Multiscale Experimental Analysis in Plasticity:
Linking Dislocation Structures to Continuum Fields

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Abstract

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Plastic deformation in metals is a complex phenomenon and is result of competition between different complicated mechanisms, and among all, dislocation nucleation and motion are the most dominant ones. Dislocation evolution is known to be a multi-scale phenomenon, and has been incorporated to crystal plasticity theories to analyze the size effect in metals for almost a decade ago. Although the theories suffice to predict the size effect in metals, they are largely phenomenological. Here a novel experimental method is developed to resolve the complexity in plastic deformation due to dislocations and to extract new material length scales that can be incorporated to numerical models. A continuum-based quantity: the geometrically necessary dislocation density (GND) that describes the signed part of the overall dislocations is measured on a nickel single crystal sample using recently developed high resolution electron backscatter diffraction (HR-EBSD) over different field of view, $90 \mu m^2 - 1 \ mm^2$ with various step sizes, $50 \ nm$ to $2,500 \ nm$. The net Burgers vector density, which includes the information of the direction of the overall dislocation motion and also quantifies the flux of atoms changing positions due to dislocations, is measured for the first time using continuum methods. A new parameter, $\beta$, that is extracted from the net Burger vector density to monitor dislocation activity on crystallographic slip planes is measured. Measurements reveals patterning in GND densities and a distribution of length scales rather than a single length scale as assumed. The length scales, such as dislocation spacing, and dislocation cell sizes are
quantified. The linear relationship between dislocation spacing and dislocation cell size is obtained, where the slope of the line varies with activation of different crystallographic slip systems and the number of the active slip systems. The slope ranges between $23 - 29$ for dominantly single slip regions, whereas it ranges between $13 - 16$ for multislip regions, which agrees with the findings from TEM analysis in the literature showing how a continuum based method can be used to obtain same material parameters.

The experimental measurements and the assumptions are elaborated in a detailed analysis. The effect of step size in EBSD results is presented, and the information loss with increasing the step size is shown. The uncertainty in GND density from the HR-EBSD measurements is found to be $10^{13}$ m$^{-2}$, which is two order of magnitude less than results from traditional diffraction methods.

The effect of dislocation mobility on microstructure evolution has been also investigated, specifically tantalum single crystal specimens tested at $77 K$ and $293 K$. The results unraveled occurrences of different deformation mechanisms: kink shear, and twinning at low temperatures. Interactions between dislocations and twin formations are observed and striking microstructure differences are examined. The dislocations density measurement results on tantalum are unique in the experimental sense and data can be used to extract length scale information.

The experimental observations have been exploited to build the foundations of a numerical model. The effect of microstructure evolution on mechanical response has been investigated numerically based upon experimental observations. One of the main outcome of the experimental analysis -the variation of GND densities in cell walls- has been incorporated into a strain gradient plasticity framework. The proposed model is demonstrated with constrained shear and pure bending problems. The results presented show patterning in the GND density profile depending on the
prescribed initial variation of the saturation value of GND densities and also change in overall mechanical response depending on the complexity of the prescribed profile.
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Dedication

Dedicated to My Family
CHAPTER 1

Introduction

The aim of this study is to enhance understanding the mechanical behavior of metals under high strain and high strain gradient conditions where permanent deformation—the plastic deformation—is induced. The field of metal plasticity has been a fundamental topic of research for many decades. In the past twenty years, much effort has gone into investigating the length scale effect in plasticity whereby a metal becomes stronger as the dimensions of a specimen decrease to the order of a few tens of micrometers [3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20]. This phenomenon is contrary to experiments of plastic deformation in metals for macroscopic specimens, where the stress at which plasticity initiates and as the flow stress evolves is independent of the specimen size.

Plasticity is an inherently multiscale phenomenon, and it occurs as a result of different mechanisms such as dislocations, diffusion, grain boundary sliding and twinning. The first among all is dislocation nucleation and motion on crystallographic planes which directly contributes to the flow and strengthening of the metal [21]. The dislocations contribute to strengthening, because they accumulate in the material either to accommodate an otherwise incompatible deformation field or they are trapped in a random way. Dislocations that accumulate under strain gradient fields to accommodate an otherwise incompatible deformation are called geometrically necessary dislocations (GND); the rest are called statistically stored dislocations (SSD) [22, 23]. A continuum interpretation of GNDs are through GND density, which is a form of defect that evolves within a material and introduces length scales in it. In order to account for these length scales, many researchers have developed computational mod-
els that employ GND densities [4, 5, 6, 16, 24, 25, 26, 27], some other researchers have tried to measure dislocation densities using diffraction methods [28, 29, 30, 31, 32]. However, in these studies, multiscale characteristics of plastic deformation has not been investigate through GND densities.

In this study, a new multiscale experimental method is developed to measure the GND densities in single crystal samples that are deformed by a wedge indenter. Measurements are taken using a recently developed high-resolution electron back-scatter diffraction (HR-EBSD) method with step sizes ranging from 50 nm up to 2500 nm over different fields of view that ranges from a few square micrometers to a square millimeter. HR-EBSD provides considerably enhanced accuracy that enables calculation of the net Burger vector density, which quantifies the flux of the overall GND motion resulting from an applied deformation. A new quantity, $\beta$ which characterize the activity of slip systems is extracted from the net Burgers vector density. The experimental measurements reveal striking features in GNDs and also patterning in GND density distribution. Among all of these features, the most remarkable ones are the quasi-periodic dislocation walls, which are seen at accessible with all step sizes (50 nm up to 2500 nm) and the dislocation cells, which were visible at higher resolutions (50 nm up to 100 nm). Cell wall dislocation structures were previously studied via transmission electron microscope (TEM) by several researchers [33, 34, 2, 35, 36]. The dislocations that were resolved on TEM micrographs are counted using an observation based method to quantify dislocation densities, dislocation spacings and dislocation cell sizes. However these methods are constrained with the limitations of TEM: tedious sample preparation in which the material characteristic and microstructure may change, sample size limitation which makes it hard to study a multiscale analysis, completely subjective dislocation density measurements capabilities, which can not resolve regions with high dislocation densities and can not distinguish between GNDs from SSDs.
In the present study, quasi-periodic dislocation structures are identified systematically and they are processed with an objective method to quantify dislocation cell sizes and dislocation spacings. The results showed a linear relationship between peak dislocation spacing in cell walls and dislocation cell sizes, which agrees with TEM based methods. This analysis not only validates the experimental results but also proves how a continuum-based quantity, GND densities, can be linked to discrete quantities such as dislocation cell sizes and dislocation spacing. This study also constitutes the basis of HR-GND microscopy, that allows an SEM-based analysis to obtain results previously accessible only via TEM methods. The multiscale analysis is presented in Chapter 2 and details on experimental procedures and experimental variables such as step size and angular accuracy in measurements are presented in Chapter 3.

Experimental measurements on indented single crystal samples have revealed non-homogeneous distributions of GND densities and distributions of different length scales. Maximum dislocation densities measured in indented single crystal samples are on the order of $10^{14} - 10^{15} \text{m}^{-2}$; most attain maxima in cell walls or in randomly distributed dislocation bundles. This type of patterning cannot be simulated with standard strain gradient plasticity models with quadratic energy formulations presented in earlier works [6, 37, 38, 39, 40, 41, 42]. In this study, a GND based strain gradient plasticity framework is extended by incorporating a physical quantity obtained from experimental observations: the saturation value of GND densities. A new hardening rule based on saturation values and the profile of GND density distribution is proposed. The proposed model is tested with constrained shear and pure bending problems. The results show patterning in the GND density profile depending on the prescribed initial variation of the saturation value of GND densities. The mechanical response of the material also show a strong dependency in hardening. The hardening rate increases with an increase in complexity in initial GND variation prescribed. The details of the numerical modeling are presented in Chapter 4.
This dissertation also includes another experimental study, in which the mechanical response of tantalum single crystals under different deformation conditions is investigated. Lattice rotation and subsequently GND density measurements on indented tantalum single crystal samples at room temperature and at cryogenic temperature will be presented. In tantalum during plastic deformation, GNDs are accompanied by twinning specifically at low temperatures, different than previous experimental studies on aluminum [43], copper [32] and nickel [1]. The HR-EBSD results with brief discussion about differences in dislocation structures at different temperatures are given in Chapter 5.

In summary, this dissertation focuses on material characterization over different length scales and is extended to different deformation conditions. The experimental technique developed in this study is unique in the way that it exploits a continuum concept to analyze a discrete problem: GND densities. The method, enables measurement of new quantities related to deformation, such as net Burger vector densities as well as $\beta$, and it quantifies a distribution of characteristic length scales in a multiscale perspective. The numerical analysis part of this dissertation, exploits the experimental observations and proposes a new method to see the effect of periodic variations in dislocation density evolution and overall mechanical response. The measurements on Ta unravels the competition of different deformation mechanisms at low temperature. The experimental measurements are unique, since similar types of measurement have not been done neither in tantalum at any temperature, nor in any other material deformed under cryogenic temperatures.
CHAPTER 2

Multiscale Experimental Analysis

2.1. Abstract

One of the fundamental challenges of materials science and applied mechanics is to predict the mechanical properties of materials from first principles. During reversible (i.e. elastic) deformation, atoms within a crystalline material remain fixed to crystal lattice positions but the underlying lattice distorts. First principles methods can be used to compute elastic properties because relative atomic positions are well-defined. However during irreversible (i.e. plastic) deformation, the crystal lattice distorts while atoms flow through the crystal lattice positions facilitated by the motion of crystalline line defects called dislocations. The mechanical properties of such an elastic-plastic material depend predominantly upon the stress—and the evolution of that stress as deformation proceeds—at which the atoms flow through the crystal lattice. That the underlying atomic configuration of the material changes during plastic deformation constitutes a formidable challenge to the development of predictive models of the deformation of materials.

Many transmission electron microscopy (TEM) studies have quantified the behavior of dislocations at very small length scales, but in general the studies are not well correlated with the macroscopic deformation state induced into the material. In this study, we report measurements of dislocation structures using High Resolution Geometrically Necessary Dislocation (HR-GND) microscopy based upon a scanning electron microscope (SEM), rather than a TEM. The measurements are multiscale in the sense that the step size of the measurements vary from 50 nm to 2500 nm over
fields of view from 1 square micrometer to 1 square millimeter. We observe the spatial variations of GND densities in various regions of the indented crystal and interpret the peaks in GND density to be incipient dislocation cell walls. We find the experimental relationship between the peak magnitude of the GND densities and the average cell wall spacing (i.e. cell size)—the results are consistent with TEM measurements in the literature. Thus, our results directly connect the macroscopic loading with the underlying dislocation distribution induced in the material.

2.2. Fundamentals

2.2.1. Dislocations. Plastic deformation in a ductile material—perhaps the most common form of inelastic deformation—occurs as a consequence of shear slip deformation between two neighboring atomic planes in the crystal lattice—similar to deformation of the earth’s crust during an earthquake induced by shear slip between two adjacent plates across a fault. In a crystalline material, the boundary separating the slipped and unslipped regions between two atomic planes is a line defect within the crystal lattice called a dislocation loop. As the slipped region expands, the dislocation loop moves through the crystal lattice and induces mass flux as the atoms flow through the crystal lattice.

Dislocations are classified by three directions within the crystal lattice as well as having a signed character. The line tangent of the dislocation loop is denoted by a unit vector, \( \mathbf{d} \), that varies with position along the loop. The shear slip in the crystal caused by the passage of a dislocation occurs on a crystallographic \textit{slip plane} denoted by the unit normal vector \( \mathbf{n} \) and in a crystallographic \textit{slip direction} denoted by the unit vector \( \mathbf{s} \) that together define a \textit{slip system}; the slip plane and slip direction are orthogonal and each typically remains constant around a \textit{dislocation} loop. At the position of the loop where \( \mathbf{s} \) is perpendicular to \( \mathbf{d} \), the dislocation is said to have edge character, and when \( \mathbf{s} \) is parallel to \( \mathbf{d} \), the dislocation is said to have screw character;
other portions of a dislocation loop are said to be of mixed edge-screw character. Each segment of a dislocation loop has a corresponding segment for which the local line tangent vectors are antiparallel. These two segments annihilate each other or form dipoles if brought sufficiently close together, so if the segments are considered individually, one segment is labeled arbitrarily as being positive and the other is labeled as being negative. Dislocations exist on multiple families of slip systems, on several crystallographically equivalent slip systems within a family of slip systems, as well as on many individual atomic planes within each of the crystallographically equivalent slip systems. To complicate matters, various types of dislocation sources can be activated such that a plethora of dislocations can exist on the individual atomic planes. In addition to their slip and sign characteristics, dislocations are quantified in terms of their volume density, defined as the total dislocation line length per unit volume, in units of inverse length squared.

The amount of plastic slip induced by the passage of a dislocation is given in terms of its Burgers vector, $\mathbf{b}$, with magnitude $b$ and direction $\mathbf{s}$. As illustrated in Fig. 2.1a, the Burgers vector can be determined via an elementary construction called the Burgers circuit whereby, following the $SF/RH$ convention, a closed circuit is made around an undeformed defect-free region of an atomic plane in the right-handed sense relative to the unit normal, $\ell$, of the plane while counting the lattice sites from the start point, $S$, to the coincident finish point, $F$. Upon mapping the circuit to a region of the crystal pierced by a dislocation, but with an otherwise undeformed crystal lattice away from the dislocation, the points $S$ and $F$ are no longer coincident, so the circuit is not closed. The Burgers vector is the lattice vector from $S$ to $F$ in the mapped configuration, as shown in Fig. 2.1b, where the edge dislocation is symbolized by the $\perp$ symbol, with the horizontal line representing the slip plane and and the vertical line representing the extra material associated with the dislocation.
Figure 2.1. Burgers circuit constructions: a) Defect-free undeformed crystal lattice plane with closed circuit starting at \( S \) and finishing at the coincident point \( F \); b) burgers vector due to introduction of edge dislocation; c) Burgers circuit around a group of dislocations d) net burgers vector.
A Burgers circuit can also be taken around a group of dislocations of different slip systems by taking a closed circuit, $\Gamma$, in an undeformed defect-free reference configuration on a crystallographic plane with unit normal vector, $\ell$, as in Fig. 2.1c, and mapping it to a region pierced by many dislocations, but with an otherwise undisturbed crystal lattice that retains its equilibrium spacing far away from the dislocation. The net Burgers vector (also called the closure failure vector), denoted as $\mathbf{b}_\text{net}$, is the lattice vector from $\mathcal{S}$ to $\mathcal{F}$ in the mapped configuration necessary to close the circuit, as in Fig. 2.1d; the superscript $p$ indicates that the net Burgers vector is due to plastic deformation. The value of $\mathbf{b}_\text{net}$ represents the vector sum of the Burgers vectors of all dislocations that pierce the atomic plane within the circuit. Any pair of dislocations with equal and opposite Burgers vectors do not contribute to $\mathbf{b}_\text{net}$, and are said to be redundant. Thus $\mathbf{b}_\text{net}$ consists of the sum of the Burgers vectors of the remaining net signed dislocations, that are said to be non-redundant. The quantities $\mathbf{b}$ and $\mathbf{b}_\text{net}$ are routinely measured via High Resolution Transmission Electron Microscopy (HRTEM) by quantifying the total total number of discrete dislocations within a crystal.

A closely related continuum quantity is the net Burgers vector density (also called the closure failure vector density), denoted as $\mathbf{B}$, that is obtained by normalizing $\mathbf{b}_\text{net}$ with the scalar area $A$ encompassed within the circuit $\Gamma$ in the reference configuration of Fig. 2.1c. Upon normalizing with respect to $A$, the dislocation content is also expressed in terms of densities with units of inverse length squared. The density of redundant dislocations is often referred to as the Statistically Stored Dislocation (SSD) density and denoted $\rho_{\text{ssd}}$. The density of non-redundant dislocations is often referred to as the Geometrically Necessary Dislocation (GND) density and denoted $\rho_{\text{gnd}}$. Thus $\mathbf{B}$ serves as a linkage between a discrete description and continuum description of the dislocation content of a crystal. From a continuum perspective $\mathbf{B}$ can be calculated
by

$$B = \alpha \cdot \ell \tag{2.1}$$

where $\alpha$ is the second order Nye dislocation density tensor first derived in the 1950s [44, 45, 23, 46, 47, 48], $\ell$ is normal vector to the plane including $\Gamma$. The Nye tensor quantifies a concept known as incompatibility of deformation and can be expressed either in terms of the elastic deformation or in terms of the plastic deformation that occurs in an elastic-plastic crystal. In that sense, $B$ serves as a linkage between plastic and elastic deformation mechanisms that operate during deformation.

2.2.2. Incompatibility and Nye Tensor. A material that remains intact as it deforms is said to undergo a compatible deformation, so that a closed circuit in a reference configuration of a material remains a closed circuit while undergoing simultaneous elastic and plastic deformation. The necessary and sufficient requirement of compatibility can be expressed mathematically as $\oint \mathbf{q} \cdot d\mathbf{u} = 0$, all circuits that can shrink to a point, where $\mathbf{u} = \mathbf{u}(\mathbf{x})$ is the displacement vector field of the deformed configuration relative to the reference configuration, $\mathbf{x}$ denotes the position vector of material points, and $\Gamma$ is a closed circuit within the singly-connected domain. Infinitesimal deformation gradients are assumed so that no distinction need be made between the reference and deformed configurations when taking derivatives of field quantities. The requirement can be rewritten $\oint \mathbf{q} \cdot (d\mathbf{u}/d\mathbf{x}) = \oint \mathbf{q} \cdot (d\mathbf{u}/d\mathbf{x}) d\xi = \oint \mathbf{q} \cdot \mathbf{U} \cdot d\xi = 0$, where $\xi$ indicates distance along $\Gamma$ from $\mathcal{S}$ to $\mathcal{F}$, and $\mathbf{q}$ is a unit vector along the circuit, $\Gamma$, in the positive $\xi$ direction such that $\mathbf{q} \cdot \ell = 0$, and, and $\mathbf{U} = d\mathbf{u}/d\mathbf{x}$ is the second order displacement gradient tensor. Finally, we adopt an additive decomposition of the displacement gradient tensor such that $\mathbf{U} = \mathbf{U}^e + \mathbf{U}^p$, where $\mathbf{U}^e$ and $\mathbf{U}^p$ are the elastic distortion and the plastic distortion tensors, respectively, that are induced during deformation beyond the elastic limit. Thus there results $\oint \mathbf{q} \cdot (\mathbf{U}^e + \mathbf{U}^p) \cdot d\xi = 0$, where $\mathbf{U}^e$ and $\mathbf{U}^p$ are the elastic distortion and the plastic distortion tensors, respectively, that are induced during deformation beyond the elastic limit. Thus there results $\oint \mathbf{q} \cdot (\mathbf{U}^e + \mathbf{U}^p) \cdot d\xi = 0$, where $\mathbf{U}^e$ and $\mathbf{U}^p$ are the elastic distortion and the plastic distortion tensors, respectively, that are induced during deformation beyond the elastic limit. Thus there results $\oint \mathbf{q} \cdot (\mathbf{U}^e + \mathbf{U}^p) \cdot d\xi = 0$, where $\mathbf{U}^e$ and $\mathbf{U}^p$ are the elastic distortion and the plastic distortion tensors, respectively, that are induced during deformation beyond the elastic limit. Thus there results $\oint \mathbf{q} \cdot (\mathbf{U}^e + \mathbf{U}^p) \cdot d\xi = 0$. 


so it is evident that $U^e$ and $U^p$ must act in concert in order to maintain compatibility. In general, $\oint q \cdot U^p ds = -\oint q \cdot U^e ds \neq 0$, so taken individually, the elastic distortion and the plastic distortion are incompatible. Because the Burgers circuit constructions in Fig. 2.1 accounts only for plastic distortion of the material, the continuum definition of the net Burgers vector is

$$b_{net}^p = \oint q \cdot U^p ds.$$ (2.2)

Likewise, we can define the net Burgers vector due to elastic distortion as $b_{net}^e = \oint q \cdot U^e ds$, where the superscript $e$ indicates elastic. Thus, when a material incurs a non-zero $b_{net}^p$ as a consequence of dislocation-mediated plastic distortion, an attendant elastic distortion field must satisfy $b_{net}^e = -b_{net}^p$ in order to maintain the closed circuit necessary for compatibility of the combined elastic-plastic deformation.

Fig. 2.2 demonstrates this process for the special case of a fully annealed and previously undeformed crystal that undergoes both elastic and plastic deformations with a single slip system defined by $s$ and $n$ (cf. Fig. 2.2a), but is otherwise unconstrained. The convention for positive and negative dislocations is indicated. We first imagine the material to be separated into a number of small elements of finite size (cf. Fig. 2.2b) after which a pure plastic slip on the $n$ plane in the $s$ direction is applied to the small elements such that a gradient of increasing plastic slip is induced in the $s$ direction (cf. Fig. 2.2c). The physical interpretation of the plastic slip, denoted as $\gamma$, is the change of angle measured in radians of two lines in the material that were originally perpendicular in the reference configuration. The number of dislocation symbols within each element increases schematically with the amount of plastic slip induced. The gaps in the material introduced by the gradient of plastic slip represent the incompatibility due to dislocation-mediated plastic distortion. Elastic deformation to rotate and distort the crystal lattice (cf. Fig. 2.2d) with an equal and opposite
incompatibility is necessary to close the gaps so as to achieve a final compatible deformation state (cf. Fig. 2.2e). In a real material, elastic and plastic deformations occur simultaneously to maintain compatibility.

We now consider the consequences of the elastic and plastic incompatibilities in terms of the final shape of the deformed elastic-plastic crystal as well as its dislocation content. The elastic distortion consists of both a rotation and a strain of the crystal lattice. From Fig. 2.2d, it is evident that there must be a gradient of increasing lattice rotation in the s direction in order to maintain compatibility which introduces a curvature of the crystal lattice, denoted here by the scalar $\kappa$. In addition, there will be a gradient of elastic strain. The lattice curvature and lattice strain represent the elastic incompatibility.

With regard to plastic deformation, a subset of the overall dislocation density induced during deformation contributes to the Statistically Stored Dislocation (SSD) density, denoted as $\rho_{ssd}$, and consists of pairs of oppositely-signed dislocations (cf. Fig. 2.2e), which correspond to the redundant dislocations in Fig. 2.1d. The SSD density increases with increasing plastic slip. The subset of the remaining dislocations, denoted as $\rho_{gnd}$, is called the Geometrically Necessary Dislocation (GND) density and consists of the net signed density of dislocations, and corresponds to the non-redundant dislocations in Fig. 2.1d. The GND density of positive edge dislocations increases with increasing gradient of plastic slip as, $\rho_{gnd(e)}b = -\nabla \gamma \cdot s$, and is thus related to the degree of plastic incompatibility[22, 48]. The relationship between GND density of positive screw dislocations and plastic slip gradient is $\rho_{gnd(s)}b = \nabla \gamma \cdot n$. Thus, the GND densities represent the plastic incompatibility.

The Net Burgers Vector Density, $\mathbf{B}$ is related to the GND densities. From the perspective of a discrete description of the dislocation content, $\mathbf{B}$ is obtained by normalizing $\mathbf{b}_{net}$ with the area inside the Burgers circuit in the reference configuration in Fig. 2.1. From the perspective of a continuum description of the dislocation content,
Figure 2.2. Incompatibility thought experiment: a) a confined volume b) separated into small elements c) plastic slip with a gradient is applied d) elastic deformation to rotate and distort the crystal lattice with an equal and opposite incompatibility e) final deformed configuration with GNDs and SSDs.
we apply Stokes’ Theorem to Eq. 2.2 to obtain $b^\text{net}_p = \int_A \ell \cdot (\nabla \times U^p) \, dA = -\int_A \ell \cdot (\nabla \times U^e) \, dA$ where $A$ is the scalar area contained within the circuit $\Gamma$. If the extent of $\Gamma$ is chosen such that the value of the integrand is relatively constant within the circuit, the integrand represents the net Burgers vector density, so that $B = \ell \cdot (\nabla \times U^p) = \ell \cdot \alpha^T$, where $\alpha$ is called the Nye dislocation density tensor and the superscript $T$ indicates transpose. Adopting the curl operation form [49], $\text{curl} \, U = -\left[ \nabla \times (U)^T \right]^T$, the Nye tensor is defined as $\alpha = \text{curl} \, U^p = -\text{curl} \, U^e$ so that the net Burgers vector density can be expressed as as in Eq. 2.1. Thus, the second-order Nye dislocation density tensor, $\alpha$, is a continuum manifestation of the Burgers circuit in that it takes $\ell$ as input and produces $B$ as output [23, 44, 45, 46, 47]. We now consider the kinematics of elastic and plastic deformation in order to derive explicit expressions for $\alpha$.

It is well-known from the kinematics of small-strain elastic deformation, that $U^e = \varepsilon^e + \Omega^e$, where $\varepsilon^e$ is the symmetric second-order strain tensor and $\Omega^e$ is the skew-symmetric second-order infinitesimal rotation axial tensor that describes infinitesimal lattice rotations denoted as $\omega_1, \omega_2$ and $\omega_3$ about the $x_1$, $x_2$, and $x_3$-axes respectively. It can be shown that the general expression for the Nye dislocation density tensor, $\alpha$, in terms of the elastic incompatibility is

\begin{equation}
\alpha = -\kappa^T + \text{tr}(\kappa)\mathbf{I} + \text{curl} \varepsilon^e \tag{2.3}
\end{equation}

where the second-order non-symmetric crystal lattice curvature tensor, $\kappa$, is defined as $\kappa = \text{grad} \omega$, the $\text{tr} (\cdot)$ operator indicates the trace of the argument, and $\mathbf{I}$ is the second-order unit tensor [23, 30, 44, 45, 46, 48].

The kinematics of small strain plastic deformation in a single crystal is $U^p = \sum_{m=1}^{M} \gamma^{(m)} s^{(m)} \otimes n^{(m)}$, where the superscript $(m)$ indicates the relevant slip system and $M$ is the total number of slip systems of slip systems. Upon taking the curl of $U^p$,
there results

\[
\alpha = \sum_{m=1}^{M_e} \rho_{\text{gnd}(e)}^{(m)} b^{(m)} s^{(m)} \otimes t^{(m)} + \sum_{m=1}^{M_s} \rho_{\text{gnd}(s)}^{(m)} b^{(m)} s^{(m)} \otimes s^{(m)}
\]

which leads naturally to a decomposition of the Nye tensor into a discrete basis of GND densities of edge and screw character, where the quantities \( \rho_{\text{gnd}(e)}^{(m)} \) and \( \rho_{\text{gnd}(s)}^{(m)} \) refer to edge and screw GND densities, respectively. Here \( b^{(m)} \) is the Burgers vector magnitude, \( s^{(m)} \) is the unit vector in its direction of slip direction, and \( t^{(m)} = s^{(m)} \times n^{(m)} \) is the unit tangent vector that indicates the line sense of the edge GND densities in the basis \([44, 48]\) on the \( m \)-th slip system. The number of edge systems in the discrete GND density basis is \( M_e = M \), whereas the number of screw systems, \( M_s \), in the GND density basis is equal to the number of unique slip directions, \( s^{(m)} \). A face-centered cubic (FCC) crystal with slip system family \( \{111\} \{110\} \) has \( M = 12 \) crystallographic slip systems, but has eighteen discrete basis GND densities because \( M_e = 12 \) and \( M_s = 6 \). Any GND density content on the crystallographic slip systems can be decomposed into the discrete GND densities basis. Any dislocation content that can not be decomposed into the discrete dislocation basis is part of the SSD density.

### 2.3. Problem definition

We now turn our attention to experimental methodologies. Stated simply, we measure the incompatibility of elastic deformation, and hence, the incompatibility of the plastic deformation from which we deduce information about the underlying GND density content. The lattice rotation and elastic strain terms in Eq. 2.3 are measured via spatially-resolved diffraction methods to determine the Nye tensor. Then, the unknown GND densities are characterized based upon Eq. 2.4 from the
known Nye tensor. The fundamental limitation to this approach is that upon equating Eqs. 2.3 and 2.4, there results an underdetermined linear system of a maximum of nine equations with, for the case of FCC crystals, eighteen unknown GND densities of the discrete basis.

We employ two methods to resolve the indeterminacy. One method involves a judicious choice of specimen and loading configuration. We induce a two-dimensional plane strain deformation state in the (110) plane—which is a mirror symmetry plane for each of the twelve crystallographic slip systems—in a FCC nickel single crystal. Thus there result six pairs of “mirrored” slip systems. The two slip systems in each pair have the same resolved shear stress, so it is assumed that the two slip systems are activated in equal amounts. For three of the slip system pairs, the plastic deformation that results from activation of a pair in equal amounts does not induce a plane strain deformation state in the (110) plane. Hence, by inducing small scale yielding (relative to the specimen dimensions) to maintain plane strain conditions in the plastically deforming volume, plastic slip on these three slip system pairs is suppressed so the GND densities for these three pairs of slip systems are zero to a very good approximation. The remaining three pairs of plastic slip systems admit plane strain deformation when activated in equal amounts. Each of these three pairs is considered to be an effective in-plane plastic slip system and the two slip systems within each pair have the same GND density. Further, each of the effective slip systems can be treated as edge systems because there are no plastic slip gradients in the out-of-plane direction. Each effective edge dislocation system can be decomposed into edge and screw GND densities of the discrete basis, with well-defined proportions between them. Thus the overall total of twelve potential crystallographic slip systems reduces to three effective plastic slip systems each with its own unknown edge GND density. However, the Nye tensor for this configuration contains only two components with non-zero values, so the linear system is still underdetermined with two equations and three unknowns.
This remaining indeterminacy may be removed in many cases by a detailed understanding of the mechanics of the deformation state under experimental consideration. In essence, if one or two of the effective plastic slip systems are known not to be activated at a material point throughout the deformation history based upon a knowledge of the evolution of local stress state, then the GND densities on those slip systems can be set to zero. In cases when that determination can be made, the linear system between the Nye tensor and GND densities can be solved exactly for the remaining GND densities.

The second method is used when an exact solution is not possible, so the only recourse to resolve the indeterminacy is to minimize a global measure of the GND density. While different global measures can be chosen, we minimize the total GND density \( \rho_{\text{gnd}}^{\text{tot}} = \sum_{m=1}^{M_e} \rho_{\text{gnd}(e)}^{(m)} + \sum_{m=1}^{M_s} \rho_{\text{gnd}(s)}^{(m)} \) subject to Eq. 2.4. This minimization can be performed easily with elementary numerical methods. However, for the two-dimensional plane strain deformation state with three effective in-plane slip systems considered in the present study, the lower bound solution for the total GND density has been derived analytically[1]. Further, the lower bound solution has the special property that it reduces to the exact solution for circumstances where the one or two of the effective slip systems is known to have a zero GND density.

A schematic representation of the three effective slip systems with unit slip vector \( \mathbf{S}^{(\alpha)} \) in the single crystal specimen is shown in Fig. 2.3. (N.B. Upper case letters and lower case letters are used to denote unit vectors for the effective in-plane slip systems and the crystallographic slip systems, respectively.) The directions of \( \mathbf{S}^{(\alpha)} \) relative to the local \( x'_1, x'_2 \) coordinate frame (with orthonormal basis unit vectors \( \hat{\mathbf{e}}'_1, \hat{\mathbf{e}}'_2, \hat{\mathbf{e}}'_3 \)) that rotates with the crystal lattice during deformation are listed in Table 2.1. The unit line tangent vector, \( \mathbf{T}^{(\alpha)} \), for each of the three effective in-plane dislocations is taken as \( \mathbf{T}^{(\alpha)} = \hat{\mathbf{e}}'_3 \). The unit slip plane normal, \( \mathbf{N}^{(\alpha)} \), for each slip system is defined
as $\mathbf{N}^{(\alpha)} = \mathbf{T}^{(\alpha)} \times \mathbf{S}^{(\alpha)}$. The crystallographic slip systems that comprise effective slip systems 1 and 3 are coplanar and those that comprise effective slip system 2 are collinear crystallographic slip systems. The full gamut of glissade and sessile dislocation junctions found in FCC crystals are formed during deformation when the various effective slip systems are activated. Hence, it bears emphasis that the crystal maintains all the complexity of a material under arbitrary loading conditions, despite the carefully controlled loading configuration.

Plastic deformation induced with a line load along the [110] direction induces rotation of the crystal lattice only about the out-of-plane axis parallel to [110] coincides with the $x_3$-direction, so the induced lattice rotation field is two-dimensional. The resulting Nye tensor has non-zero components of $\alpha_{13}$ and $\alpha_{23}$. When taking a Burgers circuit about $\ell$, the resulting Net Burgers Vector Density, $\mathbf{B}$, lies within the $x_1,x_2$ plane and the angle that $\mathbf{B}$ makes with the horizontal axis is indicated as $\beta$ as shown in Fig. 2.3.

In this study, we measure the spatially-resolved GND densities associated with wedge indentation into a single crystal of nickel. The experimental method relies on the equivalence of Eqs. 2.3 and 2.4, so that upon measurement of the components $\alpha_{ij}$ using Eq. 2.3, the values of $\rho_{gnd}^{(\alpha)}$ are deduced via Eq. 2.4. We choose an idealized experimental configuration that minimizes the practical problems [32, 1] which complicate the experimental determination of the nine independent components of the unsymmetric second-order $\alpha_{ij}$. As illustrated in Fig. 2.3, the shape of the nickel specimen as well as the relative orientations of its crystal lattice and the wedge indenter are chosen so as to induce crystal lattice rotation only about the [110] lattice direction, which also ensures that the gradients of field quantities are zero (to within experimental uncertainty) in the [110] lattice direction, at least away from the (110) free surfaces. There are only two non-zero components of the $\alpha_{ij}$ under those condi-
Figure 2.3. a) Schematic representation of the three effective slip system and with net Burgers vector density and $\beta$. b) Three dimensional representation of three effective slip systems.
Table 2.1. Unit slip vectors of effective plane strain slip systems in FCC crystal expressed in local coordinate frame. The crystallographic Burgers vector for each slip system is $b^{(1)} = b^{(2)} = b^{(3)} = b = a/\sqrt{2}$, where $a$ is the lattice constant of the FCC unit cell.

<table>
<thead>
<tr>
<th>Slip system: $\alpha$</th>
<th>Unit slip vector: $S^{(\alpha)}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>$S^{(1)} = \sqrt{\frac{1}{3}} \hat{e}_1' + \sqrt{\frac{2}{3}} \hat{e}_2'$</td>
</tr>
<tr>
<td>2</td>
<td>$S^{(2)} = \hat{e}_1'$</td>
</tr>
<tr>
<td>3</td>
<td>$S^{(3)} = -\sqrt{\frac{1}{3}} \hat{e}_1' + \sqrt{\frac{2}{3}} \hat{e}_2'$</td>
</tr>
</tbody>
</table>

tions, both of which can be determined unambiguously by measuring the state of the deformed crystal lattice on a single (110) cross-section of the specimen.

We make measurements of lattice rotations, Nye tensor components, $B$, $\beta$ and of either the exact GND densities or the lower bound on $\rho_{\text{GND}}^{\text{total}}$ in the region under the indenter.

Both $\rho_{\text{SSD}}$ and $\rho_{\text{GND}}$ affect the local value of the elastic limit threshold stress. However, $\rho_{\text{SSD}}$ does not contribute to the net Burgers density, $B$, or to the change in lattice orientation and has no long-range stress effect on other dislocations. On the other hand, $\rho_{\text{GND}}$ determines the local value of $B$ and has a long range effect on other dislocations through the lattice curvature and/or net dislocation stress fields. Thus, $\rho_{\text{GND}}$ has been invoked as a key variable in the behavior of ductile materials that have high plastic strain gradients, especially those that occur due to the small sizes of nanoscale and microscale specimens. In addition, the long-range stress fields due to $\rho_{\text{GND}}$ that exist in a materials constrained against lattice rotation may be a driving force for the evolution of dislocations structures into lower energy configuration via self-assembly mechanisms.

Dislocations move through a crystal lattice once the applied stress state (i.e. internal forces normalized by area) resolved onto the slip plane $\mathbf{n}$ and in slip direction $\mathbf{s}$ achieves a critical value thereby inducing plastic deformation. As dislocations on intersecting slip systems move, they interact with each other to form junctions that
may hinder further motion of the interacting dislocations. In addition, dislocations interact with other defects in a material such as second phase particles, grain boundaries, free surfaces, etc. All such interactions increase the stress threshold necessary for subsequent plastic deformation to occur, whereby the material is said to have hardened.

The crux of understanding the mechanical response of a plastically deforming material is to predict the relationship between the evolution of the threshold stress and the evolution of the resulting plastic deformation. Both quantities depend sensitively upon the configuration and distribution of dislocations throughout the material that are determined by the initial conditions as well as the prior history of deformation. Important length scales associated with dislocations range from the atomic length scale ($10^{-10}$ m) to the length scale at which dislocations form structures as a means to reduce their energies ($10^{-5}$ m). The evolution of dislocation distributions is inherently a multiscale phenomenon because the behavior of dislocations at the larger length scales depends sensitively upon the details of the smaller length scales.

The focus of this thesis is to chapter multiscale measurements of the distribution of dislocations within a material that has suffered extensive plastic deformation. Specifically, we measure the Geometrically Necessary Dislocation (GND) density, which is a continuum-based concept that describes the subset of the overall dislocation content that exerts a long-range (i.e. much greater than mean dislocation spacing) influence on other dislocations within the material. As such, a GND density introduces a length-scale dependence into the relationship between stress and deformation. The experimental results of this paper demonstrate the characteristic lengths to be a distribution, rather than a single value as is often assumed.

The lattice rotations on the indented Nickel single crystal sample are measured using recently developed HR-EBSD technique with $\sim 0.005^\circ$ angular accuracy. The details on HR-EBSD will be presented in Chapter 3. In order to provide a multi-scale
The results and discussions section:

2.4. Results and Discussions

The lattice rotations are used to calculate the two non-zero components of Nye’s tensor, \( \alpha_{13}, \alpha_{23} \), which in turn determines the components of the net Burgers vector density, \( \mathbf{B} \), as explained in the Section 2.3. The net Burger vector density, \( \mathbf{B} \), contains the slip system activity information which can visualized using “\( \beta \)-plot” which is a contour map of the angle that \( \mathbf{B} \) makes with the horizontal axis as indicated in Fig. 2.3. The active slip systems for different ranges of \( \beta \) are shown on the legend in Fig. 2.5, the regions where there is single effective slip is activated dominantly or double slip system is activated can easily be identified.

The rigorous lower bound for GND densities is calculated using the method presented in detail [1]. On the right column of Fig. 2.6, the total GND densities of the indented crystals are shown. It can be observed that dislocation structures are formed beneath the indented region. The length scales of dislocation spacing, cell-wall widths, and cell sizes vary over the domain. The GND density also varies over the domain. This motivates the question as to how these length scales are correlated to the magnitude of GND density.
Figure 2.4. Image Quality map of EBSD measurements with step sizes of a) 2500 nm b) 500 nm (area bounded with the white box in part a) is mapped), c) 100 nm (area bounded with the white box in part b) is mapped) d) 20 nm (area bounded with the white box in part b) is mapped)
Figure 2.5. $\beta$-plots with step size of a) 2500 nm b) 500 nm (area bounded with the white box in part a) is mapped), c) 100 nm (area bounded with the white box in part b) is mapped)
Figure 2.6. Lattice rotations map (left column) and corresponding Lower Bound Total GND densities (right column) with step sizes of a) 2500 nm b) 500 nm (area bounded with the white box in part a) is mapped), c) 100 nm (area bounded with the white box in part b) is mapped)
Figure 2.7. GND density distribution on slip system 1 with step sizes of a) 2500 nm b) 500 nm (area bounded with the white box in part a) is mapped), c) 100 nm (area bounded with the white box in part b) is mapped)
Figure 2.8. GND density distribution on slip system 2 with step sizes of a) 2500 nm b) 500 nm (area bounded with the white box in part a) is mapped), c) 100 nm (area bounded with the white box in part b) is mapped)
Figure 2.9. GND density distribution on slip system 3 with step sizes of a) 2500 nm b) 500 nm (area bounded with the white box in part a) is mapped), c) 100 nm (area bounded with the white box in part b) is mapped)
There are three effective slip systems and the distribution of GND densities and their associated length scales are expected to vary for each slip system. The data processing method resolves the dislocation densities on each crystallographic slip plane and at some locations the measured dislocation densities are exact where there is dominantly single slip or double slip. Dislocation densities on each slip system over different length scales are progressively shown in Fig. 2.7 to Fig. 2.9. The most apparent features in these plots are the quasi-periodic dislocation cell walls which are recognizable at all different step sizes, and the dislocation cells which are resolved more with decreasing the step size. The crystallographic slip traces which show the deformed slip direction are indicated in each plot. In most of the dislocation walls, the crystallographic slip traces are perpendicular to the orientation of the cell walls in the plane, which indicates that effective systems is dominating in that region. When compared to β-plots, slip system activities can be easily identified.

Another way of identifying the active slip systems over the domain, is plotting the net Burgers vector densities and superimposing them on the crystallographic slip traces. If the crystallographic traces of an effective slip system are aligned with the net Burgers vector densities, it indicates that in those regions that effective slip system is active. Some illustrative examples for this type of slip are shown in Fig. 2.10; the areas where the deformed crystallographic traces of slip system 1 are aligned with the net Burger Vector Densities are shown, in Fig. 2.10a. Similar observations are also made for slip system 2 and 3 and they are presented in Fig. 2.10b and 2.10c respectively. A smooth spatial variation of one effective slip system for this type of deformation with large strain gradient is predicted by standard crystal plasticity theory. However the experimental results strongly indicate this to not be the case. Secondary slip systems are activated which constitutes the scattered independent regions in β-plots. This secondary slip activation likely is due to the activation of other slip systems due to the back stresses, caused by the presence of the GND densities. Hence, it is more
plausible to label these regions as dominantly single slip regions rather than single slip regions.

The quasi-periodic dislocation cell walls are apparent in GND density maps in Fig. 2.7 to Fig. 2.9. However, there is no straightforward method to quantify the dislocation spacing between cell walls or a distribution for dislocation cell sizes. Several researchers suggested relationship between cell sizes and dislocation spacing using TEM micrographs [33, 2, 35, 36, 50]. The cell size and dislocations densities are areal quantities and not proper for a length scale analysis. In order to determine the characteristic length scale, Holt [36] described the areal cell size to a one dimensional measure $d$, and the dislocation density to the mean dislocation spacing which is defined as the inverse of the square root of the dislocation density $\rho^{-1/2}$. Based on experimental observations, Holt [36] introduced a relationship between the cell size dimension with the dislocation spacing: $d = K\rho^{-1/2}$, in which cell sizes, $d$, changes linearly with dislocation spacing, $\rho^{-1/2}$. In a later work by Staker and Holt [35], the cell size versus dislocation spacing relationship for copper polycrystals deformed in a tensile test was quantified. They used TEM micrographs and computed the overall densities with an observation based counting method. They showed that the cell sizes changes linearly with the dislocation density spacing with a slope of, $K = 16$. Different type of metals were studied under different deformation conditions using similar TEM micrograph methods and the cell sizes and dislocation spacing are fitted into the Holt’s equation. In a recent study, Koneva et al. [2] reviews these studies and lists $K$ values that ranges from 10 to 19 for different FCC metals deformed in room temperature. Thus the TEM micrograph method is definitely shedding light to explore the complexity in plasticity and extracting a relationship between cell size and the dislocation densities, however, it is limited to the constraints of TEM: sample preparation is tedious and material characteristic and microstructure may change during sample preparation; sample size limitation makes it hard to do a multiscale
Figure 2.10. GND density distribution on slip system a) 1 b) 2 and c) 3 with net Burger vector densities.
Table 2.2. Values of coefficient K for FCC metals and solid solutions (composition is given in at %) (from Koneva et al. [2])

<table>
<thead>
<tr>
<th>Type of material</th>
<th>Material</th>
<th>Single crystal orientation or polycrystal grain size (µm)</th>
<th>Deformation temperature T (K)</th>
<th>Value of coefficient K</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure metals</td>
<td>Al</td>
<td>–</td>
<td>293</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>Ni</td>
<td>–</td>
<td>293</td>
<td>10–15.3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>–</td>
<td>293</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td>Cu</td>
<td>[0 0 1]</td>
<td>77, 293, 673</td>
<td>16, 19, 24</td>
</tr>
<tr>
<td></td>
<td></td>
<td>54</td>
<td>293–973</td>
<td>16</td>
</tr>
<tr>
<td></td>
<td></td>
<td>–</td>
<td>293</td>
<td>9.5</td>
</tr>
<tr>
<td>Solid solutions with SRO, austenitic steels</td>
<td>Ni$_3$Fe</td>
<td>40</td>
<td>293</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>120</td>
<td>293</td>
<td>5</td>
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<td>450</td>
<td>293</td>
<td>7</td>
</tr>
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<td></td>
<td></td>
<td>[0 0 1]</td>
<td>293</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td>Cu–Al (Al: 0.5–15%)</td>
<td>60</td>
<td>293</td>
<td>2–5</td>
</tr>
<tr>
<td></td>
<td>Cu–Mn (Mn: 0.4–18%)</td>
<td>60</td>
<td>293</td>
<td>2–5</td>
</tr>
<tr>
<td></td>
<td>Fe–12.5%Mn–4.5%C</td>
<td>20</td>
<td>293</td>
<td>5–7</td>
</tr>
<tr>
<td></td>
<td>Steel 304</td>
<td>–</td>
<td>293</td>
<td>3.5–6.2</td>
</tr>
<tr>
<td>Ordered solid solution</td>
<td>Ni$_3$Fe</td>
<td>40</td>
<td>293</td>
<td>16</td>
</tr>
<tr>
<td></td>
<td></td>
<td>120</td>
<td>293</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td></td>
<td>450</td>
<td>293</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[0 0 1]</td>
<td>293</td>
<td>19</td>
</tr>
</tbody>
</table>

Thus, in pure FCC metals and Ni$_3$Fe alloys with short-range atomic order the values of the cell substructure parameters $D$ and $H$ decrease with deformation in such a way that the $D/H$ ratio remains unchanged (Fig. 4). This shows the validity of the relationship (3) during the evolution of the cell substructure.

For the Ni$_3$Fe alloy with LRO and solid solutions of Cu–Al and Cu–Mn, the relationship (2) is not satisfied.

3.5. Relationship between dislocation density and cell wall width

For the Ni$_3$Fe alloy, the relationship (3) is valid only in the case of SRO. It was established that the value of the coefficient $C$ in this relationship decreases with increasing deformation temperature (Table 3). Its value is also higher for single crystals than analysis; dislocation density measurements from TEM micrographs is a relatively subjective method which is based on a counting method, can not resolve regions where there is high dislocation densities [51]; and, GNDs can not be distinguished from SSD.

In the present study, a more objective method is developed using HR-EBSD measurements which requires relatively simpler sample preparation and eliminates the all listed constraints of TEM measurements. The method starts with careful assessment of $\beta$-plots in Fig. 2.5 and net Burgers vector density plots in Fig. 2.10, to identify active slip systems on various domains. In these identified regions, the local slip directions are perpendicular to cell walls. Since the quasi periodic cell wall arrangements are dependent on the slip direction, a quantitative analysis for GND density should be also done along slip directions. Within the regions with quasi-periodic cell wall patterns, the GND density on each slip system is extracted along the corresponding crystallographic slip directions. The extracted data is interpolated such that the data
sampling distance is same as the step size of the corresponding HR-EBSD map. The noise is filtered from the noise using a bandpass filter and the fast Fourier transform (FFT) of the extracted data is taken. The FFT analysis yields the peak GND density versus the spatial period which can be considered as the double of the mean distance between two consecutive cell walls, and also can be interpreted as cell size. Thus for each crystallographic trace we get the peak density and the cell size. This procedure is repeated for different locations and for each crystallographic traces we where there is deformation on a single slip system dominating and also for double slip system. In Fig. 2.11 and Fig. 2.12, the distribution for the cell size and the dislocation densities for four different measurements are shown.

Here using an SEM based method, similar results are obtained as shown in Fig. 2.12-2.13. The cell sizes, \( d \) are extracted along crystallographic traces increasing linearly with the dislocation spacing \( \rho_{GND}^{-1/2} \). The slope of the curve, \( K \), is different for different slip system activation. The \( K \) value is higher for dominantly single slip regions, than the double slip region. \( K \) value also differs for different slip systems. On the regions where deformations is due to dominantly through activation of effective slip system 1 and 3 (since they are identical and symmetric, they are treated as one slip system), \( K = 23.9 \), whereas in regions slip system 2 dominating, \( K = 29.5 \). In the regions where multiple system are active, \( K \) varies between 13 – 16, which agrees within the range of \( K \) value found for Ni samples in [50] using TEM micrograph method.

Additional information can be extracted from these plots is the distribution of the length scales. The data points lie under and above the solid line almost symmetrically. At lower dislocation densities —in other words, for higher dislocation spacing, data points are more dispersed around the line, as the dislocation densities increase, there is more space for dislocations to move, the cell sizes varies. This shows multiscale characteristic of the plastic deformation.
Figure 2.11.  a) Dislocation cell size vs GND densities in double slip regions of System 1 and 3 for different step sizes b) Dislocation cell size versus dislocation spacing for different step sizes.

Figure 2.12.  a) Dislocation cell size vs GND densities in double slip regions of System 2 for different step sizes b) Dislocation cell size versus dislocation spacing for different step sizes.
Figure 2.13.  a) Dislocation cell size vs GND densities in slip System 1 and 3  b) Dislocation cell size versus dislocation spacing.

Figure 2.14.  a) Dislocation cell size vs GND densities in slip System 2 with 2500 nm results  b) Dislocation cell size versus dislocation spacing
As the dislocation density increases, there is less space to move, dislocation walls collapse in each other and form smaller dislocation cells with narrower cell wall widths. Recent experimental work by Koneva et al. [2] shows a linear relationship between the cells size and cell size widths. In the present work, a quantitative assessment of for the cell-wall width and cell sizes has not been done, however succinct qualitative analysis is enough to show the relation. Generally, in the regions where the dislocation densities are lower, the cell wall widths are broader and more diffused than the regions with higher dislocation densities. This can be observed in 100 nm measurements. In upper right part of Fig. 2.10a, the dislocation walls are more diffuse and the GND density is in the order of $10^{14} \text{m}^{-2}$, whereas in the middle part of the same map, dislocation cell-wall widths get narrower and the dislocation density increases up to $10^{15} \text{m}^{-2}$. The cell-wall width is inversely related to the GND density. HR-EBSD being less restricted with size of the area interests on the sample is advantageous over TEM, since cell sizes vary between $1 - 20 \mu m$ which is a big range for an assessment in TEM.

2.5. Conclusions and Recommendations

In this study, we developed a new technique to investigate the multi-scale characteristics of the plastic deformation. The approach is unique in the sense that it exploits a continuum based concept, GND density, to investigate a discrete problem. The results from the high resolution GND density measurements showed evolution of material length scales over the microscopic to macroscopic length scales. The distribution of length scales extracted from HR-EBSD measurements corroborated with earlier findings from TEM-based methods. The length scales, dislocation spacing and the dislocation cell sizes are found to be linearly varying and they follow the Holt’s relation as found with TEM based methods. HR-EBSD, being less restricted with the size of the area of interest on the sample, is advantageous over TEM for these
measurements. High dislocation densities and wide cell walls in the order of few micrometers which are not accessible with earlier TEM measurements can be easily extracted from HR-EBSD method.

We used the the lower bound GND density solution and coupled it with a new quantity, $\beta$, to find the dominantly single slip regions where the lower bound solution becomes exact. The material length scales are extracted for each individual slip system. Although the distribution of length scales for each slip system are fitted to Holt’s relation, the slope of the linear Holt’s relation, $K$, differs for each slip system.

The measurements not only provide a quantitative distribution but also helped us to understand the evolution mechanism of the dislocations. Dislocation motions follows sort of a self-organization mechanism to seek a low energy state. In the regions with low dislocation densities, dislocations accumulate and form wide cell walls. As the dislocation density increases, dislocations re-arranges and collapse to form narrower cell walls.

Further analysis of dislocation walls can be done using the same data set. The relationship between the dislocation densities and the dislocation cell walls can be extracted, this will be a valuable input to material models incorporating GND density evolution mechanisms. More HR-EBSD data can be taken with a smaller step size in regions where there is dominantly single effective slip system is active.

HRTEM analysis should be employed to explore the discrete dislocations in the cell walls of sizes ranging 20 nm to 1 $\mu$m to have a comprehensive understanding of evolution mechanism in nano-scale.
CHAPTER 3

Experimental Measurements of GND Densities

3.1. Abstract

In the previous chapter, the spatial distribution of the lower bound of geometrically necessary of dislocation (GND) densities over different length scales, measured using HR-EBSD, are presented. The analysis presented in the previous chapter is based on several assumptions such as plane deformation conditions under special crystallographic configurations and negligible elastic strain. In this chapter results from another set of measurements are analyzed to investigate validity of these assumptions and also to assess the effect of experimental variables in the results. The details of the new HR-EBSD microscopy method is introduced, and the uncertainty in dislocation density measurements is calculated. The measurements are performed in the same Ni single crystal that is deformed in plane deformation conditions, in which the Nye’s dislocation density tensor has only two non-zero components. The lattice rotation measurements were performed with two different spatial resolutions, 500 \( nm \), and 50 \( nm \), over different fields of view. The analysis shows dependency of GND densities on spatial resolution. The effect of the elastic strain is also investigated both experimentally and numerically and it is demonstrated that the effect of elastic strains on GND density measurements is negligible for the conditions of these experiments. The plane deformation state, assumption is validated by 3D crystal plasticity numerical model.
3.2. Introduction

The size dependency of plastic deformation in material has been demonstrated by several researchers more than a decade ago [20, 52, 53]. Indentation tests are the most trivial examples to illustrate the size effect in small scale, plastically deforming volumes. The indentation tests showed that hardness of the material increases with decreasing the penetration depth to below 10 µm [32]. Following the experimental observations, to investigate further the size effect in material, so-called strain gradient based plastic theories have been developed by several researchers [54, 55, 56, 57]. In these theories either the gradient of plastic strain or GND densities [5, 16, 25, 37, 58] are incorporated into the classical crystal plasticity framework. GND densities are incorporated to these models due to their length scale characteristics (See Section 2.2.2). In addition to their intrinsic length scale characteristics, the evolution process of GND densities yields new intrinsic length scales such as dislocation cell wall widths, dislocation cell sizes.

Quantifying the dislocation densities and resolving the associated length scales would help improvement of numerical models, and it would also provide better understanding of the complexity of plastic deformation. However there is no any trivial method to characterize dislocation densities. Eq. 2.4 shows the relationship between the Nye’s tensor and the GND densities. Nine components of the Nye’s tensor, in the left hand side of the equation, can be found by lattice curvature measurements through spatially resolved diffraction methods. For a face-centered-cubic (FCC) crystal there are 12 slip planes, and 18 different types of GNDs can evolve during deformation, so on the right side of the Eq. 2.4 there will be 18 unknowns GND densities.

There are several studies where GND densities are quantified using minimization techniques. Sun et al. [30] measured lattice rotations on the surface of deformed metal crystals and presented a minimization technique to approximate GND densities for the
first time. Similar methods have been used by Mughrabi and Obs [31] and Montagnat et al. [59]. These studies are based on surface characterization techniques; therefore out-of-plane components of the elastic portion of the deformation gradient tensor are not generally accessible. In later studies, three different approaches were used to eliminate this problem. The first one is based on X-ray micro-diffraction and used by Larson et al. [60], Jakobsen et al. [61], and Ohashi et al. [62], in which the full lattice rotation field with micrometer-scale spatial resolution is measured beneath the surface of very small volumes of the material. X-ray micro diffraction methods have relatively favorable sample preparation procedures, but they are limited to micrometer spatial resolution due to the beam size that is typically in sub-micron in diameter. Another approach has been to employ serial sectioning using focused-ion-beam (FIB) in order to characterize the out-of-plane aspects of the lattice rotation field which is measured using traditional Orientation Imaging Microscopy (OIM)—also referred to as Electron Backscatter Diffraction (EBSD) [15], however FIB induces deformation on surface and also implantation of Ga ions which are not ideal for GND characterization. Compared to the X-ray technique, OIM resolves more dislocations as the probe volume of X-ray is much larger [63]. A third approach is to provide a two-dimensional deformation state by using a special crystallographic orientation proposed by Rice [64], so that all out-of-plane deformation gradients are zero. This method has been introduced by Kysar and Briant [65] in context of cracks in bicrystals and pursued with wedge indentation in single crystals by Kysar et al. [1, 32].

The method proposed by Kysar et al. [32] overcomes the limitation of the out-of-plane ambiguity of the Nye tensor. The experiments consist of plastically deforming single crystals with a wedge indenter as shown in Fig. 2.3. Three effective slip systems are activated as the indenter penetrates into the material. To a very good approximation, only in-plane lattice rotation occurs as a result of the plastic deformation. As a consequence, there are only two non-zero components of the Nye’s dislocation
density tensor, which are obtained by taking the spatial gradient of the in-plane lattice rotations that are measured with traditional EBSD technique. However, it is still not possible to unambiguously determine the associated GND density because there are three unknown GND densities and only two non-zero components of the Nye tensor, so instead a lower bound on the total GND density is determined. Previous studies [48, 32] suggested two different minimization schemes. The first one is $L^2$, minimization, is geometrically motivated and minimizes the sum of the squares of the individual dislocation densities. It is defined as

\[
L^2 = \sqrt{\sum_{n=1}^{N} \left[ \rho^{(n)}_{\text{gnd}} w^{(n)} \right]^2}
\]

where the $w^{(n)}$ is a weight factor for $\rho^{(n)}_{\text{gnd}}$ and $N$ is the number of active slip system and is equal to 3 in this case. The discretization of the dislocation space only allows certain dislocations on slip plane to exist, and the $L^2$ minimization takes this into account by being able to combine dislocation line lengths into a single dislocation line length which may not exist within the original discretization.

The other technique, $L^1$ minimization, is energetically motivated. By considering the dislocation density as a line length per unit volume, the total dislocation line energy can be minimized by finding the dislocation configuration with the smallest total line length, and can be interpreted as

\[
L^1 = \sum_{n=1}^{N} \left| \rho^{(n)}_{\text{gnd}} w^{(n)} \right|.
\]

Details can be found in references [1, 32, 48]. In this paper the $L^1$ minimization technique is used to determine the lower bound on the total GND densities as presented in Section 2.3.
The key feature of each experiment is that the orientation of the deformed crystal lattice must be measured with sufficient spatial resolution so that the lattice curvature can be determined accurately. In the work presented by Kysar et al. [32, 1], the measurements are made using traditional EBSD with 2\( \mu \)m spatial resolution. The results showed the lattice rotation field which complies with the crystal plasticity models and evolution of dislocation structures such as cell walls. However, the accuracy of the measurements of the orientation of the crystal lattice of traditional EBSD does not allow to work with higher spatial resolutions and to quantify the GND densities accurately.

In this work, we pursued the study done by Kysar et al. [1] and we performed lattice measurements using the recently developed HR-EBSD method. Whereas traditional OIM technique has an angular accuracy of \( \pm 0.5^o \), HR-EBSD has angular accuracy of \( \pm 0.005^o \) in lattice rotations and elastic strain resolution with \( \sim 7 \times 10^{-4} \). This allows us to perform measurements with higher spatial resolutions, so that we can resolve more information about dislocation structures and the length scales. The elastic strain measurements — possible with HR-EBSD — allow us to study the effect of the curl\( \epsilon_e \) component in Eq. 2.3 on overall GND densities.

In Chapter 2, we presented the GND density results and proceeded with length scale results of HR-EBSD, here we aim to analyze the effect of spatial resolution using a different experimental data set obtained from HR-EBSD measurements with 500 nm and 50 nm spatial resolutions. We determine the experimental uncertainty due to angular accuracy for both traditional EBSD and HR-EBSD. Higher spatial resolutions of 50 nm measurements enable us to make a thorough analysis of the net Burger Vector Density. In this chapter we also elaborate the plane deformation assumption by means of 3D crystal plasticity model.

This chapter is organized as follows. In Section 1, the sample preparation procedure and experimental details are described. In the Section 2, we provide background
information on HR-EBSD method and present a comparison with the traditional EBSD method with a detailed uncertainty analysis. In Section 3, we show the lower bound GND densities from both 500 nm and 50 nm spatial resolution measurements, and also include a detailed analysis of the net Burgers vector density. In Section 4, we propose a 3D numerical model that elaborates the in-plane deformation assumption.

3.3. High Resolution Electron backscatter Diffraction (HR-EBSD)

Orientation Imaging Microscopy (OIM) or traditional electron backscatter diffraction (EBSD) microscopy began to impact the study of polycrystalline materials almost two decades ago. EBSD is conducted in a scanning electron microscope (SEM) chamber, using a CCD camera and a detector with a phosphor screen. An electron beam is focused on a sample that is tilted with a certain angle, typically 70°, and beam penetrates into material about 20 nm and it is rastered horizontally and vertically to map the crystallographic orientation of the surface. The distance between two consecutive point that electron beam hits is referred to the spatial step size or the spatial resolution of the measurement. At each measurement point, the beam is diffracted by the specimen’s crystallographic planes. The scattered electrons form Kikuchi patterns that are collected on the phosphor screen. In traditional EBSD techniques, Kikuchi patterns are transformed into Hough space and are indexed based upon the position features in the diffraction pattern and then compared with the reference values of the crystal. Traditional EBSD indexation software outputs the Euler angles associated with the crystallographic orientation of the mapped region on the sample with $\sim 0.5^\circ$ accuracy.

Recent advances in EBSD-based microscopy, developed over the past decade—and particularly over the past five years—have led to fundamental extensions of EBSD-based microscopy, called High-Resolution EBSD (HR-EBSD). The main strength of the HR-EBSD method is that it uses not only the position of the Kikuchi bands
as in traditional EBSD, but also the variation of the intensities within the diffraction patterns to determine orientation. Based upon the pioneering work of Troost et al. [66] and Wilkinson et al. [67], HR-EBSD exploits cross-correlations between pairs of EBSD images in order to resolve the elastic distortions that differentiate them. The resolved elastic distortions are used to obtain the Euler angles and the elastic strains. HR-EBSD outputs Euler angles with $\sim 0.005^\circ$ accuracy, which is two order of magnitude higher than it is in conventional EBSD-based microscopy. Increasing angular accuracy is important as further numerical differentiation is required for lattice curvature calculations.

The basic idea is founded in the cross-correlation of selected regions of interest (ROI) within a pair of EBSD patterns. These ROIs span subsets of the pixilated EBSD image, typically containing $\sim 1\%$ of the full image. The reference pattern is compared with a pattern of interest, and the tiny shifts between the ROIs are measured. The resulting cross-correlated image provides peak intensities and their distance from the pattern center (PC) by a vector $q$ as shown in Fig. 3.1. The vector $q$ is a measure that shows how a feature in the selected ROI shifts when compared with the other patterns containing the same feature. The geometric relationship between the unit cell and the shift in an EBSD pattern was proposed by Wilkinson et al. [67]
and extended by Kacher et al. [68]. Using the proposed geometric relation for each ROI two equations can be established as follows:

\[
(3.7) \quad r_2 r_3 \left[ \frac{\partial u_2}{\partial x_2} - \frac{\partial u_1}{\partial x_3} \right] + r_1 r_3 \frac{\partial u_2}{\partial x_1} + r_3 \frac{\partial u_2}{\partial x_3} - r_1 r_2 \frac{\partial u_3}{\partial x_1} - r_2 \frac{\partial u_3}{\partial x_2} = r_3 q_2 - r_2 q_3
\]

\[
(3.8) \quad r_1 r_3 \left[ \frac{\partial u_1}{\partial x_1} - \frac{\partial u_3}{\partial x_3} \right] + r_2 r_3 \frac{\partial u_1}{\partial x_2} + r_3 \frac{\partial u_1}{\partial x_3} - r_1 r_2 \frac{\partial u_3}{\partial x_1} - r_2 \frac{\partial u_3}{\partial x_2} = r_3 q_1 - r_1 q_3
\]

where \( \frac{\partial u_i}{\partial x_i} \) are components of the displacement gradient tensor, which are unknown, \( r_i \) denotes the known crystallographic \( <uvw> \) directions of the unit cell within the ROIs. As the vector \( q_i \) can be obtained through cross-correlations, in order to determine the 8 unknown components in Eqs. (3.7) and (3.8), at least four ROIs must be used. In practice, about 20 ROIs spread across the complete EBSD image are evaluated and a least-squares best-fit approach is used in recovering the elastic distortion tensor.

As the vector \( q_i \) is defined from the pattern center (PC), determination of the pattern center is crucial. The PC is the point lying in the plane defined by the phosphor screen, upon which the EBSD image is formed, containing a line perpendicular to the plane of the phosphor screen that also hits the electron interaction volume of the specimen (which is typically \( \sim 20 \text{ nm} \)). The position of the PC on the phosphor must be known to the same precision that is expected of the recovered elastic distortion tensor. Thus, if for example it is desired to know the to a resolution of \( 10^{-4} \), then the placement of the PC must be known to a fraction of \( 10^{-4} \) of the width of the phosphor screen. If a typical phosphor screen used for HR-EBSD is coupled to a charge coupled device (CCD) camera comprising \( 10^6 \) pixels, the PC must be known to \( 1/10 \) pixel to insure a resolution of \( \sim 10^{-4} \) in the components of the distortion
tensor. Precision determination of the PC is thus critical to HR-EBSD for absolute determinations of the elastic distortion. In this paper, recent work by Basinger et al. [69] which a software-based approach to determine PC which has shown to have a sensitivity of $\sim 2 \times 10^{-4}$ in absolute elastic distortion.

3.4. Experimental Methods

3.4.1. Sample Preparation. The sample is cut from a nickel single crystal rod with 99.998% purity purchased from Materials Preparation Center at Ames Laboratory, which was grown by Czochralski method in random orientation. Laue X-Ray diffraction was used to orient the sample in the desired orientation to within one degree accuracy. The rod was cut into rectangular prisms $20 \times 5 \times 5$ mm cross section, using a wire electrical discharge machine (EDM) such that out of plane normal of the larger surface is (110). The specimen was mechanically polished using the skim cut (fine cutting) option in wire EDM, followed by ultrasonic cleaning to remove any adhered particles. The wedge indenter, made of tungsten carbide with an angle of 90°, penetrated into the material in the [001] crystallographic direction.

The indenter surface was polished and the lubricants were used in order to minimize the friction between the wall and the indenter. The plane deformation conditions are not satisfied near the surface, where there will be mass flux in the out-of-plane direction as the dislocation lines are piercing the plane. After the indentation, the specimen was sectioned on a mid-surface (110) plane where the out-of-plane deformation was negligible. In the next sections, this assumption is elaborated with a three-dimensional crystal plasticity model, in which indentation process is simulated. The minimum distance from the surface, where the plane strain deformation is satisfied is calculated in terms of penetration depth of the indenter. The simulations also show that the elastic stress state does not change significantly after the specimen is sectioned. The indented specimen was cut by wire EDM and the newly exposed surface
to the wire was polished prior to HR-EBSD. In previous studies [43], electropolishing technique was used, however electropolishing process is not repeatable and pitting is a major problem even at low currents. In this study, chemical-mechanical polishing is used, it is started with 9 µm diamond paste continued with 3 µm and 1 µm consecutively. Polishing is completed with a vibratory polisher with 0.05 µm colloidal silica particles. The undeformed part of the sample is scanned to check whether any deformation is induced due to mechanical polishing.

Two different regions on the polished sample are mapped with two different spatial resolutions: 500 nm, and 50 nm. The EBSD measurements were taken in Phillips XL30 S-FEG microscope equipped with a phosphor screen detector and CCD camera. TSL/EDAX OIM DC 4.6 software, is used for data acquisition. The Kikuchi patterns are collected and stored at the resolution of 1000 × 1000 pixels in the Tiff format for cross-correlation method explained in previous section and can be found in [68]. The patterns are processed offline using the program developed by Kacher et al. [68]. This program provided the Euler angles and elastic strains which are further processed to calculate the lattice rotations and the lower bound GNDs.

3.4.2. High Resolution EBSD Results. The sample was initially scanned with HKL Channel 5 EBSD system, which is a traditional EBSD system with angular resolution of ±0.5°, the results were presented shown in [1]. The measurements were taken with 2 µm spatial resolution. In Fig. 3.2a, the in-plane lattice rotation results from traditional EBSD measurements are shown. The lattice rotation field is antisymmetric about the vertical line through the indenter tip. A jump of lattice rotation about the antisymmetry line (we refer it as antisymmetry line as the lattice rotations are in the opposite sense) can be easily observed, across which there is a ±20° – 25° change in lattice rotations. In Fig. 3.2b the lower bound on total GND
Figure 3.2. The lattice rotations with $2\mu m$ spatial resolution measured with HKL Channel 5. Kysar et al.[1]

densities are shown. The measurements showed patterning in dislocation cell walls, however $2\mu m$ spatial is not sufficient to resolve the details of the cell structures.

HR-EBSD measurements are done with two different spatial resolutions to resolve more details on the same sample. First measurements are taken with $500\,nm$ spatial resolutions over an area of $100 \times 100\,\mu m$ square beneath the indented region as indicated in Fig. 3.2a. The lattice rotation maps are shown in Fig. 3.3a; it is observed that they are in the order of $\pm 20^\circ$ opposite sense on each side of the line of antisymmetry, which agrees with the traditional EBSD results. Some of the complexity of the jump of the lattice rotation becomes more apparent with this spatial resolution. The deformation at both sides of the antisymmetry plane, seems to be layered with an observable length scale. The in-plane lattice rotation is denoted as $\omega_3$ and the rotations about in-plane axes, $\omega_1$ and $\omega_2$ are zero or more precisely, negligibly small by experimental design. The in-plane lattice rotations, $\omega_3$, are used to calculate the lattice curvature to determine the two components Nye’s dislocation density tensor in Eq. 2.3. To calculate the lattice curvatures and subsequently the Nye’s tensor, lattice rotations are numerically differentiated with three point differentiation at the boundaries and with the central difference method elsewhere. The differentiation is performed in the global coordinate system as shown in Fig. 2.3, so two non-zero com-
ponents of Nye’s tensor that is obtained using Eq. 2.3 is also in global coordinate system. Nye’s tensor is transformed to local coordinate system that coincides with the crystallographic coordinate system. The GND densities on each slip system is calculated using $L^1$ minimization method explained in Section 2.3.

The lower bound on total dislocation densities, are shown for both spatial resolution in Fig. 3.3b. The 500 nm spatial resolution image shows the GND densities localized at the lattice rotation jump region. The total dislocation densities are on the order of $10^{15} - 10^{16} m^{-2}$. The GND densities of the 50 nm spatial resolution measurements show significantly more detail than that of the 500 nm spatial resolution. A similar analysis will be performed after 20 nm spatial resolution measurements.

The apportionment of the lower bound of the total GND density onto the individual slip systems is shown in Figs. 3.4-3.6. The crystallographic slip traces of each slip system, which indicate the as-deformed slip directions, are overlaid on the GND density maps. The GND density on slip system 2 is distributed symmetrically across the lattice jump region (cf. Fig. 3.5), whereas the GND density on slip system 1 is more pronounced on the right and the GND density from slip slip system 3 is more pronounced on the left. The GND density on slip system 1 accumulates and forms dislocation walls that make an angle of $50^\circ$ to $55^\circ$ from the lattice jump region where lattice rotations diminishes. The crystallographic traces are almost perpendicular to dislocation walls which indicates that the slip is caused primarily by kink-like shear that gives rise to dislocation walls that region. Finally, the slip system 3 is activated as almost the mirror image of the slip system 1, and analogous GND densities accumulate in a similar manner.

Another physical interpretation of the Nye’s tensor is through the net Burgers vector density as explained in Section 2.3. The net Burgers vector density is defined as
Figure 3.3. a) The lattice rotations in the indented single crystal sample b) Lower bound total GND density, measured with HR-EBSD with 500 nm spatial resolution and 50 nm.
Figure 3.4. GND densities on slip system 2 with 500 nm spatial resolution measurements. The lower plot shows net burgers vector density on the area bounded with the rectangular box.
Figure 3.5. GND densities on slip system 2 with 500 nm spatial resolution measurements. The lower plot shows net burgers vector density on the area bounded with the rectangular box.
Figure 3.6. GND densities on slip system 1 with 500 nm spatial resolution measurements. The lower plot shows net burgers vector density on the area bounded with the rectangular box.
For plane strain deformation conditions, $\ell$ is taken to coincide with the dislocation line direction which is the out-of-plane unit normal. Thus, the net Burgers vector density in plane will be given simply as

$$ (3.9) \quad B = \alpha \cdot \ell. $$

where $e_1$ and $e_2$ are unit vectors for the global coordinate axes. The net Burger’s vector density vectors expressed in the global coordinate system are shown in the lower part of the Fig. 3.4 to Fig. 3.6 (from different locations of the scan, indicated with white rectangular boxes on the upper part of the same figures). The crystallographic traces of the slip directions of slip system 1, 2 and 3 respectively are overlain in all three plots. It is observed that there are regions where the crystallographic traces are perfectly aligned with the net Burgers vector densities which indicates that the corresponding slip systems are predominantly activated in these regions. In Fig. 3.5b, the Net Burgers vector densities are aligned with crystallographic slip trace (2); this plot is taken from the lattice rotation jump region. The net Burgers vector density plots help to identify the source of the total dislocation density at each position, with respect to the crystallographic traces, and it gives insight to the type of deformation, whether it is pure kink shear or glide shear. If the discontinuity lies parallel to the slip direction, it is referred to as glide shear and if it is perpendicular to the slip direction, it is referred to as kink shear.

The same analysis is performed for the results obtained from the measurements with $50 \text{ nm}$ spatial resolution. The measurements are taken from the region of lattice
jump as shown in Fig. 3.3. High spatial resolution is necessary to resolve the complex dislocation networks and to identify the interaction among the slip systems. Fig. 3.7a to Fig. 3.7c show the distribution and magnitudes of GND densities for each slip system. The dislocation networks seem to be more discrete and the more localized than in the 500 nm measurements. The dislocation walls formed due to the slip system 1 are oriented perpendicularly to slip directions, as in the 500 nm measurements. The thicknesses of these dislocation density walls vary between 50 – 150 nm, suggesting a direct correlation with the spatial resolution which indicates the resolution limit of this technique. The dislocation density walls of slip system 2 are aligned with the antisymmetry line. The lower part of the Fig. 3.7b shows the net Burgers vector density and crystallographic slip traces overlaid on GND density on slip system 2. In Fig. 3.7b, the interaction among the slip system 1 and 2 can be easily observed. The net Burgers vector density changes abruptly at the upper part of the dislocation wall, which is an indication of the activation of slip system 1 in the upper part of the wall. In Fig. 3.7c, the horizontal “cuts” are observable, that are due to electron beam shift during the scan. As the spatial resolution is reduced the electron beam becomes more sensitive to any type of external vibrations or calibration errors and these experimental shifts are observed.

3.4.3. Effect of Step Size on Results and Uncertainty Analysis. In Fig. 2.1, the Burgers circuit is drawn around a single dislocation and the Burgers vector is shown. In order to capture a single dislocation, which must be considered as being geometrically necessary, the Burgers circuit must be sufficiently small. Assuming a Burgers vector of $b = 2.5 \times 10^{-10}$ m, the appropriate length scale, $L$, ranges from a few micrometers for dislocation densities of $10^{13}$ m$^{-2}$ to a few tens of nanometers for dislocation densities of $10^{16}$ m$^{-2}$, which are the very high dislocation densities at which the predominance of dislocations of like sign is expected to play an important
Figure 3.7. Lower Bound GND densities on each slip system $a)$ 1, $b)$ 2, $c)$ 3, using 50 nm spatial resolution. The lower plots show the net Burgers vector density in the regions indicated with white rectangular boxes in upper plots.
role in establishing constitutive behavior. For a planar Burgers circuit, $L$ scales as 
$L \sim Na/4$, where $N$ is the number of atomic lattice sites encountered during the 
circuit and $a$ is the atomic spacing. Kysar et al. [1] related $L$ to different length 
scales, one of which is the dislocation spacing which has been also defined as 
$1/\sqrt{\rho_{\text{GND}}}$ in Section 2.4, the other length scale is the inverse of the 
principle lattice curvature which scales as $1/b\rho_{\text{GND}}$. The gray zone in Fig. 3.8 shows an appropriate 
range for $L$ between the lines $1/\sqrt{\rho_{\text{GND}}}$ and $1/b\rho_{\text{GND}}$.

In our analysis, $L$ corresponds to the spatial step size of the HR-EBSD measurements. The HR-EBSD data is taken over uniformly spaced square grid. The edge 
length of an element equals the spatial step size of the measurement which is impor-
tant, since the lattice curvature is calculated by numerical differentiation on the 
grid. In essence, GND densities are calculated by normalizing by area of each square 
element and as the element size is increased —spatial resolution is also increased—
the GND densities will be averaged over a larger area. This is also observed from the 
500 nm and 50 nm results presented in the previous parts; it is shown that dislocation 
densities are averaged out with low resolution scanning and finer dislocation networks 
are obtained using high resolution measurements. In order to understand better the 
effect of spatial resolution on the characterization of the GND densities, 50 nm spatial 
resolution data set can be exploited. The spatial resolution is incrementally changed 
and lattice curvature as well as the GND densities are calculated for each spatial 
resolution.

In the upper row of Fig. 3.9, the lattice rotation maps for different spatial res-
olutions (50 nm, 100 nm, 150 nm, 200 nm, 250 nm) are shown; differences in lattice 
rotations with spatial resolution is very slight (piecewise linear interpolation is used 
in all contour plots). However, the effect of the spatial resolution on total GND densi-
ties is more apparent, as shown in lower row of the Fig. 3.9. As the spatial resolution 
increases, the total GND density networks are more finely resolved. The information
loss in total GND densities is observable from left to right. In order to make a quan-
titative comparison between the total GND densities and the step size, the linear
correlation of GND density data for each spatial resolution with respect to 50 nm is
calculated. Fig. 3.10 shows the linear correlation of the total GND densities using
50 nm spatial resolution results. As the spatial resolution decreases the correlation
coefficient also decreases.

The angular accuracy of traditional OIM is relatively poor at high spatial reso-
lutions, as the resolution increases the noise component of the data becomes domi-
nant and important amount information is lost [68]. In traditional OIM, the micro-
structure is characterized individually at each point independent of neighboring points,
which potentially increases the error in the data since there is error associated with
noise in each individual point. The cross-correlation based high resolution OIM meth-
ods use direct comparisons of patterns, meaning that there is only error associated
with the one measurement instead of error in each point.

The uncertainty of the GND density measurements can be quantified based on
the angular accuracy of the lattice rotation measurements. Two non-zero components
of the Nye's tensor are be calculated by the gradient of lattice rotations. We assume
that uncertainty in the lattice rotations, $\delta \omega_3$ will be same in both $x$ and $y$ axis.
The lower bound solution presented by Kysar et al. [1] and outlined in Section 2.2.2,
yields different GND density in each angular segments shown in Fig. 2.3. Since the
uncertainty in $\alpha_{13}$, denotes as $\delta \alpha_{13}$ and in $\alpha_{23}$, denotes as $\delta \alpha_{23}$ are the same, the
maximum uncertainty depends on the sum of the absolute value of the coefficients
of the both components. In this case, maximum uncertainty will be induced when
$\rho_{GND}^{total} = \frac{\sqrt{3}}{6} \alpha_{23} + \frac{1}{\sqrt{66}} \alpha_{13}$ (see Table 2, in Kysar et al. [1]). The uncertainty in $\delta \rho_{GND}^{total}$
can be written as
Figure 3.8. Appropriate range for spatial resolution, $L$, between the lines $1/\sqrt{\rho_{GND}}$ and $1/\rho_{GND}$. Uncertainty in HR-EBSD measurements (green symbol), and uncertainty of traditional EBSD.
FIGURE 3.9. The effect of spatial resolution on lattice rotations and lower bound GND densities on indented Nickel single crystal. 50 nm spatial resolution.

FIGURE 3.10. Linear correlation of lower bound GND densities based on 50 nm spatial resolution.
\[
(\delta \rho_{GND})^2 = \left( \frac{\partial \rho_{GND}}{\partial \alpha_{13}} \right)^2 (\delta \alpha_{13})^2 + \left( \frac{\partial \rho_{GND}}{\partial \alpha_{23}} \right)^2 (\delta \alpha_{23})^2.
\]

Re-arranging the terms Equation 3.11 and substituting \( \rho_{GND}^{\text{tot}} = \frac{\sqrt{3}}{b} \alpha_{23} + \frac{1}{\sqrt{6}} \alpha_{13} \)

\[
(\delta \rho_{GND})^2 = \frac{19}{6} \delta \alpha_{13} \frac{1}{b}
\]

\( \alpha_{13} = \frac{\delta \omega_3}{\text{dx}} \) which can be simply be expressed as \( \alpha_{13} = \frac{\Delta \omega_3}{L} \) where \( L \) is the spatial resolution. Assuming the uncertainty in \( \partial L \) is negligible compared to uncertainty associated to the lattice rotations, \( \delta \Delta \omega_3 \). Then \( \delta \alpha_{13} \) can be written as

\[
(\delta \alpha_{13})^2 = \left( \frac{\partial \alpha_{13}}{\partial \Delta \omega_3} \right)^2 (\delta \Delta \omega_3)^2
\]

Re-arranging the terms, \( \delta \rho_{GND}^{\text{tot}} \) can be written as;

\[
\delta \rho_{GND}^{\text{tot}} = \frac{19}{6} \frac{\delta \omega_3}{b} L
\]

Thus the uncertainty, \( \delta \rho_{GND}^{\text{tot}} \), changes inversely with the spatial resolution Table 3.1 shows the variation of uncertainty for different spatial resolutions

<table>
<thead>
<tr>
<th>( L )</th>
<th align="right">50 nm</th>
<th align="right">100 nm</th>
<th align="right">500 nm</th>
<th align="right">1000 nm</th>
<th align="right">2500 nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \delta \rho_{GND}^{\text{tot}} ) (m(^{-2}))</td>
<td align="right">( 1.25 \times 10^{13} )</td>
<td align="right">( 6.20 \times 10^{12} )</td>
<td align="right">( 1.24 \times 10^{12} )</td>
<td align="right">( 0.62 \times 10^{12} )</td>
<td align="right">( 0.24 \times 10^{12} )</td>
</tr>
</tbody>
</table>

The uncertainty in GND densities from traditional EBSD method where \( \delta \Delta \omega_3 = 0.5^\circ \) will be two order of magnitude higher than it is in HR-EBSD. For example, the uncertainty in GND density from a 50 nm spatial resolution measurement with
traditional EBSD method will be $1.25 \times 10^{15} \text{m}^{-2}$. It is a quite high variation and it limits quantification of lower dislocation densities at this spatial resolution.

In Fig. 3.8, the uncertainties in GND density measurements with both traditional EBSD (red symbols) and HR-EBSD (green symbols) are plotted. The uncertainty due to HR-EBSD results outside the range of $L$ which assures that length scales extracted from HR-EBSD is not affected by experimental uncertainty significantly. However, traditional EBSD is not within the appropriate length scale range, which means there will be significant amount of noise. The uncertainty analysis shows experimental lower bound for HR-EBSD and traditional EBSD.

3.4.4. Effect of Elastic Strain on GND densities. The curl$\varepsilon$ term in Eq.2.3 has been neglected in the calculations, in previous sections. It is assumed that under plane deformation conditions, the magnitude of elastic strain gradients are negligible compared to the lattice curvature. The elastic strains are roughly expected to be in the order of $10^{-3}$ ($\varepsilon_{\text{max}} \approx \tau_{\text{max}}/E \approx 0.001$) where $\tau_{\text{max}}$ is the approximate maximum critical resolved shear stress of the material in a highly strained state and $E$ is Young’s modulus. However, the most severe elastic strains are expected to be in the region where there is severe lattice rotation jump so it is worthwhile to use the data to verify this assumption. Thus in this section, the strain tensor obtained by high resolution measurements is incorporated into the calculations.

The elastic strain measurements are shown in Fig. 3.11. Care must be taken when interpreting them, mainly because the stress state of the sectioned crystal is different than that of the indented crystal. In addition, the experimental methods to make the strain characterization are still under development[69]. Nevertheless, we use these measurements as order-of-magnitude estimates.

The strain gradient components are included in the Eq. 2.3 and the dislocation densities are recalculated. The results for total GND densities with and without
strain gradient terms and also percentage difference between these two are shown in Fig. 3.12. The uncertainty in the elastic strain measurements amplified this difference, at some locations. These amplified differences are local, not continuous over the dislocation networks of lattice jump. The difference is in the range of $30 - 50\%$, but there are no differences in the salient features of the GND field. The results will be compared with numerical modeling results in next sections.

### 3.5. Numerical Analysis-3D Crystal Plasticity Model

A three-dimensional finite element model is developed to identify the size scale for the region where out of plane deformation is comparable with the in-plane one, and also to determine the degree of deformation that occurs after cutting the specimen.
from the midsection. The 3D numerical model of indentation problem was simulated using ABAQUS/Standard incorporating a single crystal plasticity material model written by Huang [70] and modified by Kysar [71]. The material model allows to input the crystallographic orientation, material parameters such as anisotropic elastic stiffness tensor, strain hardening components, and initial hardening rate. In the 3D model, 8-node linear brick elements with reduced integration with hourglass control element are used.

The indenter was modeled as a rigid body, since the elastic stiffness of tungsten carbide is much higher than of the nickel crystal. The surface to surface contact is used to simplify the singularity issue due to the nature of the problem. The symmetry
in deformation is utilized so only half the specimen from the indentation axis was modeled. As shown in the Fig. 3.13, in order to simulate the effect of sectioning, the nodal constraints on the midsection symmetry plane were relaxed after the indentation process was complete. The results demonstrate that the elastic strain state does not change significantly after the release.

In order to find the region where the in-plane deformation assumption is valid, the out-of-plane components of the elastic strain tensor, $\varepsilon_{13}, \varepsilon_{23}, \varepsilon_{33}$ are compared with the in-plane ones $\varepsilon_{11}, \varepsilon_{12}, \varepsilon_{22}$. The region where out-of-plane components are two order of magnitude less than the in-plane ones is found. The position of this region is expressed as in terms of the indentation depth, $d$, and it is found that the plane deformation conditions are reached at a depth of $9d$ into the crystal on the (110) plane, where $d$ is indentation depth. In the present study, the indenter penetrated 200 $\mu$m into the sample, thus plane deformation conditions are satisfied at/on planes 1.8 mm or more from the free (110) surface. The experimental sample was cut from its mid section which was 2.5 mm away from the surface. The simulation results show that at 2.5 mm depth, out of plane strain components are negligible and of the order of $10^{-8} - 10^{-7}$.

It should be noted that the simulation also shows that plastic deformation is consistent with plane deformation on 3 in-plane slip systems.

3.6. Conclusion

The HR-EBSD measurements provide rich data sets that can be utilized to resolve the complexity in the GND densities on each slip system and to understand their interactions. In Chapter 2, a detailed analysis on extracting length scales and resolving complexities is presented, and in this chapter, experimental details are presented. The analysis in this chapter can be outlined as follows.
Figure 3.13. 3D Finite Element Model
• The sample preparation in HR-EBSD consists of several stages. Among all, polishing is the most critical one. In this paper, we used mechanical and chemical polishing which provided sharp, high quality Kikuchi patterns.
• The net Burgers vector density analysis shows the activation of the slip systems and their interactions. The regions where the net Burger Vector Density aligns with the crystallographic traces are shown; in these regions where the slip traces are perpendicular to cell walls and kink type of shear is detected. These regions are resolved better with 50 nm spatial resolutions.
• The effect of spatial resolution is examined and the appropriate size of net Burgers vector is discussed. The effect of spatial resolution is presented based on 50 nm measurements. It is observed that the spatial resolution has significant influence in resolving GND densities. In general as the step size increases, the uncertainty increases and it becomes difficult to quantify low dislocation densities.
• Uncertainty analysis showed that HR-EBSD method provides adequate accuracy to extract length scales. It is shown that for 50 nm spatial resolution measurements, uncertainty is of the order of $10^{13} \text{m}^{-2}$.
• The HR-EBSD measurements provide superior accuracy, which is adequate to resolve the physical length scales. The uncertainty in traditional EBSD is in the range of physically possible length scales which makes length scale analysis not reliable.
• The elastic strains of the deformed crystal is measured. However, the elastic strains are expected to be $\sim \pm 0.001$ and the uncertainty is only half order of magnitude lower. This induces a considerable amount of noise after numerically differentiating twice. Elastic strain measurements with HR-EBSD is a novel method, and it gives important qualitative information. Yet, elastic strain measurements a still need improvements in terms of accuracy.
• A 3D finite element analysis validated the plane deformation assumption. It is shown that the sample has to be cut from the surface about 9 times of the penetration depth of the indenter.
CHAPTER 4

Length-scale Effect Due to Periodic Variation of Geometrically Necessary Dislocation Densities

4.1. Abstract

Strain gradient plasticity theories have been successful in predicting qualitative aspects of the length scale effect, most notably the increase in yield strength and hardness as the size of the deforming volume decreases. However, new experimental methodologies enabled by recent developments of high spatial resolution diffraction methods in a scanning electron microscope give a much more quantitative understanding of plastic deformation at small length scales. Specifically, geometrically necessary dislocation densities (GND) can now be measured and provide detailed information about the microstructure of deformed metals in addition to the size effect. Recent GND measurements have revealed a distribution of length scales that evolves within a metal undergoing plastic deformation. Furthermore, these experiments have shown an accumulation of GND densities in cell walls and a variation of the saturation value of dislocation densities in these cell walls as well as other dislocation structures. In this study a strain gradient plasticity framework is extended by incorporating a physical quantity obtained from experimental observations: the saturation value of GND densities. The proposed model is tested with constrained shear and pure bending problems. The results presented show patterning in the GND density profile depending on the prescribed initial variation of the saturation value of GND densities.
4.2. Introduction

The microelectronics and micro-electro-mechanical systems (MEMS) industries regularly employ highly-stressed thin metal films in micro-scale devices. Several researchers demonstrated the existence of a length-scale effect in plasticity over the past fifteen years [53, 72, 73]. Their experimental methods consisted either of bending, twisting or compressing small well-defined volumes of material, or by employing indentation on a material. The measured quantities in the experiments were effective forces and displacements applied to the boundaries of the specimens. As such the experiments characterized the global response of the specimens, but the experiments gave no direct information about the state of the material within the system.

The foremost mechanism responsible for plastic deformation in a crystal is the creation and motion of crystalline defects called dislocations, which are line defects constrained to certain crystallographic planes. It is important to note that dislocations are signed quantities. Once the applied stress state in a region of the crystal exceeds a critical value, the dislocations move through the crystal, with the positively and negatively signed dislocations moving in opposite directions. The mass flux that accompanies the motion of the dislocations induces permanent, or plastic deformation. Under conditions in which the applied stress is homogeneous over large regions of the domain, approximately equal densities of dislocations of opposite signs accumulate within the crystal. Such an accumulation of dislocations is known as a Statistically Stored Dislocation (SSD) density, which exerts no long-range influence with the material. However, when a significant gradient of stress exists within the domain, a net density of one sign of dislocations accumulates in certain regions of the crystal while a net density of dislocations of the opposite sign accumulates in neighboring regions. The net signed density of dislocations is known as the Geometrically Necessary Dislocation (GND) density. The presence of the GND densities
introduces both a curvature as well as a strain into the crystal lattice. Formally, the relationship between the GND densities and the lattice distortion follows from a consideration of the compatibility of deformation in circumstances when multiple deformation mechanisms can be activated [22]. The GND density (unit: \(1/\text{length}^2\)), inherently contains a length scale, so GND densities are often invoked as state variables in strain gradient plasticity theories as a means of predicting the plasticity length scale effect [5, 16, 41, 74, 75, 76, 77].

There are two main classes of strain gradient plasticity theories that incorporate GND densities. The first one is the work-conjugate method, introduced by Gurtin [40] in which the higher orders stresses are work-conjugate to the gradients of plastic strain; in other words, GND densities. In this type of model, the principle of virtual work is extended with the addition of this new work-conjugate term. The second class strain gradient theory is crystal plasticity theory and the GND densities are not conjugated to higher order stresses [5, 16, 75, 74]. Instead, the back stresses due to GND densities are included in a crystallographic slip rate formulation. These two classes of theories are equivalent in some cases. A particular back stress formulation can be shown to be equivalent Gurtin’s type energetic formulations, where free energy is directly proportional to the square of the plastic gradient of slip or equivalently, GND densities (see [38])

Both of the models mentioned above can be used to analyze the size effect in materials using phenomenological length scale parameters, and both models predict reasonable stress-strain response; albeit they do not provide any information about the micro-structure. Recent GND density measurements on indented single crystal samples by Kysar et al. [1] showed extensive patterning as a result of plastic deformation. Dislocations are observed to accumulate in cells walls and to develop intricate dislocation networks with apparent quasi-periodicity. The cell width and spacing are observed to vary with the amount of deformation. It is also observed that disloca-
tion densities accumulate and reach a threshold in cell walls that typically have peak values between $10^{14} \sim 10^{15}$ m$^{-2}$.

In this paper, a non-work conjugate type strain gradient plasticity framework proposed by Kuroda and Tvergaard [38] is extended by a new hardening rule that depends on the saturation value of GND densities. An initial distribution for the saturation value of GND densities is prescribed to study the effect of period of GND variation on the mechanical response. Two different problems are used as benchmarks: constrained shear and pure bending.

The format of this paper is as follows. In Section 4.3, the basic concepts to formulate the framework including the kinematics of the crystallographic slip and GND densities and constitutive relations are presented. Incorporation of GND densities into the framework and the new hardening rule are also explained in this section. The finite element implementation of the introduced framework is given in Section 4.4, and the link between the back stress formulations and the elastic-viscoplastic constitutive relation is explained. In Section 4.5, two different problems are used as benchmark: constrained shear and pure bending. The model is calibrated using phenomenological material parameter associated to the back-stresses, then a periodic variation is prescribed for the GND density within the new hardening rule. The effect of different type of variations on the mechanical response is explored. The conclusions are presented in Section 4.6.

### 4.3. Constitutive Framework

#### 4.3.1. Kinematics of the crystallographic slip and GND Densities.

In this paper, the framework developed by Kuroda and Tvergaard [16] is implemented. The formulation is based on classical crystal plasticity and it differs substantially from classical theory with the incorporation of GND densities into the constitutive
The formulation presented here is confined to the small strain assumption. The total strain rate is decomposed as

\[ \dot{\varepsilon}^{\text{tot}} = \dot{\varepsilon}^e + \dot{\varepsilon}^p \]

where \( \dot{\varepsilon}^e \) is the elastic strain rate and \( \dot{\varepsilon}^p \) is the plastic strain rate. In crystal plasticity, it is assumed that plastic deformation occurs due to the crystallographic slip on a total of \( N \) slip systems. The rate of plastic strain, \( \dot{\varepsilon}^p \), can be written by using the basic kinematic relation

\[ \dot{\varepsilon}^p = \sum_{\alpha=1}^{N} \dot{\gamma}^\alpha s^\alpha \otimes m^\alpha = \sum_{\alpha=1}^{N} \dot{\gamma}^\alpha P^\alpha \]

where \( \dot{\gamma}^\alpha \) is the slip rate, \( s^\alpha \) and \( m^\alpha \) are the slip direction and slip normal unit vector on the crystallographic plane \( \alpha \) (\( \alpha = 1, 2, \ldots N \)) respectively. For simplicity, the second order tensor, \( P^\alpha \), which is also known as Schmid orientation tensor is employed. The Schmid tensor constitutes the crystallographic basis for the plastic deformation rate.

GND densities are defined as the net signed density of all dislocations evolving during the deformation that arise due to gradients of plastic slip to preserve the lattice compatibility in the crystal. Ashby [22], related GND densities to the slip gradient with a simple linear relationship. According to his definition, a plastic slip gradient in the direction of slip leads to a density of edge dislocations and a plastic slip gradient in the direction normal to the slip is accompanied by screw dislocations. GND densities for edge and screw dislocations can be calculated based on slip gradients as follows.
where the subscript $e$ indicates the edge dislocations which are parallel to the slip direction, $s$ indicates the screw dislocations which are perpendicular to the slip direction ($p^\alpha = m^\alpha \times s^\alpha$) and $b$ is the magnitude of the Burgers vector.

4.3.2. Constitutive Relations. In this elastic-viscoplastic constitutive framework, the elastic strain rate $\dot{\varepsilon}^e$, is assumed to be governed by Hooke’s law for isotropic elasticity with $\dot{\sigma} = 4^C \dot{\varepsilon}^e$, where $\sigma$ is the stress tensor and $4^C$ is the fourth order isotropic elasticity tensor. The total strain rate, $\dot{\varepsilon}^{tot}$, in Eq. (4.15) can be expressed as

$$
\dot{\varepsilon}^{tot} = 4^C \dot{\sigma} + \sum_{\alpha} \dot{\gamma}^\alpha \mathbf{P}^\alpha
$$

where slip rates, $\dot{\gamma}$ are calculated using the viscoplastic power law suggested by Hutchinson [78] and Pierce et. al [79] and later extended by Kuroda and Tvergaard [16] in order to introduce back stresses:

$$
\dot{\gamma}^\alpha = \dot{\gamma}_0 \text{sign}(\tau^\alpha - \tau_b^\alpha) \left( \frac{|\tau^\alpha - \tau_b^\alpha|}{g_{eff}^\alpha} \right)^{1/m}.
$$

In Eq. (4.19), $\dot{\gamma}_0$ is a reference slip rate, $m$ is the the rate sensitivity exponent and $g_{eff}^\alpha$ is the effective slip resistance. The plastic slip rate on each slip system, $\dot{\gamma}^\alpha$ is related to the corresponding effective shear stress which is the difference between the
resolved shear stress, $\tau^\alpha$, and the back stress $\tau_b^\alpha$. The resolved shear stress can be found by using the relation $\tau^\alpha = P^\alpha : \sigma$. The back stress, $\tau_b^\alpha$, is due to the internal stresses induced by dislocations and it reflects contributions arising from dislocations on all slip systems. The long range internal stress is only due the GND densities. SSD densities are geometrically redundant and do not induce any long range internal stress. Evers et al. [74] suggested that the back stress should include the effect of the long-range stresses that are caused by spatial variations in the irregularity in the crystal due to the dislocations. Considering a beam with a uniform GND density and a uniform lattice curvature relative to a undeformed reference configuration, they argue that the long range stress field will cancel out due to symmetry. Hence, the long-range stresses are not due to a GND density, but rather the GND density gradient. Following this idea, the back stress is defined in terms of the gradient of GND densities only and the following functional form has been used in several studies (see [5, 16, 74])

\[
\tau_b^\alpha = f(\nabla \rho_{GND(e)}^1, \nabla \rho_{GND(e)}^2, \ldots \nabla \rho_{GND(e)}^N).
\]

(4.20)

Kuroda and Tvergaard [39], defines the back stress as the gradient of dislocation densities in the slip direction with the following specific functional form

\[
\tau_b^\alpha = b\eta \sum_{\beta=1}^{N} S^{\alpha,\beta} \nabla \rho_{GND(e)}^\beta.
\]

(4.21)
where $\eta$ is constant for all slip systems and defined as $\eta = \tau_o L^2$, $\tau_o$ is the initial slip resistance, $L$ is a phenomenological length scale parameter and $S^{\alpha\beta}$ is the slip system interaction coefficient. In this study, only self interactions of the slip systems are considered (i.e. $S^{\alpha\beta} = 0$ if $\alpha \neq \beta$).

The main connection between the dislocation density evolution and the crystal plasticity is done through the back stress relation shown in Eq. (4.20). The GND density is also linked to the constitutive relations through the $g^{\alpha}_{\text{eff}}$ term in Eq. (4.19). The slip resistance, $g^{\alpha}_{\text{eff}}$, develops due to short-range interactions between all dislocations; both SSDs and GNDs [5]. It is also referred as effective slip resistance, which is introduced in mechanism based strain gradient theories [76, 80], inspired by Taylor type hardening [81] which assumes that the slip resistance of a single-slip system is directly proportional to the square root of the sum of the SSD density, $\rho_{\text{SSD}}$, and GND density, $\rho_{\text{GND}}$:

$$
\tau = \alpha \mu b \sqrt{\rho_{\text{SSD}} + \rho_{\text{GND}}}.
$$

Here $\mu$ is the elastic shear modulus and $\alpha$ is an empirical coefficient, usually taken around $0.3 - 0.5$. In principle the SSD density, $\rho_{\text{SSD}}$, accumulates due to random trapping and there is no simple theory predicting their evolution. In mechanism based strain gradient theories, the SSD density on a single slip system $\rho_{\text{SSD}}^\alpha$ is related to the slip resistance, $g^\alpha$ of the slip system by the Taylor relation when ignoring the GND densities [76, 80], defined as

$$
g^\alpha = \alpha \mu b \sqrt{\rho_{\text{SSD}}^\alpha}.
$$
The effective slip resistance, $g_{\alpha eff}$ can be written by combining the contribution of the GND density; with the SSD density term to obtain

\begin{equation}
(4.24)
\quad g_{\alpha eff} = \sqrt{(g^\alpha)^2 + (\alpha \mu b)^2 \rho_{GND}^\alpha}.
\end{equation}

The evolution of the slip resistance can be expressed by a simple phenomenological linear hardening equation

\begin{equation}
(4.25)
\quad \dot{g}^\alpha = \sum_{\beta=1}^{N} h^{\alpha\beta} |\dot{\gamma}^\beta|
\end{equation}

where the hardening coefficient is given by $h^{\alpha\beta} = \left[q + (1 - q)\delta^{\alpha\beta}\right] h^\beta$ [80]. In this paper, $q$ is assumed to be unity implying that all slip systems harden at same rate. Various formulations for the self hardening $h^\beta$ modulus, are available based on experimental observations [82, 79]. Hardening coefficients are fitted to experimental results, by using quantities such as a yield strength, or a saturation value of flow stress that depends on a saturation value of slip resistances. In the present study, motivated by the experimental results in Kysar et al. [1], new hardening rules based on the saturation value of GND density, $\rho_{GND}^{sat}$ are used.

As outlined in the introduction, experimental measurements on indented single crystal samples have revealed non-homogeneous distributions of GND densities and a distribution of different length scales. Extensive patterning and dislocation cell formation is observed. Maximum dislocation densities are measured on indented single crystal samples are on the order of $10^{14} - 10^{15} \text{m}^{-2}$; most attain maxima on
cell walls or on randomly distributed dislocation bundles. This type of deformation can not be predicted by a quadratic energy function, which yields a homogeneous distribution of GND densities. Thus, the Kuroda-Tvergaard model with a linear GND hardening rule (Eq. (4.21)), which is equivalent to quadratic free energy formulation, can not predict this type of complex distribution. However, the hardening rule can be modified to obtain similar features.

In this study, we aim to obtain realistic patternning by introducing new hardening rules, that depend on saturated GND densities that vary spatially. A new multiplier, $G$, to the GND term in Eq. 4.24 is defined. This coefficient is based on the current GND densities, $\rho_{GND}^\alpha$, and scaled with the saturation density value of GND density, $\rho_{GND}^{sat}$ according to

\begin{equation}
G = 1 + \left( \frac{|\rho_{GND}^\alpha|}{\rho_{GND}^{sat} M_{GND}} \right)^n
\end{equation}

where the non-homogeneous distribution for the saturation value of GND density is implemented through the spatially varying $M_{GND}$ term, and $n$ which is defined to be equal 10. Choosing $n$ as relatively large number as 10, reduces the contribution of GND density that is less than the saturation value.

The general form of Eq. (4.24) can be expressed as

\begin{equation}
g_{eff}^\alpha = \sqrt{(g^\alpha)^2 + G (\alpha \mu b)^2 \rho_{GND}^\alpha}.
\end{equation}
4.4. Numerical Implementation

The constitutive relations listed in previous section are incorporated into the conventional equilibrium equation and the dislocation density field equations. The conventional equilibrium equation can be written as

\[ \text{div}\sigma + f = 0 \]  \hspace{1cm} (4.28)

where “\text{div}” is the divergence operator and \( f \) is the body force. Dislocation density equations are obtained in weak form based on Eq. (4.17). The solution for dislocation densities \( \rho_{GND(e)}^\alpha \) and \( \rho_{GND(s)}^\alpha \) necessitates prescribing boundary conditions for the GND densities. Different types of physical conditions can be modeled through these boundary condition. We can constrain dislocation densities to vanish at a free surfaces thereby modeling that dislocations are free to exit through the surface [? 16], and conversely on hard surfaces no constraint can be imposed.

Eq. (4.28) and the weak form of Eq. (4.17) are linked through back stress \( \tau_h^\alpha \) term in the constitutive relations. As shown in Eq. (4.19), the slip rate is a function of the back stress which is defined as a function of the spatial derivatives of GND densities in Eq. (4.21). Thus, if the GND densities and the spatial variation of the deformation are known, all the relevant quantities can be determined.

The governing equations are solved using a staggered scheme. The computations are performed with eight-node plane strain parametric elements with full integration for both displacements and GND densities. Displacement rates and slip rates are integrated using the Forward Euler method. Convergence to stable solutions is ensured by using small time steps.
The finite element formulation of this framework is the same as in Kuroda and Tvergaard [38]. The details for finite element implementation will not be presented here since the main focus of this study is on formulations for different hardening rules that give realistic predictions according to experiments. In the next section, two basic problems will be used as benchmarks for different type of hardening rules and further details about numerical stability will be presented.

4.5. Analysis

The multiplier, $G$, in Eq. (4.27) gives flexibility of defining different hardening profiles through $M_{GND}$ and $\rho_{GND}^{sat}$ terms in it. The profile of the $G$ is specified through $M_{GND}$ and it is scaled by $\rho_{GND}^{sat}$ term as shown in Eq. (4.26). First a constant $M_{GND}$ profile will be prescribed with a very high, $\rho_{GND}^{sat}$, value ($\sim 10^{50} \text{ m}^{-2}$), which makes the multiplier $G$ unity to obtain Taylor type hardening in order to calibrate the model with material length scale parameter $L$. After the appropriate range for $L$ is found, three different type of $M_{GND}$ profiles will be tested. Two sinusoidal profiles and one random profile for the variation of saturated GND density will be analyzed.

The profiles will be tested on two basic problems. The first problem is the pure shear problem with two slip systems that are oriented $60^\circ$ apart from each other. This problem has been analyzed by several researchers as a benchmark problem for studying size-effect [6, 38, 83]. The second problem is simple bending with three slip systems.

4.5.1. Constrained Shear. A schematic view of the plane strain constrained shear problem is shown in Figure 4.1a. A thin layer, of height $H$ is constrained at the top and the bottom ends and deformed with a constant displacement rate of $\gamma_o H$. The layer is assumed to be infinitely wide, thus periodic boundary conditions are prescribed for both the displacements and the GND densities on the sides of the
computational domain. The macroscopic boundary conditions for Eq. (4.28) can be listed as follows:

\[
\begin{align*}
    u_1(x_1, 0) = u_2(x_2, 0) &= 0 \\
    u_1(x_1, H) = \dot{\gamma}_0 H t = \Gamma(t) H, & \quad u_2(x_1, H) = 0 \\
    u_1(0, x_2) = u_1(W, x_1), & \quad u_2(0, x_2) = u_2(W, x_2)
\end{align*}
\]

whereas the GND boundary conditions are given by:

\[
\begin{align*}
    \rho_{GND}^a(0, x_2) = \rho_{GND}^a(W, x_2).
\end{align*}
\]
As plane strain conditions are assumed, the model is confined to evolution of edge dislocations with dislocation lines parallel to the out of plane direction.

The material parameters used are listed in Table 4.1. The strip is deformed until overall shear strain of $\Gamma_{\text{max}} = 0.05$ is reached.

<table>
<thead>
<tr>
<th>Table 4.1. Material parameters</th>
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<td>Variable</td>
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4.5.1.1. *Taylor Type of Hardening* ($G = 1$). A similar problem was analyzed for an isotropic material by Niordson and Hutchinson [84] using a basic $J_2$-flow theory with the incorporation of strain gradients in the hardening law as proposed by Acharya and Bassani [85]. They showed that the lower order theory leads to a vertex-type localization at the locations where the plastic strain is maximum and its gradient initially vanishes. Volokh and Hutchinson [86] related the presence of vertices to the multiplicity of solutions due to the mathematical formulation of lower order theories. A similar type of hardening law within the present framework since the slip gradient terms appear as GND density, $\rho_{GND}^g$ in the denominator of Eq. (4.19), whereas the plastic slip gradient was used to enhance the hardening modulus in [84] and [85].

Figure 4.2a shows GND density distributions on the slip system 1 with $L/H = 0.5, 1, 5$ and 10 at five deformation levels ($0.2\Gamma_{\text{max}}, 0.4\Gamma_{\text{max}}, 0.6\Gamma_{\text{max}}, 0.8\Gamma_{\text{max}}, \Gamma_{\text{max}}$). The dislocation densities on two symmetrically aligned slip systems are equal, and they attain their maximum value close to the middle of the strip where the plastic shear is maximum due to the development of vertex. The vertex starts developing early during deformation and sharpens over the time. The sharpness of the vertex depends on the discretization; as the mesh is refined the vertex in plastic strain profile becomes more acute as also discussed by Niordson and Hutchinson [84]. In the present work, another gradient term, the gradient of GND density in the slip direction, is
introduced within the back stress term, $\tau_0^\alpha$. The back-stress formulation in Eq. (4.20) includes the length scale parameter $L$ that regulates the contribution of the gradients of GND densities and has a regularizing effect on the results. Figures 4.2a-4.2d shows the evolution of dislocation density on slip system 1 (cf. Figure 4.1a) for different $L/H$ ratios. As $L/H$ increases, it is observed that the GND distribution smoothens along the $x_2$ axis. For $L/H = 10$, a nearly parabolic strain distribution is predicted which yields an almost linear GND density.

Figure 4.3 summarizes the effect of $L/H$ on GND density distribution along $x_2$ axis at the maximum deformation level, $\Gamma_{\text{max}}$, and it shows that as $L/H$ increases GND density profile becomes almost linear. This type of distribution is reasonable for uniformly deformed crystal with no imperfections. However imperfections and physical defects trigger non-homogeneous deformation. In the following section we study the effect of imperfections within the same framework.

4.5.1.2. A sinusoidal profile for saturation value of GND densities. When a significant gradient of stress exists within the domain, a net density of one sign of GNDs accumulates in certain regions of the crystal while a net density of dislocations of the opposite sign accumulates in neighboring regions. Based upon electron back scattering diffraction (EBSD) measurements on indented single crystal samples revealed patterns of such self organized dislocation structures \cite{1}. Different types of dislocation structures evolved during deformation: dislocation pile ups, cell walls, discrete dislocation bundles. Each type of dislocation structure has different stress field associated with it and formulations for the stress fields of various dislocation structure have been investigated \cite{87}. A back stress at a material point in a crystal arises due to the combination of all the stress fields of all of these dislocation structures. This results in a spatial distribution of the dislocation structures and a natural variation in the dislocation densities measured experimentally in \cite{1}, where it is observed that the dislocation densities accumulates in a quasi-periodic pattern and the GND density
Figure 4.2. Evolution of $p_{GND}^{(1)}$ along $x_2$ for different $L/H$ ratio a) 0.5, b) 1, c) 5, d) 10. Each curve corresponds to $0.2\Gamma_{max}$ of incremental deformation.
reaches a threshold value in cell walls that typically varies between $10^{14} \sim 10^{15} \text{m}^{-2}$. To capture this effect, we define a new variation for the initial distribution of saturation values of GND density to analyze how the new length scales evolve and how they are related to initial material imperfection.

The initial distribution of the GND saturation value is introduced through Equation (4.26), and it is taken to vary along the $x_2$—direction. First, a simple sinusoidal function is employed

\begin{equation}
M_{GND} = 1 + A \sin(M x_2/H)
\end{equation}

where $A$ is the maximum relative deviation from $\rho_{GND}^{sat}$ which is set to 0.3 and $M$ is the number of the variations throughout the height $H$. Two different $M$ values are
Figure 4.4. $M_{GND}$ distribution for $\rho_{GND}^{sat}$ with different frequencies a) $M = 3$, b) $M = 4$

used in the present study as shown in Figure 4.4, and they can be interpreted as the characteristic length of the periodic GND density pattern employed. A higher $M$ value will yield more the oscillations in GND density along $x_2$ axis.

In Eq. (4.19), there are two gradient terms: the GND densities (as the gradient of plastic slip) in $g_{eff}^\alpha$ and the gradient of the GND densities within $\tau_\beta^\alpha$. The multiplier $G$ in Eq. (4.27) controls the effect of GND densities whereas the material length scale parameter, $L$ in Eq. (4.21) regulates the contribution of the gradient of GNDs. As the GND density increases and an imperfection is defined, a Compton between the gradient terms starts and regulated by $G$ and $L$.

In Figure 4.5, the GND density evolution over time is shown for different saturation values with the profile $M_{GND}$ as in Figure 4.4a. The vertex type of shear localization, is observed for smaller saturation values. Specifying low saturation values, increases $G$ in Eq. 4.27 which increases the contribution of dislocation densities over the gradient of GND densities. Even though a fairly large length scale is chosen and $L/H$ is set to 5, the effect of dislocation densities -and, hence the slip gradients- is more dominant. The vertex type of shear is not observed as the saturation value is increased. For $\rho_{GND}^{sat} = 1 \times 10^{14} \text{m}^{-2}$, in Figure 4.5, a patterning and accumulation in
Figure 4.5. Evolution of $\rho_{GND}^{(1)}$ along $x_2$ axis with $M_{GND}$ as shown in Figure 4.4a) for different $\rho_{sat}^{GND}$ values a) $5 \times 10^{13}$ m$^{-2}$, b) $1 \times 10^{14}$ m$^{-2}$, c) $1 \times 10^{14}$ m$^{-2}$, d) $5 \times 10^{14}$ m$^{-2}$. Each curve corresponds to $0.2 \Gamma_{max}$ of incremental deformation.
dislocation density over time is observed. The spacing between curves decreases even though the amount of deformation is kept constant. The patterning depends on the profile of the saturation value depicted in Figure 4.4a. The saturation value takes its local minimum value at $x_2/H = 0.9$ and this is reflected as a decrease in GND density at the same point. For $\rho_{\text{GND}}^{\text{sat}} = 1 \times 10^{14} \text{ m}^{-2}$, in Figure 4.5c, the patterning starts to smoothen out since the dislocation density does not achieve the higher saturation value. If the saturation value is set to a value much higher than the maximum GND density evolved during deformation as in Figure 4.5d since the large exponent, $n = 10$ in Eq. (4.26) , $G$ approaches 1 which gives results equivalent to the Taylor type model discussed in Section 4.5.1.1.

In the previous section, it is shown that the material length scale parameter, $L$, smoothens the GND density profile along $x_2$ axis when $G$ is unity. Effect of $L$ on complex patterning, when $G$ varies should also be examined. To analyze the effect of $L$ for more complex patterning, the spatial frequency, $M$ of the sinusoidal profile $M_{\text{GND}}$ is increased 4 as shown in Figure 4.4b. The saturation value $\rho_{\text{GND}}^{\text{sat}}$ is set to $1 \times 10^{14} \text{ m}^{-2}$. The macroscopic shear strain profile along $x_2$, an overall shear strain of $\Gamma_{\text{max}}$, is shown in Figure 4.6 and it is observed that a significant increase length scale leads to a smoother dislocation density profile as shown in Figure 4.6b.

4.5.1.3. A random variation for saturation value of GND densities. A saturation value that varies randomly along the $x_2$ axis is now investigated. $M_{\text{GND}}$ is defined using a Fourier sine series with random coefficients as follows

\begin{equation}
M_{\text{GND}} = 1 + \sum_{k=1}^{K} \frac{A_k}{\max \{A_k\}} \sin(2\pi k x_2 / H).
\end{equation}
Figure 4.6. a) Macroscopic shear profile, b) $\rho_{GND}^{(1)}$ profile along $x_2$ axis for different $L/H$ ($\rho_{sat} = 1 \times 10^{14} \text{m}^{-2}$ and at $\Gamma_{max}$).

This ensures a smooth yet random profile, as shown in Figure 4.7, with $K = 6$ and the coefficients $A_k$ being normally distributed pseudo-random variables. The effect of saturation value on the evolution of GND density is illustrated through the progression of Figure 4.8a to 4.8c. The patterning at both ends depends on the imposed profile, as observed at both ends of the strip. The smoothing effect of an increase in $L/H$ is also observed.

Both of these hardening profiles show that patterning and accumulation can be obtained by introducing initial variations for the GND saturation density. The initial variations trigger the localization through GND densities and localization is suppressed by the effect of back stresses. If experimental results report a precise a quantitative distribution of the saturation value, it could be input to the model.

In Figure 4.9, the normalized shear stress versus macroscopic strain is shown for $\rho_{GND}^{sat} = 1 \times 10^{14} \text{m}^{-2}$ with different GND density profiles and Taylor type of hardening, and with $L/H = 10$, and an overall deformation of $\Gamma_{max}$. The hardening rate changes throughout the deformation. At the early part of deformation lower hardening rate are akin to Stage I, because of the low hardening rate, albeit with
double slip. The hardening increases with a steeper rate in the latter part of the deformation which corresponds to Stage II hardening. In Stage II hardening multiple slip systems are activated and patterning and cell formations evolve at early levels of this stage [88]. In the present study, the patterning is imposed initially and later manifests itself at larger strains. Figure 4.9 also shows that as patterning becomes more complex, hardening also increases. The hardening level for sinusoidal profiles with different wave lengths (c.f Figure 4.4) are nearly same, whereas for the random profile (c.f Figure 4.7), which has less variation, yields slightly lower hardening rate. The Taylor type of hardening which does not accommodate any patterning has the lowest hardening rate.

4.5.2. Pure Bending. In order to illustrate the method under a different deformation condition, a plane strain bending problem is analyzed. Three slip systems are aligned 60° from each other as shown in the schematic view in Figure 4.1b. An infinitely long beam of height $H$ is loaded by a moment $M$, which gives rise to homogeneous curvature. The beam is modeled by a strip with the following boundary conditions.
Figure 4.8. Evolution of $\rho_{GND}^{(1)}$ along $x_2$ axis with randomly varying saturation value with a base value of $\rho_{sat}^{GND} = 1 \times 10^{14}$ m$^{-2}$, for different $L/H$ values a) 5, b) 10, c) 20.
Figure 4.9. Normalized Shear Stress vs Macroscopic Shear for Taylor Type of hardening and different hardening profiles: Sinusoidal for $M = 3$ and $M = 4$ (c.f Figure 4.4), the random profile (c.f Figure 4.7) ($\rho_{\text{sat}} = 2 \times 10^{14} \text{m}^{-2}$ and $L/H = 10$)

\begin{equation}
\begin{aligned}
    u_1(0, x_2) &= 0 \\
    u_1(W, x_2) &= \dot{\gamma}_o t H \kappa x_2
\end{aligned}
\end{equation}

where $\kappa$ is the curvature as shown in Figure 4.1. In contrast to the shear case, the GND density at both top and bottom ends of the strip is constrained to be zero, and if no imperfection were to be imposed and elasticity neglected, it would be constant along the $x_2$ axis. Dislocations are assumed to be constant along $x_1$ axis and they can exit the free surfaces, thus dislocation densities are constrained with the following boundary conditions:

\begin{equation}
\begin{aligned}
    \rho_{\text{GND}}^{\alpha}(x_1, 0) &= \rho_{\text{GND}}^{\alpha}(x_1, H) = 0. \\
    \rho_{\text{GND}}^{\alpha}(0, x_2) &= \rho_{\text{GND}}^{\alpha}(W, x_2) = 0.
\end{aligned}
\end{equation}

Plane strain deformation is considered, and it is assumed that only edge dislocations evolve. The material parameters listed in Table 4.1 are also used in this example.
The beam is bent such that maximum curvature is $\kappa_{max} = 0.1/H$.

The GND saturation value profiles in Figure 4.4 are also employed in the present study. Figure 4.10a shows the predicted GND density distribution along $x_2$ axis for the deformed strip for $\rho_{sat} = 2 \times 10^{14} \text{m}^{-2}$ and $L/H = 10$ and a distribution profile with wave length $M = 3$. As the GND density approaches the saturation value, patterning can be observed. As GND density accumulates locally, the slip resistance increases and slip occurs in neighboring regions. The effect of the length scale parameter $L$ is shown in Figure 4.10b. As in the constrained shear case, it is observed that for larger $L/H$ ratios, the long range effects become dominant and the dislocation density profile smoothens out.

The random profile of the saturation value in Figure 4.7 is also used for the bending problem. In Figure 4.11, for different saturation base values, the profile of GND density on slip system 1 along $x_2$ axis is shown. It can be observed that, in regions of smaller saturation values more irregular the profile arise since slip resistance in those regions increase and localization occurs. As the reference saturation value is increased the irregularity in patterning starts decreasing.

In Figure 4.12 normalized bending moment versus normalized curvature is shown for $\rho_{sat} = 2 \times 10^{14} \text{m}^{-2}$ and $L/H = 10$. The initially imposed sinusoidal GND density profile in Figure 4.4, results in higher hardening rates compared to the randomly varying profile in Figure 4.7. Although there was a slight difference for the constrained shear case, the mechanical response for sinusoidal profile $M = 3$ and $M = 4$ are exactly the same in the bending problem. This is because of the characteristic of the problem. In pure bending, without any imperfection the dislocation density will be the same along the $x_2$ axis, whereas in the constrained shear case there is a linear variation. An imposed imperfection on the profile will affect the varying distribution more than a constant one.
Figure 4.10. a) Evolution of $\rho_{GND}^{(1)}$ along $x_2$ axis with $M_{GND}$ as shown in Figure 4.4 and $\rho_{GND}^{sat}$ values $2 \times 10^{14}$ m$^{-2}$ b) Effect of $L$ on the evolution of GND densities for $\rho_{GND}^{sat}$ values $2 \times 10^{14}$ m$^{-2}$ for maximum curvature $\kappa_{max}$

Figure 4.11. Variation of $\rho_{GND}^{(1)}$ along $x_2$ axis with $M_{GND}$ as shown in Figure 4.7 and for different $\rho_{GND}^{sat}$ values $2 \times 10^{14}$ m$^{-2}$ at $\Gamma_{max}$ deformation.
Figure 4.12. Normalized Moment vs Normalized curvature for Taylor Type of hardening and different hardening profiles: Sinusoidal for $M = 3$ and $M = 4$ (c.f Figure 4.4), the randomly varying profile (c.f Figure 4.7) ($\rho_{\text{sat}} = 2 \times 10^{14} \text{m}^{-2}$ and $L/H = 10$)

4.6. Conclusion

In this work, a non-work conjugate type of framework proposed by Kuroda-Tvergaard [38] is coupled with a new hardening rule to analyze the GND patterning behavior as a result of the plastic deformation. The new hardening rule is an extended form of Taylor type of hardening, and it is motivated by spatial variations of GND density as observed in the experimental results in Kysar et al. [1]. The experiments give insights about the mechanism of GND density accumulation. It is observed that GND density accumulates quasi-periodically and dislocations form cell walls. These cell walls can be considered as imperfections as the plastic deformation. In this paper, the imperfections are introduced as variation on saturation value of the GND densities by imposing different profiles for GND density variation with different saturation values in the constitutive relations.

Before imperfections are introduced, at first, the model is calibrated with the material length scale parameter, $L$, in the back stress formulation. It is observed that for smaller $L/H$ ratios, at the locations where the plastic strain is maximum and the
gradient approaches zero, the vertex-type of localization occurs. In the general slip rate equation, the gradient of the plastic slip appears as GND density and the gradient of the GND densities is included in back stress formulation. The contribution of the gradient of GND densities is controlled by the length scale parameter, $L$, and it has a regularizing effect on the results. As $L/H$ is increases, a regularization in GND density profile is observed and the vertex vanishes.

After analyzing the regularizing effect of $L$, imperfections have been introduced and tested on the constrained shear and the pure bending problems. The imperfections are introduced by defining different profiles for GND density variation with different saturation values. First a sinusoidal distribution along the $x_2$ axis is defined with different amplitude values. The amplitude of the sinusoidal functions can be interpreted as the maximum saturation value of GND densities. It is observed that for smaller saturation values, the patterning is more severe. As the dislocation density locally reaches the saturation value, the slip resistance becomes higher, and slip occurs in neighboring regions.

The wavelength of the sinusoidal function is changed and also a random distribution for the initial saturation value is tested. It is observed that the patterning in GND density profile differs depending on the initially defined GND density saturation profile. The overall mechanical response is also affected by the variation in GND density profile. More complex patterns yield higher hardening rates.

In this paper, it is observed that the initially defined profile and the saturation GND density value affects the final patterning. In reality as the dislocations accumulate in certain regions, the mobility of dislocation will decrease and the slip resistance will increase, so this is consistent with the foundation of the model. In this study, the interaction between dislocations on other slip systems is not considered, which would contribute to the complex patterning in the regions where there is multiple slip. While the prescribed GND saturation profile is phenomenological, if a physical
distribution can be extracted from further experimental analyze, one can input this to get more complicated patterning. However, future studies will concentrate on developing constitutive formulation for which these spatial variations in GND density naturally evolve.
CHAPTER 5

Effect of Dislocation Mobility on GND Density Structures

5.1. Abstract

This chapter is concerned with the geometrically necessary dislocation density (GND) measurements in tantalum single crystal samples at room temperature (∼ 293°K) and cryogenic temperature (liquid nitrogen temperature (∼ 77°K)). The tantalum crystal is deformed quasi-statically with a wedge indenter using the same method outlined in the Chapter 1 and Chapter 2. Different than face-centered-cubic crystal (FCC) nickel, tantalum has body-centered-cubic (BCC) crystal structure. In order to obtain plane deformation condition, the sample is rotated one quarter turn about [110] from that of face-centered cubic crystal (FCC) structure, the specimen has the same three effective slip systems with precisely the same orientations as the FCC case are activated. The measurements are performed with both traditional EBSD and HR-EBSD, however in this thesis only the results from traditional EBSD results with 2 µm spatial resolutions are presented. The EBSD results show significant differences in lattice rotations and striking evidences of twin formations at low temperatures. The dislocation structures such as quasi-periodic cell walls are easily recognized in the samples deformed at the room temperature, however they are observed to be discontinuous and accompanied by twins at low temperatures. The dislocation densities are reported as $5 \times 10^{14}$ m$^{-2}$.

5.2. Introduction

Tantalum has been widely used in electronic components mainly in capacitors, however due to it is superior refractory and mechanical properties it has started to be
used in defense and space research applications [89]. The deformation characteristic of tantalum under different conditions has been studied extensively for both single and polycrystalline materials as outlined in [90]. However the competition between dislocation slip and mechanical twinning in tantalum is not well understood yet. The conditions of twin formations are observed to evolve at low temperatures, at different loading rates or at both. Recent EBSD measurements on single crystal tantalum deformed under shock loading of 55 GPa shows heavy twinning formations in the the material [90]. The twins occur on 3 – 4 twin systems in the regions where primary Schmid factor is low, which indicates a scarcity of slip systems for dislocation motions. Another test conducted on single crystal tantalum specimens has shown twinning occurs at cryogenic temperatures under compression tests [91]. In addition, polycrystalline samples deformed under high rate of compressive loading conditions infrequently showed twinning formations [92].

The motion of dislocations is driven by the resolved shear stress on the slip system, $\tau$, and thermal activation and it is impeded by the lattice resistance which can be expressed with Peierls stress [14]. If the strain rate is kept constant, the Peierls stress will increase with a decrease in temperature especially in BCC materials. If the increase in lattice resistance is coupled with a low Schmid factor, twinning formation is observed. The dislocation density accumulation and twin formation occur simultaneously during the deformation on different part of the crystal. At high loading rates twin formations occur due to limited mobility of dislocations as well. Twin formations will also observed when the dislocation densities reaches certain value and dislocation spacing is limited [14].

In this chapter, we present GND density measurements —using traditional EBSD method— of a tantalum single crystal sample that is deformed with an wedge indenter quasi-statically at cryogenic temperature and room temperature. In the first section, the sample preparation and the experimental procedures are explained. In the second
Figure 5.1. Experimental set-up and the Ta single crystal sample

part, results and a brief analysis are presented. In the final part recommendations and some details on future work is given.

5.3. Experimental Procedure

Two samples are cut from a tantalum single crystal rod with 99.998% purity. Laue X-Ray diffraction was used to orient the sample in the desired orientation to within one degree accuracy. The rod was cut into square prisms $10 \times 5 \times 5$ mm cross section, using a wire electrical discharge machine (EDM) such that out of plane normal of the larger surface is (110) as shown in Fig. 5.1 The specimens were mechanically polished using the skim cut (fine cutting) option in wire EDM, followed by ultrasonic cleaning to remove any adhered particles. The first sample is tested in a styrofoam container filled with liquid $N_2 (77^o K)$, whereas the second sample is tested at room temperature. The wedge indenter, made of tungsten carbide with an angle of $90^o$, penetrated into the material along the $[\bar{1}10]$ crystallographic direction. The indenter surface is polished carefully to minimize the frictional effects.

After the indentation, samples are cut from a (110) midsection plane, in order to determine the in-plane deformation condition. The surface that is exposed with the wire EDM is polished prior to EBSD. Sample preparation for EBSD is more tedious than it is for nickel. Tantalum samples are first mechanically polished with EDM skin
cutting procedure and then ultrasonically cleaned to remove the adhered particles. Further mechanical polishing is performed with diamond paste starting from 6 µm to 1/2 µm. The samples are ultrasonically cleaned at intermediate steps. Mechanically polished samples are etched in 50% $H_2SO_4$, 25% $HNO_3$, 25% $HF$ volumetric solution at ambient temperature. The mechanical polishing and subsequent etching process is repeated as required. The results presented in this paper are obtained by repeating these processes twice.

The samples are scanned using HKL Channel 5 EBSD system, which is a traditional EBSD system with an angular resolution of ±0.5°. The patterns are processed online using the Salsa data acquisition and indexing software. This software provides Euler angles which are later processed to obtain the lattice rotations.

5.4. Results and Discussion

The lattice maps of the indented samples are shown in Fig. 5.2. The lattice rotations on the both samples are in the order of 25°, however the distribution of lattice rotations are different. In the sample deformed at 77°K, the rotations occur mainly in a smaller region. One of the most striking features in lattice rotation map of the low temperature sample, is the region that makes an angle of $\sim 125.4°$ with the horizontal axis, which is normal to slip system 1. This is indication of kink type shear on slip system 1 at that region that goes through the tip of the indented region. The lattice activity on the sample indented at room temperature is more than it is in the other sample.

The lower-bound GND densities are calculated based on the method outlined in Chapter 2. The total GND densities are shown in Fig. 5.2. The GND density distribution differs in both sample. In sample deformed at room temperature, the mobility of dislocations is higher and slip occurs on three effective in-plane slip system as expected. In Fig. 5.2a, quasi-periodic continuos cell walls are observed in the lower
part of the plot. This patterning is similar to the one observed in nickel crystals. The total GND density on the sample tested at \(77^\circ K\), attains higher values on the left of the anti-symmetry region. There are two possible reasons for this case: due to chemical etching the material in higher dislocation density region is etched more and later they are misindexed; or there are other deformation structures such as twinning. The Kikuchi patterns collected from this region are quite sharp and there are adequate number of bands to index, so the later reason is more probable. The mobility of dislocations depends on the temperature and the resistance on the slip system which depends on both the temperature and the applied resolved stress. At lower temperatures the resistance on the slip systems increases such that dislocation slip is inhibited.

On the lower part of the Fig. 5.2c, it is observed that the dislocation cell walls are interacting with other deformation structures. These are considered as twin formations. The lower bound method outlined in Section 2.3 does not consider twin formations. However the method identifies these structures as high dislocation density cell walls.

The dislocation densities on each slip system are resolved and they are shown in the Fig. 5.3. The dislocation density distributions on slip system 1 show similarities in both samples. On the kink shear wall, which is also observed in lattice rotation maps, crystallographic traces are perpendicular to the orientation of the wall. The twin formations are also observed as positive and negative dislocation densities. In Fig. 5.3b, the dislocation cell walls at the same region make different angle with the crystallographic traces and densities are dispersed over wider region than it is in Fig. 5.3a. The similarities are also observed on other slip systems.
Figure 5.2. Lattice Rotations on Tantalum sample indented at a) 77° K b) 293° K
Figure 5.3. GND Density distributions for three slip systems for both samples.
5.5. Conclusion and Recommendations

The dislocation density measurements are performed on the two samples at different temperatures. Both lattice rotations and GND density distributions have striking differences which have not been reported before. The dislocation densities are concentrated on narrower regions in the low temperature sample than it is in the room temperature one. The decrease in dislocation mobility due to the temperature inhibited dislocation activation so that other deformation mechanisms, mainly twinning could be activated. Further analysis should be performed on this data set, and also HR-EBSD data should be analyzed. Here there is a list of analyses that can be performed using this data.

- The twinning mechanism can be incorporated to the lower bound solution; geometrically necessary twins should be identified and their interaction with dislocation can be analyzed.
- Length scale analysis presented in Chapter 2 can be repeated both of samples and the relationship between cell sizes and the dislocation spacing can be identified.
- The twin systems on Ta should be identified and corroborated with the experimental measurements.
- The regions where twinning occurs should be scanned with using high spatial resolutions.
- High temperature experiments can be done to have a complete understanding on temperature effects microstructural evolution.
CHAPTER 6

Conclusion

The conclusions and recommendations from all experimental and numerical studies are provided within each chapter. In this part we outline the important conclusions derived from all of these studies to emphasize the novelty of the work.

In the first chapter, we presented a new approach to investigate the multi-scale characteristics of the plastic deformation. The approach is unique in the sense that it exploits a continuum based concept, GND density, to investigate a discrete problem. The GND density measurements from HR-EBSD show evolution of characteristic material length scales over microscopic to macroscopic scale. The distribution of length scales extracted from HR-EBSD measurements is corroborated with earlier findings from TEM based methods. The peak; dislocation spacing in the cell walls and the dislocation cell sizes are found to be linearly varying and they follow the Holt’s relation as shown in earlier TEM based studies. HR-EBSD, being less restrictive with regard to the size of the area interest on the sample, is advantageous over TEM. High dislocation densities and wide cell walls the order of few micrometers which are not accessible with earlier TEM measurements can be easily extracted from HR-EBSD method. We used the lower bound GND density solution and coupled it with a new quantity, $\beta$, to find the dominantly single slip regions where the lower bound solution becomes exact. The material length scales are extracted for each individual slip system. Although the distribution of length scales for each slip system are fitted to Holt’s relation, the slope of the linear Holt’s relation, $K$, differs for each slip system. The measurements not only provided quantitative distribution but also helped us to understand the evolution mechanism of the dislocations. Dislocation motions follow
sort of a self-organization mechanism to go for a low energy state. In the regions with low dislocation densities, dislocations accumulate and form wide cell walls. As the dislocation density increases, dislocations rearranges and collapse to form narrower cell walls. The further analysis on dislocation walls can be done using the same data set. The relationship between the dislocation densities and the dislocation cell walls can be extracted, which will be a valuable input to material models incorporating GND density evolution mechanisms. More HR-EBSD data can be taken on the regions where there is dominantly single effective slip system is active. HR-TEM analysis should be employed to explore the dislocation structures in the cell walls of sizes ranging 20nm to 1 \( \mu \)m to develop a comprehensive understanding of dislocation evolution mechanism.

The HR-EBSD measurements provide rich data sets that can be utilized to resolve the complexity in the GND densities on each slip system and to understand their interactions. In Chapter 2, a detailed analysis on extracting length scales and resolving complexities is presented, and in Chapter 3, experimental details are presented. A detailed net Burgers vector density analysis presented in this chapter, shows the activation of the slip systems and their interactions. The regions where the net Burger Vector Density align with the crystallographic traces are shown; in these regions where the slip traces are perpendicular to cell walls, kink type of shear is detected. These regions are resolved better with higher resolutions 50nm spatial resolutions. The effect of spatial resolution is also examined and the appropriate size of net Burgers vector is discussed. The effect of spatial resolution is presented based on 50 nm measurements. It is observed that spatial resolution has significant influence in resolving GND densities. In general as the step size increases, uncertainty increases and it becomes difficult to quantify low dislocation densities. Uncertainty analysis showed that HR-EBSD method provides adequate accuracy to extract length scales. It is shown that for 50 nm spatial resolution measurements, the uncertainty is of the
order of $10^{13} \text{ m}^{-2}$. The HR-EBSD measurements provide superior accuracy, which is adequate to resolve the physical length scales. The uncertainty in traditional EBSD is in the range of physically possible length scales which makes length scale analysis not reliable.

In the work presented in Chapter 4, a non-work conjugate type of framework proposed by Kuroda-Tvergaard [38] is coupled with a new hardening rule to analyze the GND patterning behavior as a result of the plastic deformation. The new hardening rule is an extended form of Taylor type of hardening, and it is motivated by spatial variations of GND density as observed in the experimental results in Kysar et al. [1]. The experiments give insights about the mechanism of GND density accumulation. It is observed that GND density accumulates quasi-periodically and dislocations form cell walls. These cell walls can be considered as imperfections as the plastic deformation. In this paper, the imperfections are introduced as variation on saturation value of the GND densities by imposing different profiles for GND density variation with different saturation values in the constitutive relations.

Before imperfections are introduced, the model is calibrated with the material length scale parameter, $L$, in the back stress formulation. It is observed that for smaller $L/H$ ratios, at the locations where the plastic strain is maximum and the gradient approaches zero, the vertex-type of localization occurs. In the general slip rate equation, the gradient of the plastic slip appears as GND density and the gradient of the GND densities is included in back stress formulation. The contribution of the gradient of GND densities is controlled by the length scale parameter, $L$, and it has a regularizing effect on the results. As $L/H$ is increased, a regularization in GND density profile is observed and the vertex vanishes.

After analyzing the regularizing effect of $L$, imperfections have been introduced and tested on the constrained shear and the pure bending problems. The imperfections are introduced by defining different profiles for GND density variation with
different saturation values. First a sinusoidal distribution along the $x_2$ axis is defined with different amplitude values. The amplitude of the sinusoidal functions can be interpreted as the maximum saturation value of GND densities. It is observed that for smaller saturation values, the patterning is more severe. As the dislocation density locally reaches the saturation value, the slip resistance becomes higher, and slip occurs in neighboring regions.

The wavelength of the sinusoidal function is changed and also a random distribution for the initial saturation value is tested. It is observed that the patterning in GND density profile differs depending on the initially defined GND density saturation profile. The overall mechanical response is also affected by the variation in GND density profile. More complex patterns yield higher hardening rates.

In this numerical study, it is observed that the initially defined profile and the saturation GND density value affects the final patterning. In reality as the dislocations accumulate in certain regions, the mobility of dislocation will decrease and the slip resistance will increase, so this is consistent with the foundation of the model. In this study, the interaction between dislocations on other slip systems is not considered, which would contribute to the complex patterning in the regions where there is multiple slip. While the prescribed GND saturation profile is phenomenological, if a physical distribution can be extracted from further experimental analyze, one can input this to get more complicated patterning. However, future studies will concentrate on developing constitutive formulation for which these spatial variations in GND density naturally evolve.

In Chapter 5, the dislocation density measurements are performed on the two tantalum samples at different temperatures. Both lattice rotations and GND density distributions have striking differences which have not been reported before. The dislocation densities are concentrated in narrower regions in the low temperature sample than it is in the room temperature one. The decrease in dislocation mobility due the
temperature caused activation of other deformation mechanisms, mainly twinning. Further analysis should be performed on this data set also HR-EBSD data should be analyzed.

- The twinning mechanism can be incorporated to the lower bound solution; geometrically necessary twins should be identified and their interaction with dislocation can be analyzed.
- Length scale analysis presented in Chapter 2 should be repeated on both samples and the relationship between cell sizes and the dislocation spacing can be identified.
- The twin systems on Ta should be identified and corroborated with the experimental measurements.
- The regions where twinning occurs should be scanned with using small step sizes.
- High temperature experiments should be done to have a complete understanding on temperature effects microstructural evolution.

In summary, this dissertation focuses on multiscale material characterization over different length scales and it is extended to different deformation conditions. The experimental technique developed in this study is unique in the way that it exploits a continuum concept to analyze a discrete problem: dislocation densities. The method, enables measurement of new quantities related to deformation, such as net Burger vector densities and $\beta$, and it quantifies a distribution of length scales from a multiscale perspective. The numerical part of the thesis, exploits the experimental observations and propose a new method to see the effect of periodic variations in dislocation density evolution and overall mechanical response. The measurements on Ta unravel the competition of different deformation mechanisms at low temperature. The experimental measurements are unique, since similar type of measurements have not been
done neither on tantalum at any temperature, nor on any other material deformed under cryogenic temperatures.
Bibliography


[32] Jeffrey W. Kysar, Yong X. Gan, Timothy L. Morse, Xi Chen, and Milton E. Jones. High strain gradient plasticity associated with wedge indentation into face-centered cubic single crystals: Geometrically necessary dislocation densities.


