*x^2+y^2)/(x^2+y^2)^2 D*x*(x^2-y^2)/(x^2+y^2)^2 0;...
D*x*(x^2-y^2)/(x^2+y^2)^2 D*y*(x^2-y^2)/(x^2+y^2)^2 0;...
0 0 nu*(-D*y*(3*x^2+y^2)/(x^2+y^2)^2+D*y*(x^2-y^2)/(x^2+y^2)^2)
D*y*(x^2-y^2)/(x^2+y^2)^2 D*y*(x^2-y^2)/(x^2+y^2)^2 0];

stotad = sscrewa+sedgea;
stotag1 = Qdg1*stotad*Qdg1';
stotag2 = Qg1g2*stotag1*Qg1g2';

% for beta
mager = dot(plane',beta);
projer = plane' - beta*mager;
projer_beta = projer/norm(projer);
projer_betad = Qdg1'*Qg1g2'*projer_beta;

xy = projer_betad-[0; 0; projer_betad(3)];
xy = xy/norm(xy);

% x = projer_betad(1);
% y = projer_betad(2);

sscrewb = [0 0 -G*bscrew/(2*pi)*y/(x^2+y^2);...
0 0 G*bscrew/(2*pi)*x/(x^2+y^2);...
-G*bscrew/(2*pi)*y/(x^2+y^2) G*bscrew/(2*pi)*x/(x^2+y^2) 0];

D = G*bedge/(2*pi*(1-nu));

sedgeb = [-D*y*(3*x^2+y^2)/(x^2+y^2)^2 D*x*(x^2-y^2)/(x^2+y^2)^2 0;...
D*x*(x^2-y^2)/(x^2+y^2)^2 D*y*(x^2-y^2)/(x^2+y^2)^2 0;...
0 0 nu*(-D*y*(3*x^2+y^2)/(x^2+y^2)^2+D*y*(x^2-y^2)/(x^2+y^2)^2)
D*y*(x^2-y^2)/(x^2+y^2)^2 D*y*(x^2-y^2)/(x^2+y^2)^2 0];

stotbd = sscrewb+sedgeb;
stotbg1 = Qdg1*stotbd*Qdg1';
stotbg2 = Qg1g2*stotbg1*Qg1g2';
% for gamma
mager = dot(plane', gamma);
proj = plane' - gamma*mager;
proj_gamm = proj/norm(proj);
proj_gammad = Qdg1'*Qg1g2'*proj_gamm;

xy = proj_gammad-[0; 0; proj_gammad(3)];
xy = xy/norm(xy);

% x = proj_gammad(1);
% y = proj_gammad(2);
x = xy(1);
y = xy(2);

sscrewc = [0 0 -G*bscrew/(2*pi)*y/(x^2+y^2);... 0 0 G*bscrew/(2*pi)*x/(x^2+y^2);...
-0*bscrew/(2*pi)*y/(x^2+y^2) G*bscrew/(2*pi)*x/(x^2+y^2) 0];

D = G*bedge/(2*pi*(1-nu));
sedged = [-D*y*(3*x^2+y^2)/(x^2+y^2)^2 D*x*(x^2-y^2)/(x^2+y^2)^2 0;...
D*x*(x^2-y^2)/(x^2+y^2)^2 D*y*(x^2-y^2)/(x^2+y^2)^2 0;...
0 0 nu*(-D*y*(3*x^2+y^2)/(x^2+y^2)^2+D*y*(x^2-y^2)/(x^2+y^2)^2)];

stotcd = sscrewc+sedged;
stotcg1 = Qdg1*stotcd*Qdg1';
stotcg2 = Qg1g2*stotcg1*Qg1g2';

% for delta
mager = dot(plane', delta);
proj = plane' - delta*mager;
proj_delta = proj/norm(proj);
proj_deltad = Qdg1'*Qg1g2'*proj_delta;

xy = proj_deltad-[0; 0; proj_deltad(3)];
xy = xy/norm(xy);

% x = proj_deltad(1);
% y = proj_deltad(2);
x = xy(1);
y = xy(2);

sscrewd = [0 0 -G*bscrew/(2*pi)*y/(x^2+y^2);...
0 0 G*bscrew/(2*pi)*x/(x^2+y^2);...
-0*bscrew/(2*pi)*y/(x^2+y^2) G*bscrew/(2*pi)*x/(x^2+y^2) 0];

D = G*bedge/(2*pi*(1-nu));
sedged = [-D*y*(3*x^2+y^2)/(x^2+y^2)^2 D*x*(x^2-y^2)/(x^2+y^2)^2 0;...
D*x*(x^2-y^2)/(x^2+y^2)^2 D*y*(x^2-y^2)/(x^2+y^2)^2 0;...
0 0 nu*(-D*y*(3*x^2+y^2)/(x^2+y^2)^2+D*y*(x^2-y^2)/(x^2+y^2)^2)];

stotdd = sscrewd+sedged;
stotdg1 = Qdg1*stotdd*Qdg1';
stotdg2 = Qg1g2*stotdg1*Qg1g2'
% apply the stress tensor to all possible slip systems (going off of
% Microstructure Sensitive Design pg 118)
% planes

dislo = 2; % 1 to look at perfects, 2 to look at partials
if dislo == 1
    % directions
    d = zeros(3,6);
    d(:,1) = [1;1;0];
    d(:,2) = [1;0;1];
    d(:,3) = [0;1;1];
    d(:,4) = [-1;1;0];
    d(:,5) = [1;0;-1];
    d(:,6) = [0;-1;1];

    % calculate resolved shear stress on each system
    % alpha plane
    tauRSS(1) = sum(sum(stotag2.*dyadic(d(:,1),alpha))); % alpha110
    tauRSS(2) = sum(sum(stotag2.*dyadic(d(:,3),alpha))); % alpha011
    tauRSS(3) = sum(sum(stotag2.*dyadic(d(:,5),alpha))); % alpha10-1

    % beta plane
    tauRSS(4) = sum(sum(stotbg2.*dyadic(d(:,1),beta))); % beta110
    tauRSS(5) = sum(sum(stotbg2.*dyadic(d(:,2),beta))); % beta101
    tauRSS(6) = sum(sum(stotbg2.*dyadic(d(:,6),beta))); % beta0-11

    % gamma plane
    tauRSS(7) = sum(sum(stotcg2.*dyadic(d(:,4),gamma))); % gamma-110
    tauRSS(8) = sum(sum(stotcg2.*dyadic(d(:,2),gamma))); % gamma101
    tauRSS(9) = sum(sum(stotcg2.*dyadic(d(:,3),gamma))); % gamma011

    % delta plane
    tauRSS(10) = sum(sum(stotdg2.*dyadic(d(:,4),delta))); % delta-110
    tauRSS(11) = sum(sum(stotdg2.*dyadic(d(:,5),delta))); % delta10-1
    tauRSS(12) = sum(sum(stotdg2.*dyadic(d(:,6),delta))); % delta0-11
else
    % directions
    d = zeros(3,12);
    d(:,1) = [1;1;2];
    d(:,2) = [-1;1;2];
    d(:,3) = [-1;-1;2];
    d(:,4) = [-1;1;-2];
    d(:,5) = [1;2;1];
    d(:,6) = [-1;2;1];
    d(:,7) = [-1;-2;1];
    d(:,8) = [-1;2;-1];
    d(:,9) = [2;1;1];
    d(:,10) = [-2;1;1];
    d(:,11) = [-2;-1;1];
    d(:,12) = [-2;1;-1];

    % calculate resolved shear stress on each system
    % alpha plane
tauRSS(1) = sum(sum(stotag2.*dyadic(d(:,2),alpha))); % alpha-112
tauRSS(2) = sum(sum(stotag2.*dyadic(d(:,11),alpha))); % alpha-211
tauRSS(3) = sum(sum(stotag2.*dyadic(d(:,5),alpha))); % alpha121

% beta plane
tauRSS(4) = sum(sum(stotbg2.*dyadic(d(:,9),beta))); % beta211
tauRSS(5) = sum(sum(stotbg2.*dyadic(d(:,4),beta))); % beta-11-2
tauRSS(6) = sum(sum(stotbg2.*dyadic(d(:,7),beta))); % beta-1-21

% gamma plane
tauRSS(7) = sum(sum(stotcg2.*dyadic(d(:,6),gamma))); % gamma-121
tauRSS(8) = sum(sum(stotcg2.*dyadic(d(:,12),gamma))); % gamma-21-1
tauRSS(9) = sum(sum(stotcg2.*dyadic(d(:,1),gamma))); % gamma112

% delta plane
tauRSS(10) = sum(sum(stotd2g.*dyadic(d(:,3),delta))); % delta-1-12
tauRSS(11) = sum(sum(stotd2g.*dyadic(d(:,8),delta))); % delta-12-1
tauRSS(12) = sum(sum(stotd2g.*dyadic(d(:,10),delta))); % delta-211

end

tauRSS = abs(tauRSS);

figure, plot(tauRSS)

Associated functions not standard in Matlab

% find the angle between two vectors in radians
function ang = findang(v1,v2,caRat)

if nargin == 2
    ang =
    acos(sum(v1.*v2)/((sqrt(v1(1)^2+v1(2)^2+v1(3)^2))*sqrt(v2(1)^2+v2(2)^2+v2(3)^2)))
elseif nargin == 3
    S = [1 -0.5 0; 0 sqrt(3)/2 0; 0 0 caRat];
    v1 = S*rot434(v1)';
    v2 = S*rot434(v2)';
    ang =
    acos(sum(v1.*v2)/((sqrt(v1(1)^2+v1(2)^2+v1(3)^2))*sqrt(v2(1)^2+v2(2)^2+v2(3)^2)))
end

A.4 Resolved shear stress calculation HCP

Calculating the resolved shear stress acting on all the potential emitted dislocation systems from a dislocation pileup of a single type of dislocation. Works for HCP crystal lattice.
% Estimate the local stress on an adjacent grain caused from incoming
% dislocations and estimate the resolved shear stresses on the slip planes

% Taken directly
% from LocalStressCalcNew3.m, just switching to account for 4-index
% notation and the different slip systems.

clear all
close all

% load material constants
G = 44; % in GPa
nu = 0.32; % poisson's ratio
a = 0.2950;
as = a*3/2; % done for converting to 3 index notation later
C = 0.4683;
car = C/a;
lambda = sqrt(2/3)*car;
S = [1 -0.5 0; 0 sqrt(3)/2 0; 0 0 car];
Sinv = inv(S);

% input orientations
theta = 108.5;
rotplane4 = [2 0 -2 1];
rotplane = S*rot434(rotplane4)';
g1 = eye(3);
g2 = AA2GMat([-3 -27 -11],15);
% g2 = g1;
% g1 = euler2gmat(0*pi/180,93*pi/180,91*pi/180);
% g2 = euler2gmat(355*pi/180,118*pi/180,81*pi/180);
Qg1g2 = g2*g1';
% Qg1g2 = g2;

% load the incoming burgers vector, line direction, and slip plane (in grain
% 1 frame)
bin4 = a/3*[2 -1 -1 0];
lin4 = [2 -1 -1 0];
pin4 = [0 1 -1 1];

% convert to three index notation
bin3 = [2*bin4(1)+bin4(2) bin4(1)+2*bin4(2) bin4(4)];
bin = (S*bin3)';
lin = (S*lin3)';
lin = lin/norm(lin);
pin3 = [2*pin4(1)+pin4(2) pin4(1)+2*pin4(2) pin4(4)];
pin = (S*pin3)';
pin = pin/norm(pin);

b = norm(bin);

% rough calculation of dislocation type
ang = findang(bin,lin);
bscrew = abs(b*cos(ang));
bedge = abs(b*sin(ang));

% x axis for the dislocation frame is parallel to the edge component of the
% burgers vector, defined
% by the cross product of the line direction with the slip plane normal
be = cross(pin,lin);
be = be/norm(be);

plane = be;

% y axis is defined by the plane normal and z axis is defined by the line
% direction. We can use that to calculate the dislocation to incoming grain
% transformation matrix
Qdg1 = [be'/norm(be) pin'/norm(pin) lin'/norm(lin)];

% calculate stress tensor from grain 1 (incoming dislocations) in
% dislocation frame
% rotate plane normal into the dislocation frame to get x and y for stress
% field around edge dislocation (assumes that the dislocation line
% direction lies in the boundary plane)
planed = Qdg1'*plane';

% load all available slip planes
basal4 = [0; 0; 0; 1];
prismatic14 = [1; -1; 0; 0];
prismatic24 = [1; 0; -1; 0];
prismatic34 = [0; 1; -1; 0];
pyramidal14 = [1; -1; 0; 1];
pyramidal24 = [1; 0; -1; 1];
pyramidal34 = [0; 1; -1; 1];
pyramidal44 = [1; -1; 0; -1];
pyramidal54 = [1; 0; -1; -1];
pyramidal64 = [0; 1; -1; -1];

% convert to 3 index notation and normalize
basal = S*rot434(basal4)';
prismatic1 = S*rot434(prismatic14)';
prismatic2 = S*rot434(prismatic24)';
prismatic3 = S*rot434(prismatic34)';
pyramidal1 = S*rot434(pyramidal14)';
pyramidal2 = S*rot434(pyramidal24)';
pyramidal3 = S*rot434(pyramidal34)';
pyramidal4 = S*rot434(pyramidal44)';
pyramidal5 = S*rot434(pyramidal54)';
pyramidal6 = S*rot434(pyramidal64)';

prismatic1 = prismatic1/norm(prismatic1);
prismatic2 = prismatic2/norm(prismatic2);
prismatic3 = prismatic3/norm(prismatic3);
pyramidal1 = pyramidal1/norm(pyramidal1);
pyramidal2 = pyramidal2/norm(pyramidal2);
pyramidal3 = pyramidal3/norm(pyramidal3);
pyramidal4 = pyramidal4/norm(pyramidal4);
pyramidal5 = pyramidal5/norm(pyramidal5);
pyramidal6 = pyramidal6/norm(pyramidal6);

% for each plane, get the plane normal projected direction and rotate into the
% dislocation frame
% to get the projected vector, project the grain boundary plane normal onto the
% plane of interest and normalize
% use the projected vector to calculate the stress tensor at one unit vector distance from the boundary
% for basal
mager = dot(plane',Qg1g2'*basal);
projer = plane' - basal*mager;
projer_basal = projer/norm(projer);
projer_basal = Qdg1'*Qgig2'*projer_basal;

xy = projer_basal-[0; 0; projer_basal(3)];
xy = xy/norm(xy);

x = xy(1);
y = xy(2);

sscrewb = [0 0 -G*bscrew/(2*pi)*y/(x^2+y^2);...
0 0 G*bscrew/(2*pi)*x/(x^2+y^2);...
-G*bscrew/(2*pi)*y/(x^2+y^2) G*bscrew/(2*pi)*x/(x^2+y^2) 0];
D = G*bedge/(2*pi*(1-nu));
sedgeb = [-D*y*(3*x^2+y^2)/(x^2+y^2)^2 D*x*(x^2-y^2)/(x^2+y^2)^2 0;...
D*x*(x^2-y^2)/(x^2+y^2)^2 D*y*(x^2-y^2)/(x^2+y^2)^2 0;...
0 0 nu*(-D*y*(3*x^2+y^2)/(x^2+y^2)^2+D*y*(x^2-y^2)/(x^2+y^2)^2)
];
stotbd = sscrewb+sedgeb;
stotbg1 = Qdg1*stotbd*Qdg1';
stotbg2 = Qg1g2*stotbg1*Qg1g2';

% for prismatic
mager = dot(plane',Qg1g2'*prismatic1);
projer = plane' - prismatic1*mager;
projer_prismatic1 = projer/norm(projer);
projer_prismatic1 = Qdg1'*Qgig2'*projer_prismatic1;

xy = projer_prismatic1-[0; 0; projer_prismatic1(3)];
xy = xy/norm(xy);

x = xy(1);
y = xy(2);

sscrewp1 = [0 0 -G*bscrew/(2*pi)*y/(x^2+y^2);...
0 0 G*bscrew/(2*pi)*x/(x^2+y^2);...
-G*bscrew/(2*pi)*y/(x^2+y^2) G*bscrew/(2*pi)*x/(x^2+y^2) 0];
D = G*bedge/(2*pi*(1-nu));
sedgep1 = [-D*y*(3*x^2+y^2)/(x^2+y^2)^2 D*x*(x^2-y^2)/(x^2+y^2)^2 0;...
D*x*(x^2-y^2)/(x^2+y^2)^2 D*y*(x^2-y^2)/(x^2+y^2)^2 0;...
\[
0 \ 0 \ \nu \left( -D y \left[ 3 x^2 + y^2 \right] / \left( x^2 + y^2 \right)^2 + 2 + D y \left[ x^2 - y^2 \right] / \left( x^2 + y^2 \right)^2 \right) \];
\]

\[
\text{stotp1d} = \text{sscrewp1} + \text{sedgep1};
\]
\[
\text{stotp1g1} = \text{Qdg1}' \text{stotp1d} \text{Qdg1}';
\]
\[
\text{stotp1g2} = \text{Qg1g2}' \text{stotp1g1} \text{Qg1g2}';
\]

% for prismatic2
\[
\text{mager} = \text{dot} \left( \text{plane}' , \text{Qg1g2}' \text{prismatic2} \right);
\]
\[
\text{projer} = \text{plane}' - \text{prismatic2} \text{mager} ;
\]
\[
\text{projer} \text{prismatic2d} = \text{projer} \text{norm} \left( \text{projer} \right);
\]
\[
\text{projer} \text{prismatic2d} = \text{Qdg1}' \text{Qg1g2}' \text{projer} \text{prismatic2} ;
\]
\[
\text{xy} = \text{projer} \text{prismatic2d} - \left[ 0; 0; \text{projer} \text{prismatic2d} \left( 3 \right) \right] ;
\]
\[
\text{xy} = \text{xy} / \text{norm} \left( \text{xy} \right) ;
\]
\[
\text{x} = \text{xy} \left( 1 \right) ;
\]
\[
\text{y} = \text{xy} \left( 2 \right) ;
\]

\[
\text{sscrewp2} = \left[ 0 \ 0 \ -G * \text{bscrew} / \left( 2 \pi \right) * y / \left( x^2 + y^2 \right) \right] ;
\]
\[
0 \ 0 \ G * \text{bscrew} / \left( 2 \pi \right) * x / \left( x^2 + y^2 \right) ;
\]
\[
- G * \text{bscrew} / \left( 2 \pi \right) * y / \left( x^2 + y^2 \right) \ G * \text{bscrew} / \left( 2 \pi \right) * x / \left( x^2 + y^2 \right) \ 0] ;
\]
\[
\text{D} = \text{G} * \text{bedge} / \left( 2 \pi * \left( 1 - \nu \right) \right) ;
\]
\[
\text{sedgep2} = \left[ -D y \left[ 3 x^2 + y^2 \right] / \left( x^2 + y^2 \right)^2 \ D x \left[ x^2 - y^2 \right] / \left( x^2 + y^2 \right)^2 \ 0; \ldots \right.
\]
\[
D x \left[ x^2 - y^2 \right] / \left( x^2 + y^2 \right)^2 \ D y \left[ x^2 - y^2 \right] / \left( x^2 + y^2 \right)^2 \ 0; \ldots \left. \right]
\]
\[
0 \ 0 \ \nu \left( -D y \left[ 3 x^2 + y^2 \right] / \left( x^2 + y^2 \right)^2 + D y \left[ x^2 - y^2 \right] / \left( x^2 + y^2 \right)^2 \right) \];
\]
\[
\text{stotp2d} = \text{sscrewp2} + \text{sedgep2} ;
\]
\[
\text{stotp2g1} = \text{Qdg1}' \text{stotp2d} \text{Qdg1}';
\]
\[
\text{stotp2g2} = \text{Qg1g2}' \text{stotp2g1} \text{Qg1g2}';
\]

% for prismatic3
\[
\text{mager} = \text{dot} \left( \text{plane}' , \text{Qg1g2}' \text{prismatic3} \right);
\]
\[
\text{projer} = \text{plane}' - \text{prismatic3} \text{mager} ;
\]
\[
\text{projer} \text{prismatic3d} = \text{projer} \text{norm} \left( \text{projer} \right);
\]
\[
\text{projer} \text{prismatic3d} = \text{Qdg1}' \text{Qg1g2}' \text{projer} \text{prismatic3} ;
\]
\[
\text{xy} = \text{projer} \text{prismatic3d} - \left[ 0; 0; \text{projer} \text{prismatic3d} \left( 3 \right) \right] ;
\]
\[
\text{xy} = \text{xy} / \text{norm} \left( \text{xy} \right) ;
\]
\[
\text{x} = \text{xy} \left( 1 \right) ;
\]
\[
\text{y} = \text{xy} \left( 2 \right) ;
\]
\[
\text{sscrewp3} = \left[ 0 \ 0 \ -G * \text{bscrew} / \left( 2 \pi \right) * y / \left( x^2 + y^2 \right) \right] ;
\]
\[
0 \ 0 \ G * \text{bscrew} / \left( 2 \pi \right) * x / \left( x^2 + y^2 \right) ;
\]
\[
- G * \text{bscrew} / \left( 2 \pi \right) * y / \left( x^2 + y^2 \right) \ G * \text{bscrew} / \left( 2 \pi \right) * x / \left( x^2 + y^2 \right) \ 0] ;
\]
\[
\text{D} = \text{G} * \text{bedge} / \left( 2 \pi * \left( 1 - \nu \right) \right) ;
\]
\[
\text{sedgep3} = \left[ -D y \left[ 3 x^2 + y^2 \right] / \left( x^2 + y^2 \right)^2 \ D x \left[ x^2 - y^2 \right] / \left( x^2 + y^2 \right)^2 \ 0; \ldots \right.
\]
\[
D x \left[ x^2 - y^2 \right] / \left( x^2 + y^2 \right)^2 \ D y \left[ x^2 - y^2 \right] / \left( x^2 + y^2 \right)^2 \ 0; \ldots \left. \right]
\]
\[
0 \ 0 \ \nu \left( -D y \left[ 3 x^2 + y^2 \right] / \left( x^2 + y^2 \right)^2 + D y \left[ x^2 - y^2 \right] / \left( x^2 + y^2 \right)^2 \right) \];
\]
\[
\text{stotp3d} = \text{sscrewp3} + \text{sedgep3} ;
\]
\[
\text{stotp3g1} = \text{Qdg1}' \text{stotp3d} \text{Qdg1}';
\]
\textbf{for pyramidal1}

\texttt{mager = dot(plane',Qg1g2'*pyramidal1);
projer = plane' - pyramidal1*mager;
projer_pyramidal1 = projer/norm(projer);
projer_pyramidal1d = Qdg1'*Qg1g2'*projer_pyramidal1;

xy = projer_pyramidal1d-[0; 0; projer_pyramidal1d(3)];
xy = xy/norm(xy);

x = xy(1);
y = xy(2);

sscrewP1 = [0 0 -G*bscrew/(2*pi)*y/(x^2+y^2);...
 0 0 G*bscrew/(2*pi)*x/(x^2+y^2);...
 -G*bscrew/(2*pi)*y/(x^2+y^2) G*bscrew/(2*pi)*x/(x^2+y^2) 0];

D = G*bedge/(2*pi*(1-nu));

sedgeP1 = [-D*y*(3*x^2+y^2)/(x^2+y^2)^2 D*x*(x^2-y^2)/(x^2+y^2)^2 0;...
 D*x*(x^2-y^2)/(x^2+y^2)^2 D*y*(x^2-y^2)/(x^2+y^2)^2 0;...
 0 0*(-D*y*(3*x^2+y^2)/(x^2+y^2)^2+D*y*(x^2-y^2)/(x^2+y^2)^2)/(x^2+y^2)^2];

stotP1d = sscrewP1+sedgeP1;

stotP1g1 = Qdg1*stotP1d*Qdg1';
stotP1g2 = Qg1g2*stotP1g1*Qg1g2';

\textbf{for pyramidal2}

\texttt{mager = dot(plane',Qg1g2'*pyramidal2);
projer = plane' - pyramidal2*mager;
projer_pyramidal2 = projer/norm(projer);
projer_pyramidal2d = Qdg1'*Qg1g2'*projer_pyramidal2;

xy = projer_pyramidal2d-[0; 0; projer_pyramidal2d(3)];
xy = xy/norm(xy);

x = xy(1);
y = xy(2);

sscrewP2 = [0 0 -G*bscrew/(2*pi)*y/(x^2+y^2);...
 0 0 G*bscrew/(2*pi)*x/(x^2+y^2);...
 -G*bscrew/(2*pi)*y/(x^2+y^2) G*bscrew/(2*pi)*x/(x^2+y^2) 0];

D = G*bedge/(2*pi*(1-nu));

sedgeP2 = [-D*y*(3*x^2+y^2)/(x^2+y^2)^2 D*x*(x^2-y^2)/(x^2+y^2)^2 0;...
 D*x*(x^2-y^2)/(x^2+y^2)^2 D*y*(x^2-y^2)/(x^2+y^2)^2 0;...
 0 0*(-D*y*(3*x^2+y^2)/(x^2+y^2)^2+D*y*(x^2-y^2)/(x^2+y^2)^2)/(x^2+y^2)^2];

stotP2d = sscrewP2+sedgeP2;

stotP2g1 = Qdg1*stotP2d*Qdg1';
stotP2g2 = Qg1g2*stotP2g1*Qg1g2';

\textbf{for pyramidal3}

\texttt{mager = dot(plane',Qg1g2'*pyramidal3);}
projer = plane' - pyramidal3*mager;
projer_pyramidal3 = projer/norm(projer);
projer_pyramidal3d = Qdg1'*Qg1g2'*projer_pyramidal3;

xy = projer_pyramidal3d-[0; 0; projer_pyramidal3d(3)];
xy = xy/norm(xy);

x = xy(1);
y = xy(2);

sscrewP3 = [0 0 -G*bscrew/(2*pi)*y/(x^2+y^2);...
 0 0 G*bscrew/(2*pi)*x/(x^2+y^2);...
-G*bscrew/(2*pi)*y/(x^2+y^2) G*bscrew/(2*pi)*x/(x^2+y^2) 0];

D = G*bedge/(2*pi*(1-nu));
sedgeP3 = [-D*y*(3*x^2+y^2)/(x^2+y^2)^2 D*x*(x^2-y^2)/(x^2+y^2)^2 0;...
 D*x*(x^2-y^2)/(x^2+y^2)^2 D*y*(x^2-y^2)/(x^2+y^2)^2 0;...
 0 0 nu*(-D*y*(3*x^2+y^2)/(x^2+y^2)^2+D*y*(x^2-y^2)/(x^2+y^2)^2) ];

stotP3d = sscrewP3+sedgeP3;
stotP3g1 = Qdg1*stotP3d*Qdg1';
stotP3g2 = Qg1g2*stotP3g1*Qg1g2';

% for pyramidal4
mager = dot(plane',Qg1g2'*pyramidal4);
projer = plane' - pyramidal4*mager;
projer_pyramidal4 = projer/norm(projer);
projer_pyramidal4d = Qdg1'*Qg1g2'*projer_pyramidal4;

xy = projer_pyramidal4d-[0; 0; projer_pyramidal4d(3)];
xy = xy/norm(xy);

x = xy(1);
y = xy(2);

sscrewP4 = [0 0 -G*bscrew/(2*pi)*y/(x^2+y^2);...
 0 0 G*bscrew/(2*pi)*x/(x^2+y^2);...
-G*bscrew/(2*pi)*y/(x^2+y^2) G*bscrew/(2*pi)*x/(x^2+y^2) 0];

D = G*bedge/(2*pi*(1-nu));
sedgeP4 = [-D*y*(3*x^2+y^2)/(x^2+y^2)^2 D*x*(x^2-y^2)/(x^2+y^2)^2 0;...
 D*x*(x^2-y^2)/(x^2+y^2)^2 D*y*(x^2-y^2)/(x^2+y^2)^2 0;...
 0 0 nu*(-D*y*(3*x^2+y^2)/(x^2+y^2)^2+D*y*(x^2-y^2)/(x^2+y^2)^2) ];

stotP4d = sscrewP4+sedgeP4;
stotP4g1 = Qdg1*stotP4d*Qdg1';
stotP4g2 = Qg1g2*stotP4g1*Qg1g2';

% for pyramidal5
mager = dot(plane',Qg1g2'*pyramidal5);
projer = plane' - pyramidal5*mager;
projer_pyramidal5 = projer/norm(projer);
projer_pyramidal5d = Qdg1'*Qg1g2'*projer_pyramidal5;
\( \text{proj}_{\text{pyramidal5d}} = [0; 0; \text{proj}_{\text{pyramidal5d}}(3)]; \)
\( \text{xy} = \text{proj}_{\text{pyramidal5d}}/\text{norm} (\text{proj}_{\text{pyramidal5d}}); \)
\( \text{x} = \text{xy}(1); \)
\( \text{y} = \text{xy}(2); \)
\[
\text{sscrewP5} = [0 0 \text{G-bscrew/(2*pi)} y/(x^2+y^2); \ldots
\quad 0 0 \text{G-bscrew/(2*pi)} x/(x^2+y^2); \ldots
\quad -\text{G-bscrew/(2*pi)} y/(x^2+y^2) \text{G-bscrew/(2*pi)} x/(x^2+y^2); 0]; \]
\( \text{D} = \text{G-bedge/(2*pi*(1-nu))}; \)
\( \text{sedgeP5} = [-D*y*(3*x^2+y^2)/(x^2+y^2)^2 \text{D*x}*(x^2-y^2)/(x^2+y^2)^2 0; \ldots
\quad \text{D*x}*(x^2-y^2)/(x^2+y^2)^2 \text{D*y}*(x^2-y^2)/(x^2+y^2)^2 0; \ldots
\quad 0 0 \text{n}u*(-D*y*(3*x^2+y^2)/(x^2+y^2)^2 \text{D*y}*(x^2-y^2)/(x^2+y^2)^2; 0)]; \)
\( \text{stotP5d} = \text{sscrewP5+sedgeP5}; \)
\( \text{stotP5g1} = \text{Qdg1*stotP5d*Qdg1}'; \)
\( \text{stotP5g2} = \text{Qg1g2*stotP5g1*Qg1g2}'; \)

% for pyramidal6
\( \text{mager} = \text{dot} (\text{plane}',\text{Qg1g2}^\ast \text{pyramidal6}); \)
\( \text{projer} = \text{plane'} - \text{pyramidal6*mager}; \)
\( \text{proj}_{\text{pyramidal6}} = \text{projer}/\text{norm} (\text{projer}); \)
\( \text{proj}_{\text{pyramidal6d}} = \text{projer}/\text{norm} (\text{projer}); \)
\( \text{xy} = \text{proj}_{\text{pyramidal6d}}/\text{norm} (\text{proj}_{\text{pyramidal6d}}); \)
\( \text{x} = \text{xy}(1); \)
\( \text{y} = \text{xy}(2); \)
\[
\text{sscrewP6} = [0 0 \text{G-bscrew/(2*pi)} y/(x^2+y^2); \ldots
\quad 0 0 \text{G-bscrew/(2*pi)} x/(x^2+y^2); \ldots
\quad -\text{G-bscrew/(2*pi)} y/(x^2+y^2) \text{G-bscrew/(2*pi)} x/(x^2+y^2); 0]; \]
\( \text{D} = \text{G-bedge/(2*pi*(1-nu))}; \)
\( \text{sedgeP6} = [-D*y*(3*x^2+y^2)/(x^2+y^2)^2 \text{D*x}*(x^2-y^2)/(x^2+y^2)^2 0; \ldots
\quad \text{D*x}*(x^2-y^2)/(x^2+y^2)^2 \text{D*y}*(x^2-y^2)/(x^2+y^2)^2 0; \ldots
\quad 0 0 \text{n}u*(-D*y*(3*x^2+y^2)/(x^2+y^2)^2 \text{D*y}*(x^2-y^2)/(x^2+y^2)^2; 0)]; \)
\( \text{stotP6d} = \text{sscrewP6+sedgeP6}; \)
\( \text{stotP6g1} = \text{Qdg1*stotP6d*Qdg1}'; \)
\( \text{stotP6g2} = \text{Qg1g2*stotP6g1*Qg1g2}'; \)

% apply the stress tensor to all possible slip systems (going off of % Microstructure Sensitive Design pg 118) % planes

% slip directions in four index notation
\( \text{d4} = \text{zeros}(4,9); \)
\( \text{d4(:,1)} = [1;1;-2;0]; \)
\( \text{d4(:,2)} = [1;-2;1;0]; \)
\( \text{d4(:,3)} = [-2;1;1;0]; \)

255
\[ d4(:,4) = [1; 1; -2; 3]; \]
\[ d4(:,5) = [1; -2; 1; 3]; \]
\[ d4(:,6) = [-2; 1; 1; 3]; \]
\[ d4(:,7) = [1; 1; -2; -3]; \]
\[ d4(:,8) = [1; -2; 1; -3]; \]
\[ d4(:,9) = [-2; 1; 1; -3]; \]

% convert to three index notation
\[ d = zeros(3,9); \]
\[ \text{for } i = 1:9 \]
\[ \quad d(:,i) = (S*rot434(d4(:,i))')'; \]
\[ \text{end} \]

% calculate resolved shear stress on each system
% basal plane
\[ \text{tauRSS(1)} = \text{sum(sum(stotBg2.*dyadic(d(:,1),basal))); } \% (0001)[11-20] \]
\[ \text{tauRSS(2)} = \text{sum(sum(stotBg2.*dyadic(d(:,2),basal))); } \% (0001)[1-210] \]
\[ \text{tauRSS(3)} = \text{sum(sum(stotBg2.*dyadic(d(:,3),basal))); } \% (0001)[-2110] \]

% prismatic1 plane
\[ \text{tauRSS(4)} = \text{sum(sum(stotplg2.*dyadic(d(:,1),prismatic1))); } \% (1-100)[11-20] \]
\[ \text{tauRSS(5)} = \text{sum(sum(stotplg2.*dyadic(d(:,4),prismatic1))); } \% (1-100)[11-23] \]
\[ \text{tauRSS(6)} = \text{sum(sum(stotplg2.*dyadic(d(:,7),prismatic1))); } \% (1-100)[1-2-3] \]

% prismatic2 plane
\[ \text{tauRSS(7)} = \text{sum(sum(stotp2g2.*dyadic(d(:,2),prismatic2))); } \% (10-10)[11-20] \]
\[ \text{tauRSS(8)} = \text{sum(sum(stotp2g2.*dyadic(d(:,5),prismatic2))); } \% (10-10)[11-23] \]
\[ \text{tauRSS(9)} = \text{sum(sum(stotp2g2.*dyadic(d(:,8),prismatic2))); } \% (10-10)[1-2-3] \]

% prismatic3 plane
\[ \text{tauRSS(10)} = \text{sum(sum(stotp3g2.*dyadic(d(:,3),prismatic3))); } \% (01-10)[-2110] \]
\[ \text{tauRSS(11)} = \text{sum(sum(stotp3g2.*dyadic(d(:,6),prismatic3))); } \% (01-10)[-2113] \]
\[ \text{tauRSS(12)} = \text{sum(sum(stotp3g2.*dyadic(d(:,9),prismatic3))); } \% (01-10)[-2113] \]

% pyramidal1 plane
\[ \text{tauRSS(13)} = \text{sum(sum(stotPlg2.*dyadic(d(:,1),pyramidal1))); } \% (1-101)[11-20] \]
\[ \text{tauRSS(14)} = \text{sum(sum(stotPlg2.*dyadic(d(:,8),pyramidal1))); } \% (1-101)[-12-13] \]
\[ \text{tauRSS(15)} = \text{sum(sum(stotPlg2.*dyadic(d(:,6),pyramidal1))); } \% (1-101)[-2113] \]

% pyramidal2 plane
\[ \text{tauRSS(16)} = \text{sum(sum(stotp2g2.*dyadic(d(:,2),pyramidal2))); } \% (10-11)[1-210] \]
\[ \text{tauRSS(17)} = \text{sum(sum(stotp2g2.*dyadic(d(:,7),pyramidal2))); } \% (10-11)[11-2-3] \]
\[ \text{tauRSS(18)} = \text{sum(sum(stotp2g2.*dyadic(d(:,6),pyramidal2))); } \% (10-11)[-2113] \]

% pyramidal3 plane
\[ \text{tauRSS(19)} = \text{sum(sum(stotp3g2.*dyadic(d(:,3),pyramidal3))); } \% (01-11)[-2110] \]
\[ \text{tauRSS(20)} = \text{sum(sum(stotp3g2.*dyadic(d(:,5),pyramidal3))); } \% (01-11)[-2113] \]
\[ \text{tauRSS(21)} = \text{sum(sum(stotp3g2.*dyadic(d(:,7),pyramidal3))); } \% (01-11)[-2113] \]

% pyramidal4 plane
\[ \text{tauRSS(22)} = \text{sum(sum(stotP4g2.*dyadic(d(:,1),pyramidal4))); } \% (1-10-1)[11-20] \]
\[ \text{tauRSS(23)} = \text{sum(sum(stotP4g2.*dyadic(d(:,5),pyramidal4))); } \% (1-10-1)[-12-1-3] \]
\[ \text{tauRSS(24)} = \text{sum(sum(stotP4g2.*dyadic(d(:,9),pyramidal4))); } \% (1-10-1)[-211-3] \]

256
% pyramidal5 plane
tauRSS(25) = sum(sum(stotP5g2.*dyadic(d(:,2),pyramidal5))); % (10-1-1) [1-210]
tauRSS(26) = sum(sum(stotP5g2.*dyadic(d(:,4),pyramidal5))); % (10-1-1) [11-23]
tauRSS(27) = sum(sum(stotP5g2.*dyadic(d(:,9),pyramidal5))); % (10-1-1) [-211-3]

% pyramidal6 plane
tauRSS(28) = sum(sum(stotP6g2.*dyadic(d(:,3),pyramidal6))); % (01-1-1) [-2110]
tauRSS(29) = sum(sum(stotP6g2.*dyadic(d(:,8),pyramidal6))); % (01-1-1) [1-21-3]
tauRSS(30) = sum(sum(stotP6g2.*dyadic(d(:,4),pyramidal6))); % (01-1-1) [11-23]

tauRSS = abs(tauRSS);
figure, plot(tauRSS)

A.5 HCP plane spacing and angle

Calculates the ratio between plane spacings and angle between plane normals for HCP crystal lattices with a known c/a ratio. Equations are mostly taken from the electron tomography books by Williams and Carter. Useful code as the diffraction patterns for Ti in both Edington’s and William and Carter’s electron microscopy books contain mistakes.

% Calculates the angle between two plane normals and ratios of the plane % spacing for an HCP lattice

clear all
close all

% input lattice parameters and c/a ratio
a = 2.950; % Units don’t matter as long as they are consistent
c = 4.683;
caRat = c/a;
lambda = sqrt(2/3)*caRat;

% Input the vectors in 4 index notation
vec1 = [1 -1 0 1];
vec2 = [1 0 -1 1];

% quick check
if abs(vec1(1)+vec1(2)+vec1(3)) > 1e-15 || abs(vec2(1)+vec2(2)+vec2(3)) > 1e-15
disp('you put in a bad vector')
return
end

% output the angle between the vectors in degrees
theta =
acosd((vec1(1)*vec2(1)+vec1(2)*vec2(2)+0.5*(vec1(1)*vec2(2)+vec1(2)*vec2(1)) +
0.75*vec1(4)*vec2(4)*(1/caRat)^2)/ ... 
(sqrt(vec1(1)^2+vec1(2)^2+vec1(1)*vec1(2)+0.75*vec1(4)^2*(1/caRat)^2)*sqrt(ve
c2(1)^2+vec2(2)^2+vec2(1)*vec2(2)+0.75*vec2(4)^2*(1/caRat)^2)));

% if theta > 90
% theta = 180-theta;
% end
disp('theta =')
disp(theta)

% convert to three index notation
v13(1) = 3/2*vec1(1);
v13(2) = sqrt(3)/2*(vec1(1)+2*vec1(2));
v13(3) = vec1(4);

v23(1) = 3/2*vec2(1);
v23(2) = sqrt(3)/2*(vec2(1)+2*vec2(2));
v23(3) = vec2(4);

% calculate the plane d-spacing
d1 = 1/sqrt(4/9*(v13(1)^2+v13(2)^2)/a^2+v13(3)^2/c^2);
d2 = 1/sqrt(4/9*(v23(1)^2+v23(2)^2)/a^2+v23(3)^2/c^2);

% calculate the ratio of the g-vectors
disp('g1/g2 = ')
rat = d2/d1;
disp(rat)

A.6 FCC diffraction patterns and twin spots

Calculates FCC diffraction patterns and twin spot locations.

% write a code to calculate a twinned diffraction pattern. Set up for fcc % patterns
clear all
close all

% enter the zone that you're looking down
u = 0;
v = 3;
w = 1;
% enter the twin plane
p = 1;
q = 1;
r = 1;

% enter the spot that you want mirrored. Note that this spot must be
% perpendicular to uvw
H = 0;
K = -1;
L = 3;

% figure out all the spots that will be present in the pattern with the max
% index being 6
m = 1;
for h = -6:6
    for k = -6:6
        for l = -6:6
            if mod(h,2) == 0  && mod(k,2) == 0 && mod(l,2) == 0
                if h*u+k*v+l*w == 0
                    hklvis{m} = [h k l];
                    hklvis{m+1} = -[h k l];
                    m = m+1;
                end
            end
            if mod(h,2) == 1  && mod(k,2) == 1 && mod(l,2) == 1
                if h*u+k*v+l*w == 0
                    hklvis{m} = [h k l];
                    hklvis{m+1} = -[h k l];
                    m = m+1;
                end
            end
        end
    end
end

% calculate the vector for the twin spot
hp = (p*(p*H+2*q*K+2*r*L)-H*(q^2+r^2))/(p^2+q^2+r^2);
kp = (q*(2*p*H+q*K+2*r*L)-K*(p^2+r^2))/(p^2+q^2+r^2);
lp = (r*(2*p*H+2*q*K+r*L)-L*(q^2+r^2))/(p^2+q^2+r^2);

% Figure out how to plot the spots
map = ones(1001,1001);
center = round(length(map)/2);
map(center-5:center+5,center-5:center+5) = 0;

% set up coordinate frame
% uvw frame
uvw = [u; v; w]/sqrt(u^2+v^2+w^2);
HKL = [H; K; L]/sqrt(H^2+K^2+L^2); % this must be orthoganol to uvw
third = cross(uvw,HKL);
third = third/norm(third);

% create direction cosine matrix to rotate reference frame into something
% more manageable
gmat = [third(1) HKL(1) uvw(1)];
    third(2) HKL(2) uvw(2);
third(3) HKL(3) uvw(3)];

% rotate all these into a plot-able frame (so that the z direction is 0)
% hklplot = cell(size(hklvis));
for i = 1:length(hklvis)
    hklplot = gmat'*hklvis(i);
    if abs(hklplot(1)*100) < center-10 && abs(hklplot(2)*100) < center-10
        map(center-5+round(hklplot(1)*100):center+5+round(hklplot(1)*100),
            center-5+round(hklplot(2)*100):center+5+round(hklplot(2)*100)) = 0;
    end
end

% % plot the twin spot
% twinspot = gmat*[hp; kp; lp];
% map(center-5+round(twinspot(1)*100):center+5+round(twinspot(1)*100),
%     center-5+round(twinspot(2)*100):center+5+round(twinspot(2)*100)) = 0.5;

% Or maybe I can just figure out what the zone axis will be from the
% twinned grain, and plot all the correlating spots
% gmatTwin = AA2GMat([1 1 1],60);
% uvwTwin = gmatTwin*[u; v; w];
% HKLTwin = gmatTwin*[H; K; L];

% figure out all the spots that will be present in the twin pattern with the
% max
% index being 6
m = 1;
for h = -6:6
    for k = -6:6
        for l = -6:6
            if mod(h,2) == 0 && mod(k,2) == 0 && mod(l,2) == 0
                if h*uvwTwin(1)+k*uvwTwin(2)+l*uvwTwin(3) == 0
                    hklvis{m} = [h k l];
                    hklvis{m+1} = [-h k l];
                    m = m+1;
                end
            end
            if mod(h,2) == 1 && mod(k,2) == 1 && mod(l,2) == 1
                if h*uvwTwin(1)+k*uvwTwin(2)+l*uvwTwin(3) == 0
                    hklvis{m} = [h k l];
                    hklvis{m+1} = [-h k l];
                    m = m+1;
                end
            end
        end
    end
end

% set up coordinate frame
% uvw frame
uvwTwin = uvwTwin/norm(uvwTwin);
HKLTwin = HKLTwin/norm(HKLTwin); % this must be orthoganol to uvw
thirdTwin = cross(uvwTwin,HKLTwin);
thirdTwin = thirdTwin/norm(thirdTwin);
% create direction cosine matrix to rotate reference frame into something more manageable

\[
gmat = \begin{bmatrix}
\text{thirdTwin}(1) & \text{HKLTwin}(1) & \text{uvwTwin}(1); \\
\text{thirdTwin}(2) & \text{HKLTwin}(2) & \text{uvwTwin}(2); \\
\text{thirdTwin}(3) & \text{HKLTwin}(3) & \text{uvwTwin}(3) \\
\end{bmatrix}
\]

% rotate all these into a plot-able frame (so that the z direction is 0)

\[
hklplot = \text{cell}(\text{size(hklvis)});
\]

\[
\text{for } i = 1:\text{length(hklvis)}
\quad \text{hklplot} = \text{gmat}'*\text{hklvis}(i)';
\quad \text{if} \ \text{abs(hklplot(1)*100)} < \text{center}-10 \ \&\& \ \text{abs(hklplot(2)*100)} < \text{center}-10
\quad \quad \text{map}(
\quad \quad \text{center}-5+\text{round(hklplot(1)*100)}:\text{center}+5+\text{round(hklplot(1)*100)},...
\quad \quad \text{center}-5+\text{round(hklplot(2)*100)}:\text{center}+5+\text{round(hklplot(2)*100)}) = 0.5;
\quad \end{\text{if}}
\quad \text{end}
\text{end}
\]

\[
\text{imagesc(map), colormap(gray), axis equal}
\]