STUDIES ON THE MEASUREMENT AND MODELING
OF LATTICE STRAINS IN ROLLED ZIRCALOY-2

by

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Abstract

Neutron diffraction is a widely used technique for measuring internal stresses inside polycrystalline materials. By examining the diffraction patterns collected during *in situ* uniaxial deformation, the lattice strains along various crystallographic directions can be calculated. These lattice strains give insight into the active deformation mechanisms active within the material during plastic deformation. This is most commonly done by fitting model results to the experimentally measured lattice strains through an iterative process of refining the model parameters.

A numerical optimization technique was successfully applied to the problem of refining the input parameters of an elastoplastic self-consistent (EPSC) model. The results were found to be comparable to those obtained by a past researcher manually refining the model parameters and subjectively judging the fit to the experimental data. The numerical optimization method was able to reach an acceptable result much faster than is possible by a human being (days as opposed to weeks or months), meaning that it has the potential to reduce the turn-around time from data collection to interpretation/publication significantly.
At the same time, common experimental techniques for conducting diffraction experiments during uniaxial deformation tests were examined. It is common to use an interrupted loading scheme where the sample is brought to a certain loading condition and then held steady while the neutron data is collected, a process that often takes several minutes. This interrupted loading may be done such that the sample is held at constant stress, strain, or simply by having the load frame stay in a constant position. Each of these different loading modes results in a particular type of relaxation within the sample as it is being held, so a series of experiments were conducted to investigate any impact these different relaxation types may have on the measured values of the lattice strains. Overall it was found that both qualitative and quantitative differences in the recorded data can arise as a result of the different loading modes, and that such differences tend to manifest themselves at or near the point at which the material begins to yield macroscopically.
Statement of Co-Authorship

This thesis is my original work, however, the content of Chapter 3 is based on a co-authored paper published in the Journal of Applied Crystallography [1]. The other authors who contributed to the work were Charles Mareau, who developed the Genetic Algorithm code, and my supervisor Mark Daymond. All experimental data in Chapter 3 was collected by Feng Xu during a previous study [2].

Likewise, the contents of Chapter 4 are based on a paper currently under review for publication in Materials Science & Engineering A; authored by myself, Bjorn Clausen, and Mark Daymond. The experiments themselves were conducted by Bjorn and I. The analysis and writing were done by me, with guidance from Mark, who originally proposed the experiment.

The contents of Chapter 5 are planned for publication at some time in the future and the analysis and writing contained in it (along with all other sections in this thesis) were performed by myself under the supervision of Mark Daymond.
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Glossary

\(a\) Twin hardening parameter used by EVPSC.

\(\text{bas}\) Basal slip.

BCC Body Centered Cubic.

\(b^g\) Burgers vector of dislocations in the \(g\)th slip system.

CANDU Canada Deuterium Uranium pressurized heavy water reactor.

\(\chi\) Material constant used in the calculation of \(\tau^g_{\text{dis}}\).

Comp XD/YD Compression along XD (where \(X=\text{N}, \text{R}, \text{or T}\)).

Crossover One of two operations used by the GA to create the next generation of individuals. Parameter values for the new individual is taken to be the weighted average of the values of two individuals from the previous generation. The weighting factors used are a random number between 0 and 1 (\(\alpha\)) and (1-\(\alpha\)).

CRSS Critically Resolved Shear Stress.

\(d_{\text{h}k\text{i}l}\) Lattice plane spacing in grain family \(hkil\) measured during deformation.

\(\dot{\rho}\) Change in dislocation density with respect to time.

\(\dot{\gamma}\) Plastic strain rate.

\(d^\text{ref}_{\text{h}k\text{i}l}\) Lattice plane spacing in grain family \(hkil\) measured prior to deformation.

Elastic Anisotropy Variation of elastic properties with crystallographic direction.

EPSC Elasto-Plastic Self-Consistent model.

\(\epsilon_{\text{h}k\text{i}l}\) Lattice strain in grain family \(hkil\).
**EVPSC** Elasto-Visco-Plastic Self-Consistent model.

*f*  Twin volume fraction used in EVPSC calculations.

**FCC** Face Centred Cubic.

**GA** Genetic Algorithm.

**Γ** Accumulated plastic shear.

**Generation** A group of individuals that have been created by the GA, each sent to a separate instance of the model so they can be tested in parallel. The individuals in a generation each have their modeling results compared to experimental data, and the best performing individuals are used to create a new generation of individuals.

**Grain Family** Grain orientations with a particular hkil aligned within a few degrees of a nominal direction in the sample.

**h** Matrix describing the interactions between different slip systems.

**Hardening** The change in CRSS with respect to accumulated shear.

**HCP** Hexagonal Close Packed.

**HEM** Homogeneous Effective Medium.

**h^{s'/s}** Coupling coefficients for determining the hardening effects that deformation mode s' has on mode s.

**In-situ test** A mechanical test performed in the spectrometer for the purposes of neutron diffraction measurements during deformation.

**Individual** A set of model input parameters selected by the GA and sent to the model for evaluation.

**k_1^g and k_2^g** Material parameters used in the calculation of dislocation generation.

**k_{hp}** Hall-Petch parameter.

**k_{lat}^{tw} and k_{self}^{tw}** Twin hardening parameters used by EVPSC.

**L** Directional mean free path of dislocations.

**Lankford Coefficient** Ratio of the strain in the sample measured along the two Poisson directions after deformation. By convention the Lankford Coefficient is always greater than or equal to unity.
**Macroscopic Flow Curve**  A plot of macroscopic stress versus macroscopic strain. (Also known as a stress-strain curve).

**Micro-yielding**  When the applied load causes the CRSS to be exceeded for grains with certain orientations, causing them to deform plastically before any macroscopic plasticity is observed.

**MRD**  Multiples of Random Distribution.

**µ**  Effective shear modulus.

**Mutation**  One of two operations used by the GA to create the next generation of individuals. A parameter value for the new individual has a probability equal to $P_{mut}$ of being randomly selected from between the upper and lower bounds specified by the user.

**ND**  Normal Direction.

**$N_{gen}$**  Number of generations used by the genetic algorithm during parameter selection.

**$N_{hri}$**  Number of highest ranked individuals used by the genetic algorithm to create the next generation.

**$N_{ind}$**  Number of individuals used by the genetic algorithm during parameter selection.

**NRU**  National Research Universal Reactor.

**Plastic Anisotropy**  Variation in plastic deformation properties of a material with crystallographic direction.

**$P_{mut}$**  Probability for mutation to occur in an individual during genetic algorithm parameter selection.

**Poisson Direction**  One of the two principle directions that are perpendicular to the direction of loading.

**pr**  Prism slip.

**pyr**  Pyramidal slip.

**RD**  Rolling Direction.

**$\rho$**  Total dislocation density.
\(\tau_0^s\) Initial CRSS for deformation mode \(s\).
\(\tau_0^s + \tau_1^s\) Final back-extrapolated CRSS for deformation mode \(s\).
\(\tau_c^g\) Critically resolve shear stress of deformation mode \(g\).
\(\tau_{dis}^g\) Contribution of dislocation hardening to CRSS.
\(\tau_{hp}^g\) Contribution of Hall-Petch hardening to CRSS.
\(\tau^s\) Instantaneous CRSS for deformation mode \(s\).

TD Transverse Direction.

TensXD Tension along XD (where \(X=N,R,\) or \(T\)).

\(\theta_0^s\) Initial hardening rate of deformation mode \(s\).
\(\theta_1^s\) Asymptotic hardening rate of deformation mode \(s\).

\(\theta_{hkil}\) Diffraction angle \(hkil\) measured during deformation.
\(\theta_{hkil}^{ref}\) Diffraction angle \(hkil\) measured prior to deformation.

TOF Time of Flight.

VPSC Visco-Plastic Self-Consistent model.
Chapter 1

Introduction

1.1 Zirconium in the Nuclear Industry

Zirconium and its alloys (such as Zircaloy-2, Zircaloy-4, and Zr-Nb alloys) are of critical importance to the nuclear industry because of their corrosion resistance in water, good mechanical properties, and low neutron absorption cross-section. Zircaloy-2 (the material of primary interest in this thesis) is nominally composed of the following elements: Sn 1.2-1.7wt%, Fe 0.07-0.2wt%, Cr 0.05-0.15wt%, Ni 0.03-0.08wt%, 1400wppm oxygen, Zr (balance) [4]. The Cr, Fe, and Ni are added in order to give improved corrosion resistance to the Zr without compromising the mechanical properties [5]. Zircaloy-4 has a composition similar to that of Zircaloy-2, with one notable exception being the absence of any Ni. The reduced mechanical strength that resulted from the removal of Ni was compensated for by increasing the Fe content to 0.24% giving Zircaloy-4 strength comparable to Zircaloy-2 as well as significantly reduced hydrogen uptake at the cost of a slight reduction in corrosion resistance [5].
Today, Zircaloy-2 and Zircaloy-4 are widely accepted as industry standards for structural components in several types of fission reactors [5].

For the most part, Zircaloy-2 and Zircaloy-4 are processed into tubes for use in reactors. The production techniques, as well as the final size and thickness of the tubes depends on the particular application. For example, tubes used as fuel cladding in Canada Deuterium Uranium Pressurized Heavy Water (CANDU), Boiling Light Water (BLW), and Pressurized Heavy Water (PHW) reactors are formed via a hot extrusion process followed by several steps of cold working. This is required in order to obtain the proper texture, hydride orientation, and mechanical properties for components directly exposed to both fuel and coolant. Tubes used in Calandria tubes in CANDU reactors, however, are generally exposed to only small stresses and are thus generally rolled from sheet material and seam-welded.

Components within the reactor core are subject to not only extreme temperatures, but also damaging neutron radiation fields. This results in deformation via radiation induced creep and growth which can cause major problems if not properly accounted for [6]. Calandria tubes in CANDU reactors, for instance, undergo creep sag which can lead to contact with reactivity mechanisms, as well as axial elongation causing stresses, and ovality which can result in contact between the pressure tube and Calandria tube [6]. For these reasons, among others, understanding the deformation characteristics and mechanisms of Zr alloys is of the utmost importance to guarantee the safe operation of nuclear power reactors.
1.2 Internal Stresses, Deformation Mechanisms, and Texture

The behavior of Zr alloys can be influenced by many different factors. These range from external factors such as the ambient temperature and stresses being applied to that material, to internal factors such as microstructure, texture, alloying elements, and intergranular stresses. Plastic deformation of the material is particularly dependent on these factors, since it is controlled by microscopic deformation mechanisms interacting with each other in various combinations of external loading and temperatures.

Deformation modes in hexagonal close packed (HCP) materials are more restricted than those found in cubic materials. While FCC materials have 12 slip systems, and BCC materials have 48, in HCP materials similar to Zr (i.e. those with a c/a ratio less than 1.633) there are only 3 primary slip systems. Because of this, Zr alloys require the activation of either slip systems on other planes or twinning in order for there to be the 5 independent deformation modes needed for a single crystal to support an arbitrary plastic strain. Twinning and slip on these non-primary slip planes are more difficult to activate than the primary slip modes, which leads to significant levels of plastic anisotropy in the material. This plastic anisotropy, when combined with the elastic and thermal anisotropy also present in the material, drives the development of textures and intergranular stresses, which in turn can place limits on which deformation modes become active (or inactive) under external loading conditions. Thus,
deformation modes, intergranular stresses, and crystallographic texture are all dependent upon one another.

1.3 Summary of the Material

The material studied in this work is a 70mm thick warm-rolled, recrystallized slab of Zircaloy-2. The composition is as follows: 1.43-1.45 wt% Sn, 0.13-0.14 wt% Fe, 0.1 wt% Cr, 0.05 wt% Ni, 1260-1440 wt ppm O, and 150-160 wt ppm C; with the balance of the material being Zr. The processing route of the material consisted of the following steps: (1) forging at 1289K to 5-5/8” x 9-1/4” x 26”, (2) β quench in water from 1289K, (3) condition to 5-1/4” x 9” x 26”, (4) preheat to 700K (maximum) and roll along the length of the material to obtain 50% reduction, (5) condition to 2-1/2” x 9” x length, (6) saw to final length. Transmission electron studies showed that the material had been fully recrystallized [7], indicating that a temperature of at least 900K had been reached during processing, which is believed to be the result of adiabatic heating. The grains are equi-axed and have an average grain size of approximately 20µm. The texture of the material is typical of rolled material that has been recrystallized, with most {0002} poles oriented towards the normal direction (ND) with a spread of ±50° towards the transverse direction (TD) and a smaller spread of ±30° towards the rolling direction (RD). The {1010} poles are concentrated in orientations of ±30° away from RD in the RD-TD plane, and the {1120} normals are aligned in RD. The variation in texture throughout the sample has previously been determined, showing that there is major variation through the thickness of the slab, but that the texture is fairly uniform through the width of the
slab at mid-thickness [8]. Therefore, any mechanical testing specimens should be cut from the slab such that their gauge regions are centered at mid-thickness, but may be cut from varying locations along the width of the slab.

1.4 This Thesis

1.4.1 Motivation

Studying the mechanical properties and deformation behavior of Zircaloy-2 is of use to the field of materials science in general. It is a good candidate for studying HCP materials since it is easily deformable at room temperature, has a manageable number of active deformation modes, and requires no special safety precautions during handling or machining. On a more practical level, the properties of this material are of particular interest to the nuclear power industry. Understanding the deformation behavior of Zircaloy-2 is important not only for the manufacturing processes involved in the creation of structural components in nuclear reactors, but also in predicting the in-service properties of those components (irradiation creep and growth, for example).

In addition to the value in gaining understanding of the material itself, it is also important to examine the methods and tools used to conduct these studies. This is the more immediate motivation behind the current research: examining ways to improve the efficiency and accuracy of both experimental and modeling-based approaches to characterizing the physical properties of Zircaloy-2, and by extension all metals and alloys.
1.4.2 Objectives

The research presented in this thesis has two main objectives. The first is to attempt to improve the efficiency and accuracy of using diffraction studies to examine the lattice strain evolution of metals during uniaxial deformation. This goal is pursued using two different approaches; first, through the application of a numerical technique with the potential to greatly speed up and automate the process of fitting model results to experimental diffraction data, and secondly through the investigation of how variation in the loading conditions imposed during deformation can affect lattice strain results. The specifics of each of these projects will be discussed in Chapters 3 and 4, respectively.

The second objective of this thesis is to investigate the mechanical response of Zircaloy-2 under various loading schemes during uniaxial compression. This is accomplished through \emph{in situ} neutron diffraction experiments used to collect lattice strain data as well as some limited observations on texture evolution. Additionally, self-consistent modeling of the material was carried out using the loading conditions imposed during the experiments in an attempt to determine if the different loading modes typically used during neutron diffraction studies have any affect on the deformation behavior of the material. This work is addressed in Chapters 4 and 5.

1.4.3 Guide to the Thesis

This thesis is presented in a manuscript form. Chapter 2 presents an overview of the published literature relevant to the topics studied throughout the rest of the thesis.
Chapters 3 through 5 are individual manuscripts which each cover self-contained, but related research that contributes to fulfilling the overall objectives of the thesis. The following is a brief description of the work presented in those chapters.

Chapter 3 outlines the work done in applying a numerical optimization technique known as a genetic algorithm to the problem of selecting the hardening parameters to be used by an elasto-plastic self-consistent model during simulation of Zircaloy-2 under tension and compression. A single set of hardening parameters is selected by the algorithm in order to model uniaxial deformation along all three material directions in both tension and compression, and the results are compared to an existing large experimental dataset [9]. Experimentally measured macroscopic flow curves (i.e. stress-strain curves), lattice strains, and Lankford coefficients are all compared to results generated by the model. The main question explored in this research is whether or not a numerical optimization technique can select hardening parameters that result in a fit comparable to that obtained by the labor-intensive process of having a human manually select the parameters through trial and error, as was recently done by Xu [10].

Chapter 4 investigates the effects that using different loading modes can have on lattice strain data obtained via neutron diffraction. Samples of Zircaloy-2 were compressed uniaxially using commonly applied loading techniques. Since neutron scattering facilities tend to require several minutes to tens of minutes to capture a single spectrum (or one data point on a lattice strain curve), in situ tests at such facilities are typically carried out using incremental loading with the samples held at
either constant stress, constant strain, or with the actuator position of the compres-
sion rig held constant. Each of these loading schemes causes the sample to experience
a unique path of loading and relaxation, and the effects of these different loading
modes on the lattice strain data is the primary topic of concern, although the affects
on twinning and texture evolution are also touched upon.

Chapter 5 serves as a bridge of sorts between Chapters 3 and 4. The optimization
technique discussed in Chapter 3 is applied in order to fit self-consistent models to
the data collected during the experiments presented in Chapter 4. This allows for
further insight into the causes behind some of the phenomena observed during the
experiments, as well as a comparison between the application of time-dependent and
time-independent models to lattice strain observations. This comparison is important
because although there can exist significant amounts of time dependent sample relax-
atation during neutron diffraction, until recently the models most commonly applied to
predict the results of these experiments have been time-independent.

Finally, Chapter 6 offers some general conclusions on the thesis as a whole, with
an emphasis on the extent to which the objectives of the thesis have been met. Addi-
tionally, some suggestions are made with regards to what work could be done in the
future to expand and improve upon the contributions made by this research.
Chapter 2

Literature Review

Most zirconium alloys used in engineering applications are polycrystalline. Often times, during manufacturing of these alloys, a preferred orientation of crystals begins to form. The interaction of the grains making up these polycrystalline materials results in inter granular (type-2) stresses [11]. In this chapter, the deformation mechanisms for single-crystal and polycrystalline zirconium alloys will be reviewed, as will the technique of measuring such strains using neutron diffraction. The modeling of internal strains will also be discussed, with particular attention paid to self-consistent methods.

2.1 Properties of Single Crystal Zirconium

At room temperature, pure zirconium has a hexagonal close packed (HCP) structure, as shown in Figure 2.1. This gives single crystal zirconium different physical properties (elastic, plastic, thermal) along the $a$ and $c$ axes. These anisotropic single crystal
properties, combined with the material’s texture, are what cause the anisotropy observed in the macroscopic properties of polycrystalline zirconium.

Figure 2.1: Diagram of an HCP unit cell.

The thermal expansion coefficients of zirconium have been reported to lie somewhere in the range of $5.0 < \alpha_a < 5.7 \times 10^{-6} \text{K}^{-1}$ for the a-axis and $7.5 < \alpha_c < 12 \times 10^{-6} \text{K}^{-1}$ for the c-axis [12]. Room temperature, stress free lattice spacings in the a and c axes are 3.23118 and 5.14634 Angstroms, respectively, with a c/a ratio of 1.59271 [13]. The elastic constants of single crystal zirconium at room temperature are as follows: $C_{11}=143.5$, $C_{22}=143.5$, $C_{33}=164.9$, $C_{44}=C_{55}=32.1$, $C_{66}=35.5$, $C_{12}=C_{21}=72.5$, and $C_{13}=C_{31}=C_{23}=C_{32}=65.4$ (all values are in GPa) [13]. The room temperature Young’s modulus of the material varies with crystallographic direction, from a minimum of 89GPa to a maximum of 125GPa along the c-axis. The a axis has a Young’s modulus of 99MPa.
Plastic deformation in the material is controlled by the activation of various deformation modes (either slip or twinning). These deformation modes are activated by a combination of applied stress and temperature, the higher the temperature of the material, the lower the stress needed to activate a given mode and vice versa. In single crystal α-Zr, the main deformation mode is the slip system on the \( \{10\bar{1}0\} \) plane with a Burgers vector of \( \langle 11\bar{2}0 \rangle \), commonly known as prism \( \langle a \rangle \) slip. Prism slip in single crystals of Zr has been observed at temperatures of 77K, 300K, 575K and 1075K [14, 15, 16]. Regardless of temperature, prism slip remains the most easily activated slip system [17].

The \( \{0001\} \langle 11\bar{2}0 \rangle \) slip system, otherwise known as basal \( \langle a \rangle \) slip, has been observed at temperatures above 850K, in crystals that were oriented in such a way that prism \( \langle a \rangle \) slip was unfavourable [17]. Slip along \( \langle 11\bar{2}3 \rangle \), or \( \langle c + a \rangle \) slip, as been observed in single crystals at high temperatures or under specific constraints, with both \( \{10\bar{1}1\} \) [17] and \( \{11\bar{2}1\} \) [18] reported as the slip plane. Twinning was reported to be active on four different planes in single crystal Zr by Rapperport [15, 16]; the twin planes observed being \( \{11\bar{2}1\} \), \( \{11\bar{2}2\} \), \( \{11\bar{2}3\} \), and \( \{10\bar{1}2\} \). At all test temperatures, ranging from 77 to 1075K, \( \{11\bar{2}1\} \) twins were present; \( \{10\bar{1}2\} \), \( \{11\bar{2}2\} \), and \( \{11\bar{2}3\} \) (listed in decreasing order of importance) also appeared under certain conditions.
2.2 Deformation mechanisms of polycrystalline Zr and its alloys

In polycrystalline Zr and its alloys, the primary deformation mechanism is the same as in single crystal Zr, prism slip \( \{10\overline{1}0\} \langle 11\overline{2}0 \rangle \). At room temperature, deformation along the \( c \)-axis is accommodated by pyramidal \( <c + a> \) slip [19, 20], rather than by the \( \{11\overline{2}2\} \) compression twinning that is observed in single crystals. In fact, during room temperature deformation of polycrystalline Zr, the only type of twinning yet reported has been \( \{10\overline{1}2\} \langle \overline{1}011 \rangle \) tensile twinning.

The evidence for the active slip and twin systems in Zr alloys has been studied and documented in multiple electron microscopy studies [20, 21]. The presence of prism \( <a> \) [22, 23, 24, 18], pyramidal \( <c + a> \) [18, 23, 25], and tensile twinning [15, 22, 26] have all been particularly well reported and documented.

As stress is increased within a polycrystal, the first slip system activated is always prism slip. As more \( <a> \) dislocations accumulate due to rising stress, local stress concentrations are formed where cross-slip becomes possible. This build-up of \( <a> \) dislocations has been pointed out as a likely mechanism for the initiation of basal slip [27]. Cross slip of \( <a> \) dislocations onto basal or pyramidal planes was also observed during cold-rolling of Zr by Phillipe et al [28]. More recently, it has been shown that cross slip on the basal plane gives a better explanation than pyramidal \( <a> \) slip with regards to the observed evolution of lattice strains in Zr [29].
2.3 Internal Stresses

Residual stresses within a material can exist on a variety of length scales and can arise due to causes relating to the material’s plastic, elastic, or thermal properties. It is common practice to divide residual stresses into three categories, determined by the length scale being studied [30]. Type 1 stresses exist on length scales comparable to the dimensions of the macroscopic structure of a component (mm or larger). Type 1 stresses are continuous over the entire structure of the material and generate internal forces and bending moments which are balanced in all cross sections and around all axes, respectively. Type 2 stresses are also known as intergranular stresses and vary over length scales comparable to the grain size of the material. Type 2 stresses are continuous within a single grain but discontinuous at grain boundaries. Type 3 stresses typically operate on a length scale equal to several atomic spacings (i.e. nm scales). They are continuous within a particular sub-grain (regions within a grain which are separated by low angle lattice mismatch) but discontinuous between multiple sub-grains. The work contained in this thesis is concerned mainly with type 2 (intergranular) stresses.

Type 2 stresses may arise in Zirconium and Zirconium alloy polycrystals due to several phenomena. Usually, the stresses are created by some combination of the anisotropic single crystal properties, the crystallographic texture of the bulk, and whatever thermomechanical load the material is subjected to [11]. Cooling an HCP material from high temperature introduces residual intergranular stress caused by the thermal anisotropy of the single crystal, since strain compatibility and stress balance between adjacent grains must be maintained to account for the mismatch in thermal
strains. Thermal stresses can also have an effect on the elasto-plastic behavior of HCP materials, as opposed to materials with cubic symmetry which are thermally isotropic. For example, the thermal stresses can cause the material to exhibit different yield stresses and elasto-plastic transitions in tension from those displayed under compression [31]. Mechanical loading also introduces intergranular stress due to the elastic and plastic anisotropy of the individual grains. When the applied load reaches a level that corresponds to the CRSS of grains oriented most favorably for slip, then these grains will plastically deform (or micro-yield) before there is macroscopic yielding observed in the bulk material. Meanwhile, those grains which have not had their CRSS reached by the applied load will continue to deform in a purely elastic manner [32]. For example, if we consider a sample of material with a completely random texture which is subjected to uniaxial compressive loading, then the plastically “hard” grains (those oriented for slip to occur on systems with a relatively high CRSS) will accumulate very high stresses at the cost of the plastically “soft” grains (those oriented for slip to occur on systems with a low CRSS) which will exhibit yielding. When the sample is unloaded then the plastically “hard” grains will be left with residual compressive stresses, while the plastically “soft” grains will develop residual tensile stress [32].

### 2.4 Neutron Diffraction

Here, the technique of neutron diffraction will be reviewed, using information gathered mainly from Refs. [33, 34, 35].
Neutrons used for the purposes of diffractions studies are known as thermal neutrons, meaning that they have relatively low kinetic energy (as opposed to “fast” neutrons), and have a wavelength of the same order of magnitude of the lattice spacings of crystalline solids. Thermal neutrons are ideal for bulk diffraction studies, due to their ability to penetrate most engineering materials as deeply as several cm. By comparison, X-rays from laboratory sources will typically penetrate only tens of µm, and even more energetic synchrotron X-rays will typically only penetrate a few mm. This deep penetration depth allows for bulk measurements of large samples without any need for destructive sectioning or surface preparation. Not only does this save time in avoiding tedious sample preparation and make in situ tests easy to perform, but it also improves measurement statistics by sampling a large number of grains. Additionally, the behavior measured in the bulk of the material is more physically meaningful than measurements taken only from a thin surface layer since it better represents the actual response of the material; measurements made at the surface (or near-surface) are likely to be influenced by sample preparation, surface roughness, and the relaxation of stresses normal to the surface, among other things. This ability to probe deeply into the bulk of the material has made neutron diffraction a popular method for characterizing crystalline materials.

Neutron diffraction, like all other types of diffraction, is governed by Bragg’s law: \( n\lambda = 2d\sin \theta \), where \( n \) is the order of diffraction (1,2,3,4...), \( \lambda \) is the wavelength, \( d \) is the plane spacing, and \( \theta \) is the diffraction angle. Figure 2.2 shows the Bragg condition from two adjacent lattice planes where \( n = 1 \), and it is easy to see that the path difference between the beams (represented by dotted arrows) is equal to \( 2d\sin \theta \).
The scattering vector, also known as the Q-vector, bisects the incident and diffracted beams, and is the direction along which lattice strains are measured in the material. Note that the angle $\theta$ is not measured directly, but instead the angle measured during experiments is equal to $2\theta$.

![Figure 2.2: Diagram showing the geometry involved in a first order diffraction.](image)

### 2.4.1 Time of Flight Diffraction at SMARTS, LANSCE

The time of flight (TOF) neutron spectrometer known as SMARTS (Spectrometer for Materials Research at Temperature and Stress) uses a “white” beam of neutrons, containing a continuous distribution of wavelengths. The neutrons are generated via spallation and pass through a water moderator to achieve wavelengths useful for diffraction. Basically, the neutrons leave the moderator and enter into a guide section that collimates them into a beam which strikes the sample and is diffracted towards a detector at a specific angle. The time of flight from moderator to detector is directly proportional to the neutron wavelength, which is in turn proportional to the lattice spacing of the diffraction plane in the material. By measuring changes in the time of flight of the neutrons, changes in the lattice spacing of the material (i.e. lattice strain) can be easily calculated. This section summarizes information describing the
The basic operation of SMARTS, originally presented by Bourke et al. [3]

In SMARTS the flight path between the neutron source and the sample is approximately 30.75m, with the neutrons passing from the moderator through a series of scrapers, which act as the first stage of the beam collimation process. Approximately 5 meters from the moderator, the beam enters a neutron guide which extends into the cave and terminates 3m from the sample. Two sets of aperatures located between the end of the beam guide and the sample are used to control the final size and shape of beam that strikes the sample, allowing for a continuous range of cross sectional area between 1 and 100mm$^2$.

The neutrons interact with a certain gauge volume of the sample, determined by beam size and the sample geometry. Different grain orientations within this gauge volume will cause neutrons to be diffracted at different angles, in accordance with Bragg’s law. Two detector banks are placed perpendicular to the incident beam, consisting of a total of 384 single ended $^3$He tubes. Each of these banks cover an angular range of approximately 30°. Using TOF measurement techniques, each of these tubes can measure a spectrum simultaneously, and then the data can be combined to give an average spectrum over the entire angular range. A diagram of the SMARTS cave showing the general layout of the apparatus is shown in Figure 2.3

The lattice strain present in the material is determined by changes in the time of neutron flight, which is related to the changes in lattice spacing and neutron wavelength, i.e. $\epsilon_{hkl} = \frac{d_{hkl} - d_{ref}}{d_{ref}} = \frac{t_{hkl} - t_{ref}}{t_{ref}} = \frac{\lambda_{hkl} - \lambda_{ref}}{\lambda_{ref}}$, where $d$ is the plane spacing, $t$
is the time of flight, and $\lambda$ is the wavelength. The collected neutron counts are then often plotted against d-spacing, $2\theta$, or time of flight. Single peak or Rietvelt fitting techniques can be used to fit the peaks of the data and obtain the lattice spacing and other micro structural information.

In TOF experiments involving *in situ* loading, the loading axis is typically aligned horizontally at a 45° angle to the incident beam. This allows for one detector bank to measure lattice strains along the loading axis, and the other to measure them along one of the two Poisson directions (i.e. the two directions perpendicular to loading). This means that a minimum of two samples are required in order to measure lattice strains in all three sample directions.
2.5 Self-Consistent Modeling

Self-consistent modeling is a technique within the broader scope of polycrystalline models. Since most engineering materials exist as polycrystals, it is important to be able to understand the complex grain interactions that occur within them. Polycrystalline models attempt to do this by representing the polycrystal as a collection of weighted grain orientations. By doing this, they can make predictions about the macroscopic behavior of the material as a whole. These models were developed over time, becoming increasingly robust and complex with advances in both computing power and fundamental understanding of the principles involved.

The Taylor model [36] and the Sachs model [37] (also known as the upper bound model and lower bound model, respectively) were the earliest incarnations of polycrystalline modeling. In the Taylor model, the local strain field is assumed to be uniform throughout the material, and simply equal to the global strain of the material. The global stress of the material is obtained by averaging over the stress states of all grains. This results in a preservation of intergranular compatibility (by definition), but leads to violations of intergranular stress equilibrium. The Sachs model, by contrast, uses a local stress field that is uniform throughout the material and equal to the global stress tensor, while the global strain is obtained by averaging the local strains present in all grains. This results in behavior that is the opposite of the Taylor model; intergranular stress equilibrium is always preserved, but the compatibility condition is violated.

In order to find useful middle ground between the two extremes of the Taylor and Sachs models, self-consistent modeling was created. Based upon the inclusion theory
first put forth by Eshelby [38], the self-consistent model individually considers each grain embedded in a homogeneous effective medium (HEM). The HEM possesses the average properties of the polycrystalline material. Far from the inclusion, the stress and strain fields are completely uniform and have values equal to the global stress and strain [39]. The stress and strain states of each grain are then found by solving separate boundary value problems for each one. This has the effect of ensuring compatibility and stress equilibrium between all grains and the surrounding homogeneous medium, but effects due local interactions between individual grains are lost. As the stress/strain states of the grains are changed, the properties of the HEM are iterated to ensure that they always reflect the average properties of all grains in the polycrystal.

The self-consistent technique gave rise to a new generation of polycrystalline modeling, utilizing fully elastic coupling between the grains and the HEM. These models, developed by Kröner [40] and Budiansky-Wu [41], predict some heterogeneity in the polycrystal, though only in small amounts.

The third generation of polycrystalline models began with the work of Hill [42] and Hutchinson [39]. The coupling used in these models is elasto-plastic, allowing for more realistic heterogeneity to be predicted in the polycrystal than was possible with purely elastic coupling. This generation of models includes the visco-elastic [43], visco-plastic [44], elasto-plastic [45, 46], and elasto-visco-plastic [47] self-consistent models.
2.5.1 Limitations of Self-Consistent Modeling

The elasto-plastic self-consistent (EPSC) and other common implementations of the self-consistent modeling approach read a variety of input files to take in information about the material and the processes it is to be subjected to. For example, single crystal data such as the elastic constants, thermal expansion coefficients, and coefficients describing the active slip and twinning modes in the material (CRSS, hardening parameters, etc). A separate file contains information describing the initial crystallographic texture of the material by specifying a number of discrete orientations. Normally the number of grains included in this texture file is approximately 2000, in an effort to find a balance between accurate representation of the measured texture and computational cost. In addition to the material properties, boundary conditions for the thermomechanical process(es) must be set. In the case of mechanical loading, the boundary conditions may be set in terms of stress or strain. This simply determines whether the material is incremented in set amounts of strain, requiring the corresponding stress response to be calculated, or vice versa. Macroscopic response, such as traditional stress-strain curves, as well as microscopic behavior such as lattice strains (for EPSC) or changes in texture (VPSC) can be calculated. One very useful feature of the self-consistent approach is the ability to calculate the relative activities of the various deformation modes in the material, which are very difficult to determine experimentally.

One of the core assumptions of the self-consistent model is the idea of a homogeneous medium surrounding each of the grains. This assumption implies that all grains in a polycrystal that have the same orientation should behave identically to
one another. In reality, this is unlikely to be true, since the behavior of a grain can be influenced by its neighbors, and it is unlikely that a given grain will be neighbored only by grains of the same orientation.

The lattice strains calculated by the model also contain certain inaccuracies, the major one being based on the discrete texture data used by the model. In typical diffraction experiment, the lattice strain measured for a given diffraction peak is the result of the average response of an entire “family” of grains. In other words, a populations of grains oriented such that they all have a particular \{hkl\} aligned with a nominal direction in the sample (within a few degrees). However, this condition still allows for the grains to have one free axis of rotation, allowing for their overall orientations to differ greatly among one another. For example, if we consider the \{0002\} pole of a grain is aligned in the Z direction, it is still free to have its \{10\overline{1}0\} pole point in any direction perpendicular to Z (ie anywhere in the X-Y plane). If the experimental sample contains a greater variety in the orientations of grains contributing to a given lattice strain than the discrete texture file accounts for, then information will be lost during modeling.

Most implementations of self-consistent models represent hardening effects within the material using an extended Voce hardening equation [48]. The Voce law is a completely empirical relationship and doesn’t actually describe phenomena such as dislocation generation and interaction, but instead offers a simplified numerical relationship meant to emulate the results of such behavior. This approach only functions
well under simple loading conditions, since it cannot account for effects such as mal-
terial relaxation during unloading. A more complex hardening model, based on the
generation, annihilation, and interaction of dislocations associated with each deforma-
tion mode has been implemented in the EVPSC model [47] in an attempt to improve
accuracy and deal with this issue. Both the Voce and dislocation-based hardening
models will be utilized in this research.

EPSC and VPSC have historically both dealt with twinning in a less than ideal
way. The models treat twinning as a slip system that is only capable of operating
in one direction [49]. This simplification does not properly account for the strain
accommodation and stress relaxation that occurs during the true twinning process
in which the orientation of the twin undergoes a rapid change of approximately 90
degrees relative to the “parent” grain in which it was nucleated. Newer versions of the
EPSC model have been created to deal with this limitation and properly represent
the reorientation and relaxation effects brought on by twinning [50].

One final factor that can affect the effectiveness of self-consistent modeling is the
proper selection of input parameters. Regardless of which version of EPSC, VPSC, or
EVPSC is being used, there are a great number of material parameter which must be
defined before the simulation is run. The number of different combinations of input
parameters is extremely large and finding the “correct” set for a given material is
a challenging problem. Tuning the parameters properly to obtain results that agree
with experimental data is further complicated by the inherent inaccuracies of the
model, which may prevent a perfect solution from ever being found in many cases. A
past attempt has been made to find an appropriate set of EPSC modeling parameters for Zircaloy-2 by performing a systematic search of the parameter space and comparing the model results to a large experimental data set [10]. While this solution yielded reasonable results, the time consuming nature of the project lead to the development of an automated numerical approach which will be described later in this thesis.

2.5.2 Finite Element Modeling of Polycrystalline Materials

Since the purpose of polycrystalline models is to generate the overall response of the material by taking the average response of what is typically thousands of grains, it can be useful to combine this type of model with a finite element approach. This has been done in two different ways in the past, either through directly coupling the finite element model into the finite element framework (known as a hybrid approach) [51], or through having the self-consistent model generate a large array of data that the finite element program can use as a ‘look up table’ (typically called a de-coupled approach) [52].

Another common approach using the finite element method involves the use of crystal plasticity models, which are based on modeling dislocation movement directly. This model has an advantage over self-consistent modeling in that it can model the behavior exhibited by sets of interacting grains. In other words, it is not limited by the assumption of a homogeneous medium in the same way that the self-consistent approach is. These combined crystal plasticity and finite element models are therefore capable of describing the heterogeneous distributions of stress and strain within single
grains and between different grains. This method is particularly useful for studying the phenomena of twin nucleation and propagation, due to the fact that twin behavior is extremely sensitive to the local stress/strain fields near and around the twin [53].

2.6 Relevant Studies Of Lattice Strain Development

2.6.1 Lattice Strain Measurements Via Neutron & X-Ray Diffraction

Several studies have been done investigating the development of internal strains in Zr alloys during uniaxial deformation in both tension and compression, as well as during various heating and cooling processes (for the purposes of studying residual thermal strains). For example, the lattice strains of Zircaloy-2 specimens that have been formed through swaging and subsequently recrystallized have been measured both during \textit{in situ} uniaxial deformation tests [31, 32], as well as during controlled cooling from 900K to 300K [54]. Samples of Zr, Zircaloy-2, and Zircaloy-4, with rolling texture have also been investigated using diffraction methods to determine either lattice strain evolution using \textit{in situ} measurements [9, 2], or \textit{ex-situ} measured residual strains [55, 56, 57, 58, 59]. Thus far, there has been very little done to study the possible effects of relaxation in the material during measurement via diffraction and how such relaxation would affect the observed lattice strains in the material.

The only published study at the time of this writing that begins to throughly
investigate the phenomena of sample relaxation during \textit{in situ} diffraction studies focuses on AZ31 [60], a magnesium alloy that has markedly different deformation characteristics than Zr and its alloys. Additionally, while the paper makes several valuable comparisons between experimentally measured lattice strains, deformation mode activities, and diffraction peak intensity with those values predicted by the EVPSC model, there is no comparison made between the amount of macroscopic sample relaxation observed and that predicted by EVPSC. Without knowing the extent to which the model can replicate the basic macroscopic response of the material, it is difficult to judge the significance of the predicted lattice strains, which vary in accuracy.

2.6.2 Interpretations of Lattice Strain Data

Interpretation of lattice strains in polycrystalline material is generally done through a combination of experiments and modeling. Through careful study of these lattice strains it is possible to better understand the overall macroscopic behavior of the material. Due to the difficulty in measuring the response of individual grains within a polycrystalline matrix directly, matching model results to the experimental data is the only practical method that can be used to determine quantities such as the CRSS’s and hardening behavior of the active deformation modes in the material. Self-consistent models are generally used for this by varying the CRSSs and hardening parameters of the individual deformation modes. The values of these parameters are found by varying them over a wide range and choosing the combination of parameters which give the best correspondence between model and experimental data.
lack of currently available data regarding the true physical values of these parameters constitutes a major problem in the process, since it requires that a large number of parameters be refined with a relatively small amount of experimental data to compare the model results with.

Each deformation mode included in the modeling process has several parameters used to describe it. This makes it useful to know the minimum combination of deformation modes needed to successfully simulate the behavior of the material, since fewer deformation modes will result in fewer parameters that need to be refined. Because of this, it may be tempting to include (in the case of Zr, for example) only prism $<a>$ slip, and pyramidal $<c+a>$ slip or tensile twinning, since these modes together are capable of accommodating strains in any direction within the material. It has been found, however, that the inclusion of basal $<a>$ slip in the model increases agreement with experimentally measured texture development [61, 62], lattice strain curves [29], and macroscopic flow curves [29]. Pyramidal $<a>$ slip has also been proposed as an alternative to basal $<a>$ slip in modeling [63], but a lack of experimental evidence for pyramidal $<a>$ slip occurring in Zr alloys, combined with the conclusions of Xu et al. [29] that the inclusion basal $<a>$ slip gave satisfactory model results, would indicate that pyramidal $<a>$ slip does not likely occur in great enough amounts (assuming it occurs at all) to be worth including during the modeling process. For these reasons, the deformation modes considered during this research will be limited to prism $<a>$ slip, basal $<a>$ slip, pyramidal $<c+a>$ slip, and tensile twinning.
Values are often difficult to obtain for the various CRSS’s and hardening parameters for the various deformation modes. This can be for a variety of reasons, but a major one appears to be the size of the experimental data set used. Since the number of degrees of freedom (ie the number of parameters being refined) is very high when refining the hardening parameters for multiple deformation modes, it is necessary to have as large a data set as possible to provide adequate constraint. This problem has been explored previously by Xu [10], who performed a search of the parameter space by fitting against a large data set of experimentally measured macroscopic flow curves, lattice strain curves, and Lankford coefficients, which were each measured in all three principle directions [2] in both tension and compression.
Chapter 3

EPSC Optimization using a Genetic Algorithm

3.1 Introduction

A major difficulty encountered in determining the single crystal properties of many engineering alloys is the nearly impossible task of growing large enough crystals to make direct measurements of the single crystal properties. When considering for example mechanical properties, this problem is further compounded by the fact that a single, unconstrained crystal can exhibit a significantly different mechanical response than one that is constrained within a polycrystal, due to surface effects. For these reasons, one practical method to study the single crystal properties of engineering alloys is to fit experimental results to predictions of polycrystalline models that include physics based descriptions of the interaction between the single crystal grains that make up the polycrystal [64].
In order to determine the single crystal properties of the material, the input parameters of the model must be optimized such that the model output fits the experimental data as closely as possible. This task is complicated somewhat by the fact that the physical limitations of any given model make it likely that the best possible fit will still fail to capture some features present in the experimental data. Thus, it can be difficult to determine if any disagreement is due to a poor choice of single crystal parameters, or simply because of the shortcomings of the model. In addition, there are multiple parameters to fit, with typically an indirect relationship between changes in model parameters and the resultant changes in experimentally measurable parameters. This makes an optimal choice of input parameters challenging, particularly for numerical (as opposed to analytical) models. In this paper a Genetic Algorithm approach is used to optimize the agreement between a model of polycrystalline plasticity and diffraction data sets, as well as macroscopic data in the form of stress-strain curves and Lankford coefficients.

Zirconium alloys have extensive applications in the nuclear industry, primarily in structural components within both experimental and commercial power reactors. One such alloy, Zircaloy-2, is made up primarily of zirconium (>98 wt%) with a hexagonal close packed (HCP) structure which results in anisotropic thermal, plastic and elastic properties. The coefficient of thermal expansion in the $c$ direction is larger than that of the $a$ direction by nearly a factor of two. The elastic constants are generally assumed to be equal to those of a pure zirconium single crystal [4]. During plastic deformation of zirconium it has been well established [17, 21, 20] that prism $< a >$ slip $\{10\bar{1}0\} < 1\bar{1}20>$ is the most easily activated deformation
mode, followed by pyramidal \(< c + a > \) slip \(\{10\overline{1}1\} < 11\overline{2}3 >\) or tensile twinning \(\{10\overline{1}2\} < 10\overline{1}1 >\) and then finally compressive twinning \(\{11\overline{2}2\} < 11\overline{2}3 >\). Although prism \(< a >\) slip can accommodate strains in only the \(a\) direction, both pyramidal \(< c + a >\) and twinning are able to accommodate strains in both the \(a\) and \(c\) directions.

In addition to these deformation mechanisms, there has been a certain amount of contention in the literature over the activity of basal \(< a >\) slip at room temperature [65, 66, 67, 68, 55, 69, 32, 61, 70], with some authors [28, 49, 71] preferring to use pyramidal \(< a >\) slip instead. More recently, however, it was shown that basal \(< a >\) slip is moderately active during plastic deformation, while pyramidal \(< a >\) slip can be reasonably neglected [29].

The differences in the stress states of grains with different orientations embedded in a Zircaloy polycrystal have various sources which are all due to the combination of an anisotropic grain being constrained in a polycrystal, which may itself be anisotropic if the material is textured. For example, stresses are induced in the material as it is cooled after a thermal treatment [54]. Under mechanical loading, the load will be distributed unevenly among the grains in a manner analogous to composite load sharing, due to the differing elastic properties of the grains [32]. Any plastic strains will also be anisotropic due to a combination of differences in critical resolved shear stress (CRSS) of various deformation modes, differences in the Schmid Factor, and differences in stresses among grains due to elastic anisotropy.

Due to the elastic and plastic anisotropy, differently oriented grains in the material will regularly exhibit markedly different local stress-strain responses. Likewise,
thermal anisotropy can result in residual stresses being left within the material after thermal processing is performed. Polycrystalline models can be used to interpret the complex intergranular stresses that arise due to thermal and mechanical processing [72], giving insight into the contributions of various micromechanical deformation modes to the overall material response. If properly implemented, these models can be used to predict lifetimes of reactor components and improve processing routes. Two popular types of models are finite element crystal plasticity models, and elastoplastic self-consistent models. In this paper the latter will be used.

Here we examine an extensive set of experimental data showing the internal strain evolution of samples cut from a moderately textured Zircaloy-2 slab. The measurements were made by neutron diffraction and have been reported previously [2]. Tension and compression tests performed in situ were done in all three principal directions (rolling, transverse, and normal), to macroscopic strains of at least 10%. In each test, lattice strains were measured in all three principal directions. The results obtained from the elastoplastic self-consistent model can be used to interpret the lattice strain evolution by exploring the relative contributions of the different deformation mechanisms.

3.2 Summary of Experiments

The experiments performed to collect the experimental data used for model fitting have been well described and documented in the past [2] and will only be summarized briefly here.
The stock material used was a slab of Zircaloy-2 that had been warm rolled after preheating to approximately 700K and then cooled to room temperature. Transmission electron microscopy indicated that the material had been fully recrystallized, meaning it had reached a temperature of at least 900K during processing [7]. This was likely due to adiabatic heating. Grains in the material are equi-axed with an average size of approximately 20µm.

Texture measurements have shown that the basal plane normals are oriented primarily in the normal direction with a spread of ±50° towards the transverse direction (TD) and ±30° towards the rolling direction (RD). Pole figures describing the texture are available in [8] and also shown later in Figures 4.1(a) and 4.1(b). This pattern implies that more basal normals can be observed in the TD than the RD. The \{10\overline{1}0\} normals are weakly clustered at approximately 30° away from the RD in the RD/TD plane. This uneven distribution of basal normals results in the material exhibiting significantly different mechanical responses in different principal directions, both macroscopically and also in terms of lattice strain evolution.

All the data in Ref. [2] was collected by neutron diffraction with the gauge volume centered at the midpoint of the sample. Lattice strains were calculated by measuring the peak shifts relative to the peak position before loading began, as demonstrated in equation 3.1 where \( \epsilon_{hkil} \) is the lattice strain of the grain family \( hkil \); \( d_{hkil}^{ref} \) and \( d_{hkil} \) are the plane spacing before and after deformation; and \( \theta_{hkil}^{ref} \) and \( \theta_{hkil} \) are the diffraction angles before deformation and while under load. Note that because \( d_{hkil}^{ref} \)
is measured at room temperature just before the uniaxial tests were carried out, it will include thermal strains that were incurred during the cooling of the material to room temperature after processing.

\[ \epsilon_{hkil} = \frac{d_{hkil} - d_{hkil}^{ref}}{d_{ref}} = \frac{\sin \theta_{hkil}^{ref}}{\sin \theta_{hkil}} - 1 \]  

All plotted lattice strains are calculated with respect to the start of each test, and thus represent only the increment produced by loading. It should also be noted that the lattice strain is the average response of all grain orientations that satisfy Bragg’s law for a particular \( hkil \) in the gauge volume while neutron data is being collected.

### 3.3 Self-Consistent Modeling

An elastoplastic self-consistent polycrystalline model [45] was used to simulate the macroscopic flow curves, Lankford coefficients, and lattice strain evolution as a function of applied strain. These results can then be used to infer the relative contributions of various deformation mechanisms to the overall behavior of the material.

In brief, the EPSC model functions by assuming each grain is an elastoplastic inclusion embedded within a homogeneous effective medium (HEM), and the HEM is treated as an anisotropic elastoplastic aggregate. Interactions between the grain and HEM are calculated using an elastoplastic Eshelby approach. The model iterates until a solution is found where the HEM is representative of the average response of the grain population used. A grain population is chosen such that the measured
texture is represented. For a full description of the model, see [45].

The model uses an empirical extended Voce hardening law to describe each of the deformation modes, shown in equation 3.2.

\[
\tau^s = \tau^s_0 + (\tau^s_1 + \theta^s_1 \Gamma)[1 - \exp(-\theta^s_0 \Gamma / \tau^s_1)]
\] (3.2)

Where \(\tau^s\) is the instantaneous CRSS and \(\tau^s_0\) and \(\tau^s_0 + \tau^s_1\) are the initial and final back-extrapolated CRSSs, respectively. \(\theta^s_0\) and \(\theta^s_1\) are the original and asymptotic hardening rates, and \(\Gamma\) is the accumulated plastic shear in the grain. The possibility of self and latent hardening is also accounted for by defining coupling coefficients \(h^{s/s'}\), which empirically account for the obstacles to deformation that mode \(s'\) represents for mode \(s\).

\[
\Delta \tau^s = \frac{d\tau^s}{d\Gamma} \sum_{s'} h^{s/s'} \Delta \gamma^{s'}
\] (3.3)

Lattice strains calculated by EPSC for comparison with neutron diffraction measurements are the averaged response of an entire family of grains. This means that all grains with a particular \(\{hkil\}\) within \(\pm 7.5^\circ\) relative to a nominal direction within the sample will have their lattice strain states averaged. This angular spread is required to ensure a reasonable sampling of grains during simulation and is comparable to the corresponding angular spread of grains captured by ENGIN-X [73], where the compression tests were carried out. The angular spread of the grains averaged during the tensile tests performed at the NRU facility at Chalk River, Canada is smaller than that of Engin-X and is controlled principally by the incident beam divergence and detector spread. Regardless, the effects of this averaging of lattice strain measurements over grains with slightly varying orientations has been previously investigated
and these different averages are not expected to have a significant result on any results reported here. These angles also represent the same angular averages used in the modeling of Xu [10] which we compare to directly here. The results can then be compared directly to the neutron diffraction measurements made experimentally. Note that the version of EPSC used here does not include texture development, and the twinning model does not account for any grain reorientation or local stress relaxation due to twinning.

In order to obtain the initial thermal strains for the subsequent mechanical loading, cooling was modeled from a stress-free state at 898 K down to 298 K [46]. Boundary conditions were applied to model the mechanical loading processes to the strain states at which neutron diffraction measurements were performed. All thermal macroscopic and internal strains due to the cooling process were subtracted from those calculated due to mechanical loading so that the results could be compared directly to experimental strains measured by diffraction.

In the EPSC calculations, the elastic constants of Zircaloy-2 were assumed to be equal to those of pure zirconium, as reported by Fisher and Renken [13]: $C_{11} = 143.5$ GPa, $C_{33} = 164.9$ GPa, $C_{12} = 72.5$ GPa, $C_{13} = 65.4$ GPa, and $C_{44} = 32.1$ GPa. The thermal expansion coefficients were previously determined by experiment to be $\alpha_a = 5.3 \times 10^{-6} K^{-1}$ and $\alpha_c = 10.1 \times 10^{-6} K^{-1}$ [8]. These values are reasonably consistent with those reported for pure single crystal zirconium, and were also the same values used during the manual refinement process. All grains were assumed to be spherical in shape. The representative volume element consisted of 1944 grains with
volume fractions chosen to be representative of the initial crystallographic texture of the Zircaloy-2 slab.

Plastic deformation can be correlated with the activities of the various deformation modes, which are themselves controlled by several factors including elastic and plastic anisotropy, grain orientations, and external loading. During modeling, the Voce hardening parameters $\tau_0, \tau_1, \theta_0$, and $\theta_1$ were used as adjustable fitting parameters. The hardening coefficients $h^{s/s'}$ were set at the optimal values found previously [10]. The deformation systems taken into account during modeling are prism $< a >$ slip $\{10\bar{1}0\} < 11\bar{2}0 >$ (pr), basal $< a >$ slip $\{0001\} < 11\bar{2}0 >$ (bas), pyramidal $< c + a >$ slip $\{10\bar{1}1\} < 11\bar{2}3 >$ (pyr) and $\{10\bar{1}2\} < 10\bar{1}1 >$ tensile twinning (tt). Note that although tensile twinning is included in the model, it is treated as a unidirectional slip system; with an assigned CRSS and a shear strain representing the twinning. Some degree of stress relaxation can be accounted for as well, but this relaxation is typically insufficient to match experimental observations [75].

A past attempt has been made to optimize the parameters of each deformation mode to optimise agreement [10]. The procedure used involved the manual optimization of eight unique model parameters simultaneously (tau-zero and theta-zero, Eq-1, for each of the four deformation modes, see next section for more detail). An eight dimensional grid of possible values was generated and initially a coarse search (manual examination) of the grid space was performed in order to find an approximate solution. The point on this coarse grid providing the best fit to the experimental data was then used as the centre point for a finer grid used to further improve the
results. While this process yielded good results, parameter selection and goodness of fit were judged manually in a subjective, time-consuming manner. We therefore present an alternative method using a numerical fitting technique which is completely automated and uses a genetic algorithm (GA) for parameter optimization.

The validity of any fitting of model data to experimental data is dependent on two main factors. First, there are the number of degrees of freedom, or put another way, the number of material parameters being adjusted. Secondly, there is the size of the experimental data set; a larger set of experimental data results in fewer combinations of model parameters being able to reproduce the experimental results accurately. For example, having both compression and tensile data makes it easy to reject a parameter set that fits tensile data well at the expense of providing very poor agreement in compression, or vice versa. In this study, a single set of parameters was fit to all experimental data simultaneously, both in tension and compression and in all three principal directions.

3.4 Genetic Algorithm

A genetic algorithm at its most basic level is simply an optimization technique, and can be applied to any number of problems including data fitting. In order to limit the size of the possibility space, the GA begins by accepting upper and lower bounds on the parameters to be adjusted and generates several sets of these parameters, distributed randomly throughout the available possibility space. Each set of parameters is known as an individual and the first set of randomly generated individuals form
the first generation. The algorithm then sends each individual to a separate instance of the problem being optimized (i.e. in this case the polycrystalline plasticity code). The various individuals then have the results of their application to the problem evaluated by a fitness function of some sort. Once all the individuals have been evaluated and ranked relative to one another, a sample of the highest ranking individuals are taken and used as potential parent individuals to form the next generation.

The formation of each generation after the first one occurs via two operators, crossover and mutation. Crossover is the process of creating an individual for generation n from two individuals in generation (n-1). In the present case, crossover was performed by randomly selecting two parent individuals amongst the highest ranking individuals of generation n. For example, if two individuals in a given generation n with parameter sets of \((a_1; a_2; ...; a_i)\) and \((b_1; b_2; ...; b_i)\) are used in a crossover process, the newly formed individual has the following parameters:

\[
(\alpha_1 a_1 + (1 - \alpha_1)b_1; \alpha_2 a_2 + (1 - \alpha_2)b_2; \alpha_i a_i + (1 - \alpha_i)b_i) \quad (3.4)
\]

where \(\alpha_1, \alpha_2, ..., \alpha_i\) are random real numbers comprised between 0 and 1. Mutation is analogous to biological mutation; it is a random process in which an individual is given one or more traits which were not present in either parent. Mutation is important in that it maintains a level of diversity amongst individuals of the same generation, which is useful in helping the GA to avoid becoming trapped in a region of the parameter space which is only locally optimal. If we were to add mutation to the previous example involving crossover, a potential result is:

\[
(\alpha_1 a_1 + (1 - \alpha_1)b_1; x_2; \alpha_i a_i + (1 - \alpha_i)b_i) \quad (3.5)
\]
Table 3.1: Values of the various parameters used during genetic algorithm refinement

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$N_{gen}$</td>
<td>90</td>
</tr>
<tr>
<td>$N_{ind}$</td>
<td>40</td>
</tr>
<tr>
<td>$P_{mut}$</td>
<td>0.02</td>
</tr>
<tr>
<td>$N_{hri}$</td>
<td>8</td>
</tr>
</tbody>
</table>

Note that $x_2$ is an entirely new trait not previously seen in the parent individuals. The genetic algorithm is thus controlled by the following parameters: $N_{gen}$, the total number of generations; $N_{ind}$, the number of individuals per generation; $P_{mut}$, the mutation probability; $N_{hri}$, the number of highest ranking individuals that can be used to form the next generation; $(l_1; l_2; \ldots; u_i)$ and $(u_1; u_2; \ldots; u_i)$, the lower and upper bounds for the different parameters. The values used for these parameters during refinement are shown in Table 3.1, with the exception of the upper and lower bounds, which are discussed below.

A genetic algorithm is well suited to the problem at hand for a variety of reasons. First and foremost, the structure of the algorithm makes it ideal for use with parallel computing techniques, since the various individuals don’t interact with each other except during creation of the next generation. This means that each individual can have its own parallel process dedicated to it, speeding up the overall fitting procedure considerably. Additionally, unlike some simpler optimization techniques, genetic algorithms have some ability to overcome local minima and keep searching for a better solution. While some techniques such as a gradient descent method, are only capable of reaching a single minimum, the genetic algorithm will continue to search for better fits until it reaches some end condition specified by the user. The speed of the calculations performed by the algorithm is also sufficiently fast that the total time taken
for parameter selection is essentially determined entirely by the runtime of EPSC.

Four deformation processes are taken into account during modeling are subsequently refined by the GA: prism \(< a >\) slip, basal \(< a >\) slip, pyramidal \(< c + a >\) slip, and tensile twinning. The CRSS and hardening parameters for all of these mechanisms are deduced by simultaneously fitting the macroscopic curves, Lankford Coefficients, and lattice strain evolution for all diffraction peaks for all 18 combinations of measurement and loading directions.

As stated in the previous section, the parameters being adjusted are \(\tau_0, \tau_1, \theta_0,\) and \(\theta_1,\) which have separate values for each of the four deformation modes, for a total of 16 parameters. In keeping with the previous attempt at fitting EPSC output to this dataset, linear hardening of the deformation mechanisms was assumed (\(\tau_1 = 0\) and \(\theta_0 = \theta_1\)). This reduces the number of free parameters from 16 to 8.

The bounds for the fitting parameters were chosen as follows: \(85 \leq \tau_0^{pr} \leq 125\text{MPa},\) \(110 \leq \tau_0^{bas} \leq 250\text{MPa},\) \(230 \leq \tau_0^{pyr} \leq 350\text{MPa},\) \(170 \leq \tau_0^{tt} \leq 290\text{MPa},\) and \(0 \leq \theta_0 = \theta_1 \leq 1.\) These ranges were chosen to agree with published polycrystal data, and are also roughly equal to the bounds used in previous fitting attempts. After some trial and error to find a good balance between total run time and the final quality of the results, a fitting procedure was developed. Forty individuals were used in each generation, and the algorithm was allowed to run for a total of 90 generations, resulting in a total of 3600 separate parameter sets being run through EPSC. Running these EPSC simulations in series would have been extremely time consuming, so instead
the High Performance Computing Virtual Laboratory was utilized to run 40 instances of EPSC in parallel, which allowed an entire generation to be tested simultaneously. A total of 40 processing cores with speeds of approximately 1.8GHz were used, with each instance of EPSC having its own dedicated core. Even with this highly parallelized configuration, it still took roughly 4-5 days to run all 90 generations.

The goodness of fit was judged separately for each data series, and then combined in a weighted sum to give a measure of the overall fitness to the data. The fitness was estimated for each data series by calculating the area of the domain comprised between the experimental and model curves with a trapezoidal rule. This area was normalized against the total area under the experimental data for each data series. Thus the fitting criteria is the percentage off difference in area under the model-generated and experimental curves, a unitless quantity. Since the Lankford coefficients do not have an independent variable associated with them, they were numbered between one and six and the curve defined by the six points of \( <i>, R_i \) was used as the “curve to compare between model and experiment. (Where \( R_i \) is the \( i^{th} \) Lankford coefficient in the list of experimental values). The goodness of fit associated with each data series being compared are then added up in a weighted sum and divided by a scaling factor to give the overall error associated with the set of hardening parameters used to generate the model data. Different weightings were given to the various data series depending on what type of curves were being compared. Each of the macroscopic flow curves were given a weighting of 15, each lattice strain curve measured parallel to the loading axis was given a weighting of 3, lattice strain curves perpendicular to loading each had a weighting of 1, and the Lankford coefficients had a combined weighting of
Table 3.2: Critical resolved shear stresses and Voce coefficients found by GA

<table>
<thead>
<tr>
<th></th>
<th>GA $\tau_0$ (GPa)</th>
<th>GA $\theta_0 = \theta_1$</th>
<th>Manual $\tau_0$ (GPa) [10]</th>
<th>Manual $\theta_0 = \theta_1$ [10]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prism $&lt; a &gt;$ slip</td>
<td>0.101</td>
<td>0.011</td>
<td>0.100</td>
<td>0.020</td>
</tr>
<tr>
<td>Basal $&lt; a &gt;$ slip</td>
<td>0.149</td>
<td>0.049</td>
<td>0.160</td>
<td>0.020</td>
</tr>
<tr>
<td>Pyramidal $&lt; c + a &gt;$ slip</td>
<td>0.347</td>
<td>0.494</td>
<td>0.320</td>
<td>0.400</td>
</tr>
<tr>
<td>Tensile Twinning</td>
<td>0.178</td>
<td>0.003</td>
<td>0.240</td>
<td>0.100</td>
</tr>
</tbody>
</table>

15. These values were chosen subjectively, based on a prior determined assumption of the relative importance of the data. Different variations on these weighting factors were attempted in an attempt to find values that gave good results, but due to the long computation time involved in doing so, the search was far from exhaustive and the selection of these weighting factors should not be seen as optimal. The macroscopic curves were given the highest weightings because they represent the overall behavior of the material, and have the least uncertainty associated with them both in modeling and in experimental measurement. Lattice strains parallel to the load were weighted more heavily than ones measured perpendicular to the load because EPSC is known to produce results which are less accurate for perpendicular lattice strains [76]. The values found to give the best fit to the data are listed in Table 3.2.

### 3.5 Results

The following subsections contain direct comparisons of the model data to experimental measurements, as well as model data previously produced by selecting parameters manually. It will be seen that in some cases the GA produced a better fit to the experimental data, while in other cases manually selecting parameters gives better
results. While it is difficult to definitively say that one method is better than the other overall, the fact that the GA is both faster and totally automated makes it more convenient to use, especially on large data sets such as this one.

3.5.1 Macroscopic Behavior

The macroscopic flow curves predicted by EPSC for all three directions in both tension and compression are shown in Figure 3.1. It is evident that in the elastic regime of the curves, the model is a very good fit, indicating that the elastic parameters did not need to be adjusted and that the initial texture of the material has been well represented in the model. The model also predicts the yield stresses of the material well for most directions, with the notable exception of the tensile test performed in the normal direction. In the Tens.ND test, the parameters selected by the GA result in yielding occurring at a lower stress than is observed experimentally, while the manually selected parameters give a much more accurate representation of the yield strength in that test. Since the difference in yield strength in tension and compression in the normal direction are due to the relative CRSS values for pyramidal \(<c + a>\) slip and tensile twinning, this may indicate the solution proposed by the GA has a poor ratio of \(\frac{\tau_{pyr}}{\tau_{tt}}\). The curvature of the elasto-plastic transitions is captured well by both fitting attempts, with the amount of curvature being greatest in the rolling direction for compression tests, and greatest in the normal direction for tensile tests. The changes in curvature are controlled by the residual thermal strains in the material and the activation sequence of the deformation modes, which is in turn controlled by their initial CRSS’s, hardening behavior, and the grain orientations relative to the
external loading. Although the yield strength of the material is underestimated while under tension in the normal direction, the rest of the curves show good agreement in yield strength and curvature, indicating that the CRSS values are likely valid for most, if not all, deformation modes.

In the plastic regime, the model gives mixed results. In the normal direction, excellent agreement is given in compression, but in tension the model underestimates the yield strength of the material. In the transverse direction, the parameters selected by the GA show a significant improvement over those selected manually; the flow curves in both tension and compression show excellent agreement with the experimental data. The fact that the TD is the one best represented by the selected parameters is interesting, because the TD contains an intermediate concentration of basal normals. This intermediate concentration of basal normals gives the TD deformation behavior that is part way between the two extremes of the RD and ND, particularly with regards to the prevalence of twinning. The rolling direction shows results which are the opposite of those seen in the ND, here we see that it is the tensile data which is well represented, while the compressive data is fitted relatively poorly. One possible explanation for the excellent agreement in Comp.ND and Tens.RD and poor agreement in Comp.RD and Tens.ND is that the CRSS selected for tensile twinning may be too low. Due to the concentrations of basal normals, we expect large amounts of twinning in the Tens.ND test as well as the Comp.RD test (due to \{0002\} directions perpendicular to the load being placed in Poisson tension), which were the two least accurately predicted macroscopic flow curves. Also, the CRSS for tensile twinning used in the manual parameter selection was significantly higher than the
Figure 3.1: Modeling of the macroscopic flow curves in tension and compressing in ND (a), RD (b) and TD (c).
value selected by the GA, which would explain why the manually selected parameters fit the Tens.ND and Comp.RD curves better.

The Lankford coefficients were also calculated for all tests and compared to the experimental values and are summarized in Table 3.3. Where the Lankford coefficient \( R = \frac{\epsilon_{XD}}{\epsilon_{YD}} \) (XD is the Poisson direction with relatively larger contraction or expansion, and YD is the other Poisson direction). All Lankford coefficients were calculated at approximately the same strain as the last experimental data point in the macroscopic flow curve (13% for the ND tests, 12% for RD, and 17% for TD). As shown, the fit of the data to the Lankford coefficients varies significantly. This is due in part to the fact that two compression samples were used in each test direction and loaded to different amounts of total strain. The values used were the ones from the samples loaded to total strains closest to those used in calculations (13%, 12% and 17% for the ND, RD and TD tests, respectively), but were still not exact matches. Although the absolute values do not show particularly good agreement, it should be noted that the relative trend among the Lankford coefficients is reasonably well represented.
3.5.2 Lattice Strain Evolution

In this section, the calculated evolution of lattice strain will be compared to the experimental data, as well as the calculated results derived from the manual fitting procedure. The lattice strain evolution for most diffraction peaks contain several inflection points, indicating changes in load sharing among different orientations as deformation modes activate. Families of grains which have their \{hkil\} normals aligned so that they diffract in the same directions will be referred to as “orientations” or “\{hkil\} oriented grains”. Note that equivalent angular averages were extracted from the model data as measured experimentally. Orientations giving diffraction peaks which result in lattice strain curves exhibiting a high elastic modulus will said to be elastically “stiff” while orientations with a low elastic modulus will be called elastically “compliant”. Likewise, orientations that deform at applied stresses corresponding to the activation of a deformation mode with a high CRSS in the plastic regime will be called plastically “hard”, and those which show deformation indicated a deformation mode with a low CRSS will be called plastically “soft”. The notation of XD/YD will be used, where XD refers to the loading direction and YD is the measurement direction, e.g. ND/RD refers to loading in ND and measurement in RD.

Compression in ND

Figure 3.2 compares the lattice strain development predicted by the model to the experimental data. In the direction of loading (Figure 3.2(a)), the model is in reasonable agreement with the experimental data for most crystallographic directions in terms of both magnitudes and trends. Some exceptions are the \{10\bar{1}1\}, which
has its lattice strains overestimated by the model, as well as the \{10\overline{1}0\} direction, which has its initial yield point underestimated, but then shows better agreement at higher strains. The \{0002\} and \{10\overline{1}2\} directions are very well estimated by the model, showing good agreement throughout the plastic region and capturing the inflection points in the curves well. The GA shows some improvement over the previous optimization attempt, particularly in the yielding of the \{10\overline{1}1\} oriented grains, as well as the calculation of the lattice strains of the \{10\overline{1}0\} oriented grains at high load.

There are various stress values in ND that correspond to significant inflection points in the data. These points occur at -180MPa, -360MPa and -500MPa and are represented in the modeling results with varying degrees of success. At -180MPa the model correctly predicts micro-yielding in the \{10\overline{1}0\} direction, but the amount of yielding is overestimated, resulting in the underestimation of the associated lattice strains. The \{10\overline{1}2\} curve shows good agreement between model and experiment at the point where it begins to show non-linearity at -360MPa, likewise with the \{10\overline{1}1\} orientation. Finally, the yielding of the hardest \{0002\} oriented grains at -500MPa is well represented by the model.

The corresponding relative activities of the slip systems (as calculated by EPSC using the parameters selected by the GA) are also shown and can be used to explain why the inflections in the lattice strains occur. The relative activity of a given deformation mechanism (i.e. a slip or twin system) represents the proportion of plastic deformation currently being created via that mechanism. Therefore, it says nothing about the total amount of plasticity, but simply indicates which modes are more or
Figure 3.2: Modeling of the lattice strains in ND (a), RD (b), and TD (c) during compression in ND.
less active than others. The first inflection occurs with the onset of prismatic slip, which is also accompanied by a small amount of tensile twinning. The fact that the contribution of tensile twinning is small in this orientation makes sense, because most of the basal normals are aligned in the ND and are thus under compression. The inflection at -300MPa coincides with the basal slip activity becoming comparable to that of the prismatic slip, and by -360MPa the basal slip system has become the dominant deformation mode, causing another inflection. The activation of the pyramidal $<c+a>$ slip system at approximately -500MPa also causes some inflections in the lattice strains. Looking back at Figure 3.1(a) it can be seen that the macroscopic flow curve begins to exhibit significant plastic deformation at -500MPa, which would indicate that the activation of the pyramidal $<c+a>$ slip system is what primarily controls yielding in that orientation. It should be noted that all activities plotted are relative, and thus will always sum to one. The average number of deformation systems operating in a grain changes as deformation progresses, growing from zero (totally elastic deformation) to an average value of roughly 3.41 when highly plastic behavior is observed.

In the two Poisson directions, reasonable agreement between experiment and model is achieved as shown in Figures 3.2(b) and 3.2(c). The strong inflection point at -300MPa exhibited in the \{0002\}, \{10\overline{1}1\}, and \{10\overline{1}2\} curves in both TD and RD are attributed to the strong activity of basal slip at that stress. Due to the very small activity of tensile twinning in this orientation, the large relaxation exhibited by the \{0002\} at a stress of -360MPa is most likely due to basal slip supplanting prismatic slip as the most active deformation mode. For the most part the GA and the manual
refinement result in similar trends in the lattice strains being calculated, with a major exception being the \{0002\} oriented grains in both TD and RD. In the case of the \{0002\} oriented grains, the lattice strains are underestimated by the GA and over-estimated by the manual parameter selection. Additionally, the manual parameter selection predicts that the \{0002\} oriented grains will continue to harden in both the TD and RD, while the GA predicts a slight softening behaviour. The scatter of the experimental data makes it difficult to make any definite conclusions, the GA’s softening trend seems more accurate in TD while the manual selection’s hardening trend is better suited to the data in RD. The scatter seen in the \{0002\} orientations is likely due to the very low intensity of the \{0002\} peak in the observed diffraction spectra, since the majority of \{0002\} poles are oriented towards ND. The low intensities of the peaks in TD and RD make it difficult to measure the peak shifts, a problem which becomes even more significant as peak broadening effects occur at higher plastic strains.

**Compression in TD**

Calculated lattice strains for compression along TD are compared to the experimental results in Figure 3.3. In the direction of loading (Fig. 3.3(c)) the initial yielding of the prismatic orientations occurs at -230MPa and is very well reproduced by the model. This initial yielding is correlated with prism \(< a >\) slip being the only significantly active slip system. By the time the load has reached approximately -320MPa, significant load partitioning can be observed among the various orientations, likely due to the corresponding increase in the activities of both basal slip and tensile twinning.
There are two major inflection points in the \{0002\} lattice strain that are not captured well by the model, occurring at -377MPa and -430MPa. Although the model does predict a small strain relaxation at -377MPa, it is not nearly as significant as the one observed experimentally. It is believed that the two inflections in the \{0002\} curve are caused by tensile twinning, the activity of which has risen significantly by -377MPa, and that the previously mentioned deficiencies of the model with regards to twinning are to blame for the disagreement with the experimental observations. The genetic algorithm shows markedly better performance than manual parameter selection in the \{10\overline{1}0\} oriented grains, with a more modest improvement in the \{0002\} orientation. The \{10\overline{1}1\} and \{10\overline{1}2\} orientations see the GA give results that are on par with manual selection, until -377MPa, at which point the GA diverges in an unfavourable way.

Both Poisson directions (Fig. 3.3 (a) and (b)) show lattice strains that are reasonably well predicted for loads less than -377MPa. In TD/ND, the lattice strains are reproduced very well for most orientations, even at loads well above -377MPa. In TD/RD, however, very poor agreement is shown for all orientations at high loads. This is believed to be due to the model’s inability to properly represent twinning. The inflections visible at -320MPa can be attributed to the increase in activity of the basal slip and tensile twinning systems. Shortly afterwards, another inflection point is observed at -370MPa corresponding to a slight decrease in basal slip activity as well as small increases in prismatic and pyramidal \(< c+a >\) slip activities. In TD/ND the GA performs very well when compared to manual parameter selection, doing a better job of predicting both magnitudes and trends of the lattice strains in all orientations.
Figure 3.3: Modeling of the lattice strains in ND (a), RD (b), and TD (c) during compression in TD.
except for \{10\bar{1}0\}, which contains too much scatter to accurately determine which fitting method is performing better. In TD/RD both methods of optimization give results that are unsatisfactory.

**Compression in RD**

Compression along the RD results in three inflections in the lattice strains parallel to loading, shown in Figure 3.4(b). As in TD and ND, the first inflection point is caused by the initiation of prism slip. This initial inflection occurs at -250MPa and the deviation from linearity at that point is well demonstrated by the model. The second inflection point at -340MPa appears in the \{10\bar{1}1\} and \{10\bar{1}2\} curves. This inflection can be attributed to the increased activity in both basal slip and tensile twinning, both of which have approximately the same level of activity at this point. At -370MPa a third inflection is visible in the \{0002\} and \{10\bar{1}1\} orientations. Unlike the other observed inflections, there is no clear change in activity observed in the model at -370MPa, which may indicated that the inflection is partially due to stress relaxation caused by the reorientation of grains due to tensile twinning, which the model does not account for. With the exceptions of the \{0002\} and \{10\bar{1}1\} orientations, the lattice strain evolution predicted by the model agrees reasonably with the experimental data for loads less than -420MPa. Beyond this level of stress there is no lattice strain data available, because the model did not reach that load before the final macroscopic strain of the test was reached, as shown in Figure 3.1(b). Manual parameter selection produced more accurate lattice strain calculations in RD/RD,
mostly in grain orientations where neither optimization method produced particularly accurate results, such as in \{0002\} and \{10\overline{1}1\}.

In RD/TD, many of the lattice strains are not well predicted past the point of yielding. In particular, the \{0002\} and \{10\overline{1}2\} strains are very poorly predicted after the initial deviation from linearity. The underestimation of the yield point of the \{0002\} oriented grains may indicate that the CRSS chosen for tensile twinning is too low. Furthermore, the \{10\overline{1}0\} strains are underestimated, but the general behavior of the curve does seem to be well reproduced. Neither method of parameter selection produced accurate lattice strains in RD/TD, although the \{0002\} oriented grains show an interesting divergence between the two; the GA underestimated the lattice strains for that orientation while the manual selection overestimated them. In RD/ND, the GA does an adequate job of reproducing the general trends observed in the experimental lattice curves. By contrast, the manual parameter selection completely diverges from the experimental curves once yielding begins and never manages to recover. This demonstrates the important of fitting more than just the macroscopic flow curves in order to capture what is physically happening in the material; looking only at the macroscopic flow curve would give the impression that the manual parameter selection was far superior replicating the behaviour of the material while under load in RD, but by looking at the lattice strains in RD/ND it can be seen the comparison is actually much more nuanced.
Figure 3.4: Modeling of the lattice strains in ND (a), RD (b), and TD (c) during compression in RD.
Tension in RD

Tension in RD results in mainly prismatic and basal slip, as shown in Figure 3.5. Very little tensile twinning is predicted, due to the texture of the material, and virtually no \( <c + a> \) slip. The initial activation of prism \( <a> \) slip coincides with the first signs of load partitioning in the \( \{1122\} \) oriented grains along the loading axis at 180MPa. The model shows the low-order orientations \( \{2020\}, \{1120\}, \) and \( \{2021\} \) yielding at the slightly higher load of 200MPa. The activation of the basal slip system coincides with a clear inflection in the \( \{1122\} \) curve, with a reduction in the lattice strain indicating that yielding of the \( \{1122\} \) oriented grains has begun. In RD, the GA and manual parameter selection resulted in nearly identical calculations for the lattice strains parallel to loading.

In the Poisson directions, the \( \{0002\} \) curves show reasonably good agreement in both ND and TD. The lower-order orientations, however, have their strains under predicted beyond the stress at which significant levels of basal slip activity occur. Micro-yielding can be seen in the model results at 180MPa, and is controlled exclusively by Prism \( <a> \) slip, which is the only significantly active slip system at that stress level. Although some tensile twinning activity is apparent at the point of this micro-yielding, it is not believed to be causally related since previous modeling results have shown the same micro-yielding behavior with no tensile twinning activity. At higher loads it is clear that basal slip plays an important role in the load segregation between the basal and lower-order orientations. The GA and manual parameter selection methods both showed similar results in calculating the Poisson strains. It can be noted, however, that in cases where both methods produced unsatisfactory
Figure 3.5: Modeling of the lattice strains in ND (a), RD (b), and TD (c) during tension in RD.
results, such as \{2021\} and \{1120\}, the GA produced results that were slightly closer to the experimental values.

**Tension in TD**

For tension in TD, the relative activities show an initial burst of tensile twinning at 150MPa that quickly decays off as prism slip takes over by the time the load surpasses 200MPa. The high twinning activity seems to have only a small effect on the lattice strain evolution since the region it occurs in seems to be still within the elastic regime. Again, it is worth mentioning that just because the relative activity of twinning is high, this says nothing about the absolute amount of deformation occurring. The lattice strain evolution parallel to loading is similar to that in the RD, with the exception that the inflections occur at higher stresses due to the different distribution of basal normals among the RD and TD. This distribution of basal normals is also the reason for the higher relative activity of tensile twinning in the TD case, which occurs at the expense of the prism slip activity. The model agrees reasonably well with the experimental data, with minimal differences between the values found manually and those found through use of the GA (Figure 3.6(c)).

Perpendicular to the loading direction (Figures 3.6 (a) and (b)), the \{0004\} strains in TD/ND and the \{2021\} strains in TD/RD are well reproduced by the model, while the other orientations have only their very basic trends captured. In the case of the \{2020\} and \{1120\} lattice strains, even their behavior relative to one another is not
Figure 3.6: Modeling of the lattice strains in ND (a), RD (b), and TD (c) during tension in TD.
modeled correctly; the model shows \{20\overline{2}0\} exhibiting a sharper deviation from linearity at 300MPa than \{11\overline{2}0\}, but in the experimental data the behavior of the two is reversed. As with the Poisson strains during loading in RD, the GA shows some increased performance over manual selection, but generally in cases where both methods produced results that differed significantly from the experimental data.

**Tension in ND**

Tension in ND differs from tension in TD and RD in that prism \(< a >\) slip is never the dominant deformation mode. Instead, deformation is carried out mainly through tensile twinning, with basal \(< a >\) slip taking over once the load surpasses 400MPa. In ND/ND (Figure 3.7(a)) the lattice strains agree reasonably well with the experimental data with the exception of the \{0004\} orientation, which deviates from linearity at a lower stress than what is observed experimentally. The first inflection occurs in the \{0004\} curve at 250MPa, brought about by the activation of tensile twinning, but as mentioned before this inflection is not observed in the experimental data. At 290MPa, prism \(< a >\) slip activity has reached significant levels, causing inflections in the \{11\overline{2}0\}, \{11\overline{2}2\} and \{20\overline{2}1\} lattice strains. Later, at 360MPa, the \{11\overline{2}2\} curve shows an inflection caused by the sudden increase in basal slip activity. When basal slip begins to dominate the deformation behavior of the material at 400MPa, inflections are visible in the \{11\overline{2}0\}, \{11\overline{2}2\} and \{20\overline{2}1\} lattice strain curves. It should be noted that the accuracy of the relative activity plots above 420MPa appears questionable due to the rapid oscillation of the activities of prism slip, basal slip, and tensile twinning. These oscillations are believed to be an artifact.
of the model. The most striking difference between the two model solutions is in the \{0004\} lattice strains. The GA’s proposed solution results in premature yielding of the \{0004\} oriented grains, due to the activation of tensile twinning, whereas the manual parameter selection method reproduces the experimental data much more accurately. This discrepancy suggests that the manually selected values for the tensile twinning CRSS are more representative of the actual physical values.

In ND/RD and ND/TD (Figures 3.7 (b) and (c)), the lattice strain evolution is very well reproduced except for the \{11\bar{2}2\} in ND/TD. In ND/TD an inflection occurs at 250MPa in the \{11\bar{2}0\} direction, and is believed to be caused by the onset of prism $<a>$ slip. Both the \{20\bar{2}0\} and \{11\bar{2}0\} curves in ND/RD exhibit a similar inflection at 300MPa, which is also believed to be the result of prism $<a>$ slip. The GA and manual selection methods showed roughly equal performance in ND/TD, but in ND/RD the GA appears to conform better to the experimentally measured lattice strains.

3.6 Discussion

3.6.1 Evolution of the Fitting Parameters

The progress of the GA as it searches for an optimal fit is shown in Figure 3.8. The data plotted represents the best fit found for each generation, with the value of the error function being minimized on the Y axis and the generation number on the X axis. The error calculated by the GA for the manually selected parameters is included
Figure 3.7: Modeling of the lattice strains in ND (a), RD (b), and TD (c) during tension in ND.
Figure 3.8: Plot of the overall disagreement between the model and experiment for each generation.

as a horizontal line for comparison. As shown in the plot, most of the optimization occurs in the first 30 generations of refinement with relatively minor improvements from that point on. One might then question why such a large number of generations were used when the algorithm gives such severely diminished returns past this point. The simple answer is that the behavior of the algorithm is difficult to predict, and that large sudden improvements in the space of a single generation were not uncommon during testing. These sudden ‘jumps’ in the error generally happen in the first 30 generations, as shown in Figure 3.8, but some trials of the algorithm had significant sudden increments in much later generations. With this in mind it was decided to simply err on the side of caution and allow the algorithm to run for a very large number of generations.
3.6.2 Deformation Mode Activities

A comparison of the macroscopic flow curves generated by the model to the ones measured experimentally shows that there are two tests which are not well reproduced. These two tests, TensND and CompRD, are the ones where the largest amount of tensile twinning is expected to occur. The lattice strain curves for the TensND and CompRD tests are similarly unreliable, particularly those curves containing inflections associated with the onset of tensile twinning. The tests containing smaller amounts of tensile twinning, for example CompND or tens RD, have their behavior predicted with significantly greater accuracy. With that in mind, along with knowledge gained from past experience, it is reasonable to conclude that a large portion of the discrepancy between the model results and the experimental data is actually due to the limitations of the twinning model used in EPSC and not the result of the GA failing to find an appropriate set of hardening parameters.

Another trend observed in the results is the fact that lattice curves parallel to the direction of loading are more accurately calculated than those perpendicular to the loading axis. This is frequently observed in tests of this type, as it is believed that the EPSC model is less reliable when simulating lattice strains in the Poisson directions [76].

The predicted relative activities of the deformation modes is reasonable for the most part. For most tests, prism $< a >$ slip is the primary mode, followed by basal $< a >$ slip; the two exceptions to this are TensTD and TensND, which both have tensile twinning as the primary deformation mode for at least part of the test. The
unexpected dominance of tensile twinning during tensile testing in TD and ND is likely due to a combination of the inadequate twinning model used in EPSC, and the fact that the GA selected a lower CRSS for tensile twinning than what would generally be expected. Still, the twinning activity does respond properly to texture, with the largest amount of twinning occurring when ND is under tension, less when under tension in TD, and a negligible amount when RD is in tension. Under compression, the trend is reversed, with a large amount of twinning when RD is in tension, a moderate amount while under tension in TD, and barely any observed twinning when ND is placed in tension.

3.6.3 Fit of the Twinning Parameters

As shown in Table 3.2, the parameters selected by the GA for tensile twinning differed significantly from those selected manually. As previously stated, the manually selected twinning parameters resulted in more accurate predictions of certain phenomena such as the macroscopic yield stress in the TensND and CompRD tests, as well as some inflection points in the lattice strain data that are known to be caused by twinning. It is believed that the twinning parameters selected by the manual search process are closer to the real physical value than those selected by the GA. The underperformance of the GA in this area of the fitting procedure seems to indicate that there is more subjectivity involved in the fitting of twinning hardening coefficients as opposed to those pertaining to slip. This makes sense if the limitations of the twinning model used in EPSC is considered. During a manual optimization where the goodness of fit is being judged subjectively by a human being, the limitations
of the model can be accounted for easily. For example, if it is known that there are certain inflection points in the lattice strains that occur due to grain reorientation during twinning, then a human can choose to ignore how well a set of parameters reproduces such an inflection in favour of paying more attention to other features. The GA has no such working knowledge of the strengths and weaknesses of the model it is optimizing, and so it is at a disadvantage when trying to optimize parameters where the limitations of the model play a larger role.

To test this theory, a fit was attempted using the newer EPSC4 model, which can account for grain reorientation and stress relaxation due to twinning [50]. The values for the parameters obtained from EPSC4 are shown in Table 3.4. The values for $\tau_0$ did not differ from the EPSC3 values by more than approximately 7%. The $\theta_0$ values, however, showed a much larger change, with the basal and pyramidal values changing by 30%, and the tensile twinning value increasing by nearly two orders of magnitude when the EPSC4 model was used. Furthermore, the error value used by the GA to judge goodness of fit between the model and experiment improved by approximately 4% compared to the EPSC3 results.

### 3.6.4 Fitting to Subsets of the Experimental Data

In order to determine the size of the dataset required for fitting, the GA was run on different subsets of the data in addition to the full dataset. Determining the minimum size of the data set required to achieve an acceptable fit from EPSC is potentially useful, given the high value of beam time for diffraction experiments. There were
three different subsets tested: one set that only contained lattice strains in one Poisson direction instead of two, another set that contained only data from compression tests, and a third that only contained compression data and only used one Poisson direction when measuring lattice strains. As shown in Table 3.4, the values found for the $\tau_0$ work hardening coefficients are relatively insensitive to the size of the dataset, most varying by approximately 10% or less, with the exception being the value associated with tensile twinning. It is believed that genetic algorithm has an easier time fitting the tensile twinning parameters when smaller subsets of the data are considered, because there are fewer lattice strain curves that contain phenomena that EPSC is incapable of simulating. Of course any optimization technique should experience performance increases when fitting to smaller dataset, since the degrees of freedom have remained the same.

Based on this analysis it appears that for a plate symmetry textured hcp material, a set of three neutron tests (with loading in the plate principal directions, and measurements parallel and in one perpendicular direction) is sufficient to obtain a reasonable set of single crystal plasticity parameters. It should be remembered that in this analysis that changes in the interaction between slip systems was ignored - in the case where this interaction needs to be tuned, a larger data set, and presumably a better materials model will need to be included.
Table 3.4: Hardening parameters obtained while fitting against various subsets of the experimental data, using the EPSC-3 model [45], and for the whole dataset using the EPSC-4 model [50]

<table>
<thead>
<tr>
<th></th>
<th>Full Dataset</th>
<th>Compressive Tests Only</th>
<th>1 Poisson Direction</th>
<th>Compressive Tests, 1 Poisson Direction</th>
<th>Full Dataset (EPSC4)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\tau_0$ (GPa)</td>
<td>$\theta_0, \theta_1$</td>
<td>$\tau_0$ (GPa)</td>
<td>$\theta_0, \theta_1$</td>
<td>$\tau_0$ (GPa)</td>
</tr>
<tr>
<td>Prism $&lt; a &gt;$ slip</td>
<td>0.101 0.111</td>
<td>0.099 0.014</td>
<td>0.092 0.013</td>
<td>0.091 0.018</td>
<td>0.094 0.008</td>
</tr>
<tr>
<td>Basal $&lt; a &gt;$ slip</td>
<td>0.149 0.049</td>
<td>0.134 0.056</td>
<td>0.159 0.030</td>
<td>0.144 0.050</td>
<td>0.160 0.035</td>
</tr>
<tr>
<td>Pyramidal $&lt; c + a &gt;$ slip</td>
<td>0.347 0.494</td>
<td>0.345 0.490</td>
<td>0.348 0.497</td>
<td>0.344 0.490</td>
<td>0.335 0.496</td>
</tr>
<tr>
<td>Tensile Twinning</td>
<td>0.178 0.003</td>
<td>0.209 0.006</td>
<td>0.215 0.003</td>
<td>0.217 0.011</td>
<td>0.181 0.115</td>
</tr>
</tbody>
</table>

### 3.7 Conclusions

A genetic algorithm was applied in order to solve the problem of optimizing the performance of an elastoplastic self-consistent polycrystalline plasticity model. The parameters optimized were the work hardening coefficients for the active slip and twinning systems in Zircaloy-2. The model results were fit to a large experimental dataset consisting of macroscopic flow curves in all three principal directions, as well as lattice strains measured both parallel and perpendicular to the loading axis, and Lankford Coefficients. The results obtained were comparable in quality to those obtained during a previous attempt to optimize the same parameters manually.

The genetic algorithm failed to produce satisfactory results for the hardening parameters associated with tensile twinning, whereas during the manual optimization procedure the twinning behaviour of the material was more accurately predicted. This is believed to be due to the subjectivity involved in judging the goodness of fit manually, and the fact that the person performing the optimization had a working
knowledge of the limitations of the twinning model utilized by the plasticity model. No such information was available to the genetic algorithm, resulting in it attempting to fit features in the lattice strain curves that the plasticity model was not equipped to deal with. Although the results obtained through use of the algorithm were not ideal, the fact that it is able to produce results comparable to those found by a human being in a fraction of the time means that it could be of great use in optimizing model parameters, and it is believed that if combined with a more accurate model then the algorithm’s performance would be significantly improved.
Chapter 4

Effect of Loading Mode on Lattice Strain Measurement

The study of lattice strain evolution during uniaxial deformation via in situ neutron diffraction is a well established technique for characterizing the deformation behaviour of metals. However, the relatively low flux of neutron facilities results in count times on the order of several minutes, requiring experimenters to choose between either applying a very slow strain rate, or loading the sample incrementally rather than continuously. Here we investigate the effects on lattice strain data brought on by using stress, strain, and position controlled incremental loading, as well as continuous loading, on samples of Zircaloy-2 under uniaxial compression. It was found that both qualitative and quantitative differences in lattice strain behaviour can occur just after the onset of yielding. The differences in lattice strain evolution brought on by the variation in loading modes are believed to be the result of thermally activated dislocation motion.
CHAPTER 4. EFFECT OF LOADING MODE ON LATTICE STRAIN MEASUREMENT

4.1 Introduction

Diffraction studies utilizing either neutrons or x-rays are a common method of characterizing the internal strain state of crystalline solids. Such experiments are often performed in situ under various thermal and mechanical loading conditions. Neutron diffraction studies are particularly useful due to the high penetration depth of neutrons in many metals; this high penetration allows for a large number of grains to be sampled, leading to improved statistics and greater precision in measuring the internal strains. The trade-off associated with this advantage is the fact that neutron sources have significantly lower flux than synchrotron x-ray sources, which can result in measurement times on the order of tens of minutes per data point.

The typical way to accommodate the slow rate of data collection during neutron diffraction is to load the sample incrementally, so that it may be held at a constant state throughout the neutron scattering process [2]. This solution functions well during purely elastic deformation, but once plastic deformation begins, complications will arise due to relaxation of the material. For example, if the sample is deformed incrementally and held at constant strain during measurement, then the stress will drop over time; inversely, if held at a constant stress, then the strain will increase (creep) over time, even at room temperature. This room temperature “creep” is seen in many HCP (Zr, Mg, Ti) and FCC (Fe,Al) metals [77, 78]. A third option is to hold the actuator heads of the load frame at constant position, but this results in relaxation in both stress and strain. Each of these methods (referred to as strain control, stress control, and position control, respectively) are routinely used by researchers conducting in situ neutron diffraction experiments, but to date there has
been no study to determine what effect the different loading modes offered by each has on the lattice strain evolution of HCP materials.

The mechanisms behind room temperature creep were explored in a recent TEM study [79] performed on samples of pure titanium and magnesium, and showed that room temperature creep is caused primarily by the activation of a single slip system within the affected grains. The authors concluded that creep occurred within the material due to slow work hardening occurring due to weak interactions between dislocations. Furthermore, the formation of deformation twins within the material was found to result in reduced creep behavior, due to the twins acting as a barrier to dislocation motion. Similar mechanisms have likewise been shown to result in the relaxation of stress in polycrystalline materials held at a constant strain. If a material is loaded and held at a constant strain, the stress will drop over time due to thermally activated dislocation motion [80, 81, 82]. Over time, dislocations within the material that have become pinned by obstacles such as grain boundaries, precipitates, or other dislocations, will free themselves and potentially annihilate, reducing the overall energy state of the material. The amount of time it takes the material to relax is dependent on the number of dislocations which are in motion and their velocity, which is in turn controlled by the obstacles within the material, temperature, and the resolved shear stress in the grain [82].

One could also perhaps consider this relaxation in terms of the classical division between a thermal and athermal contribution to dislocation resistance [83, 84]. The athermal resistance is considered to arise due to the interaction of the moving (slip)
dislocations with long range obstacles. Examples include dislocations on parallel slip planes, grain boundaries and second phase particles. The athermal resistance to dislocation motion depends on temperature via the temperature dependence of the shear modulus. The thermal resistance to dislocation motion arises from the interaction of dislocations with shorter range obstacles, and is influenced by both temperature and strain rate. These shorter range obstacles include solute atoms, clusters, point defects and forest dislocations. Thus the relaxation following a rapid change in applied strain can be considered as a move from the thermal plus athermal resistance that is initially observed, towards the athermal resistance alone that would be observed at very slow strain rates.

Zircaloy-2 is used in structural components essential to both heavy and light water nuclear reactors [4]. Creep and growth of these structural components within a reactor can lead to serious problems during operation, such as contact between the pressure tube and calandria tube in CANDU reactors. Such deformation can be correlated with the evolution of internal stresses and strains in the material [85]. For this reason, several x-ray [55] and neutron [9, 2, 86, 87] diffraction studies have been conducted on Zircaloy-2 and other hcp materials in order to characterize the evolution of these internal strains during deformation.

The composition of Zircaloy-2 is nominally of the following elements: Sn 1.2-1.7wt%, Fe 0.07-0.2wt%, Cr 0.05-0.15wt%, Ni 0.03-0.08wt%, 1400wppm oxygen, Zr (balance) [4]. At room temperature, and under operating conditions typical to a nuclear reactor core the alloy has a hexagonal close packed (hcp) structure and
thus exhibits elastic, plastic, and thermal anisotropy. During plastic deformation of zirconium alloys it has been previously shown [17, 21, 20, 29] that \(< a >\) slip \(\{10\bar{1}0\} < 11\bar{2}0 >\) is the most easily activated deformation mode, followed by basal \(< a >\) slip \(\{0001\} < 11\bar{2}0 >\), then pyramidal \(< c + a >\) slip \(\{10\bar{1}1\} < 11\bar{2}3 >\) or tensile twinning \(\{10\bar{1}2\} < 10\bar{1}1 >\) and finally compressive twinning \(\{11\bar{2}2\} < 11\bar{2}3 >\), although compressive twinning is usually thought to only be active at cryogenic temperatures [88]. The precise relative activities of these various deformation modes determine the overall stress-strain response of the material.

Tensile twinning is unique among the deformation modes of Zircaloy-2 in that unlike slip, it is a one way process (i.e. only occurs when the \(c\)-axis of the parent grain to twin is in tension) [89], and it results in a dramatic \(\sim 86^\circ\) reorientation of the crystal lattice within a subsection of a grain. In basic terms, when the material is loaded in compression the twinning process occurs in one of the Poisson directions, resulting in the reorientation of the “parent” lattice to a plastically harder twinned orientation. As the external load is transferred to the new plastically hard twin, the entire parent grain may reorient itself into a twin [90]. If instead tensile twinning is activated in a sample under tension then the newly twinned grain has an orientation that makes slip easier to occur, rather than harder. Thus, under compressive loading, twins begin as a relaxed, plastically hard “inclusion” within the surrounding matrix and end up as areas of very high stress within the material as the load shifts to them throughout deformation. Twins contribute to the overall work hardening of the material through this inclusion-like process [91], and by acting as barriers to dislocation movement.
In this paper we present a comparative study of stress control, strain control, and position control as methods of conducting in situ uniaxial compression tests on samples of highly textured Zircaloy-2 cut from a rolled plate. The lattice strain evolution of this material has been well characterized via neutron diffraction for all three principal directions under both compression and tension [2]. The findings from these earlier experiments were used in conjunction with self-consistent modeling to investigate the relative activities of the various slip and twinning modes present in zirconium alloys [10]. It was found that due to the texture of the material, the mechanism for accommodating plastic strain along the grain c-axes varied depending on which material direction the stress was applied in. When the samples were compressed along the plate normal direction, there was no evidence of significant tensile twinning, and pyramidal \( <c+a> \) slip was found to reach approximately 20% of the overall deformation activity at stresses greater than 500MPa. On the other hand, samples loaded in the RD direction showed significant grain reorientation due to tensile twinning and an absence of pyramidal \( <c+a> \) slip. These differences in deformation mechanisms and lattice strains also manifested in the macroscopic flow curves of the material, which showed significantly different yield and work hardening behaviour for the normal and rolling directions. The transverse direction behaved in a way that was a combination of the two, and so in light of limited beam time and sample material we will only be considering loading along the normal and rolling directions in the present study, since they represent the two extreme cases of material behaviour.
4.2 Experiments

4.2.1 Material

The material used was a warm rolled plate of Zircaloy-2. Previously performed texture measurements have shown that the basal plane normals are oriented primarily in the normal direction with a spread of ±50° towards the transverse direction (TD) and ±30° towards the rolling direction (RD) [8]. This pattern implies that more basal normals can be observed in the TD than the RD. The \{10\overline{1}0\} normals are weakly clustered at approximately 30° away from the RD. Figures 4.1(a) and 4.1(b) show the pole figures for the \{10\overline{1}0\} and \{0002\} directions in the undeformed material. Basal texture was found to vary significantly through the thickness of the plate, but was uniform for ~20mm in the mid-thickness. Therefore care was taken to cut the samples from this region of the plate.

![Pole figures](image)

Figure 4.1: Pole figures of undeformed samples for the \{10\overline{1}0\} (a) and \{0002\} (b) normal directions. The vertical axis represents the rolling direction and the horizontal axis is the transverse direction.

Cylindrical compression samples with a diameter of 9mm and a length of 22mm
were machined from the plate in two of the three principal directions. The directions chosen were the ND and RD, which contain the highest and lowest concentration of basal normals, respectively. By choosing the principal directions with the greatest difference in texture it was hoped that any effects of texture on loading and/or relaxation would be readily identifiable during the experiments. The dimensions of the samples were chosen so that a sufficiently large scattering volume would be present with the most uniform texture possible, and so that the sample would be long enough that the load frame would not block any part of the neutron beam during compression.

4.2.2 Uniaxial Compression Tests

A total of eleven *in situ* compression tests were performed using the time-of-flight neutron scattering technique at the Spectrometer for Materials Research at Temperature and Stress (SMARTS) beamline at the Los Alamos Neutron Science Center (LANSCE). During a test the sample is placed in the load frame, which is aligned such that the loading axis is set horizontally and rotated 45° to the incident neutron beam e.g., [92]. This alignment allows for measurements of lattice strain to be made parallel and perpendicular to the loading axis. Two detector banks located at scattering angles of ±90° collect the Bragg diffraction spectrum within the d-spacing range of 0.42 to 3.88 Angstrom. The sample was held in the load frame with a small initial stress of -10MPa, which has a negligible effect on lattice strain evolution, but is useful for holding the sample in place and preventing it from becoming misaligned. An extensometer with an initial gauge length of 10mm and a full scale reading of 10% was attached to the midpoint of the sample’s length with razor blades to measure the
accumulated macroscopic strain. To prevent the edge of the beam from scattering off the extensometer a Cadmium plate coated in gadolinium oxide paint was placed between it and the incident beam.

All the internal strains measured by neutron diffraction are relative to the initial measurement taken at -10MPa and thus do not take into account any thermal strains introduced during manufacturing, but it will be important to account for those strains during any future modeling. All incremental loading tests began in stress control and remained that way until the stress reached -150MPa. This was done because stress control is the more stable at low loads than strain or position control, and since the material is still behaving elastically at these low loads, there should be a negligible impact on lattice strain development. In addition to the incremental loading tests, samples were deformed continuously at two different strain rates to observe effects of strain rate variation independent of loading mode, using the same setup and load frame for consistency.

In order to ensure a valid comparison between the various tests, care was taken to make certain that all of the tests were completed in approximately the same time frame. Both the measurement time for each data point and the time taken to increment the load on the samples between data points were kept constant throughout all of the incrementally loaded tests. The tests performed under continuous deformation were carried out at two different strain rates; one that represented the average strain rate of the incrementally loaded test performed under strain control ($\dot{\epsilon} = 7.34 \times 10^{-6}\text{s}^{-1}$), and another that was approximately three times slower ($\dot{\epsilon} =$...
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2.32 × 10^{-6} s^{-1}). The PID settings used to control the stress, strain, or position during testing are listed in Appendix 1.

The neutron diffraction data was collected in one of two ways. In the case of the continuously deformed samples, neutron data was collected for six minutes at a time and each of these six minute bins corresponded to a single data point. This process of counting neutrons for six minutes was simply repeated until the deformation was complete. The sample is changing its state during the test as loading occurs, and the measurement is a time average of the sample state. In the case of the incrementally loaded samples, a slightly different approach was taken. At each increment (of stress, strain, or position), the sample was held for one minute before any neutron counting was carried out. This was done to allow the period of fastest relaxation to pass, so that the state of the sample would be relatively uniform during diffraction and is typical of the approach usually presently taken for such experiments. After this initial one minute wait, the sample was held for an additional six minutes while neutron data was collected, after which the sample was loaded up to the next increment. The measurement of each data point was based on time, instead of the total number of recorded neutron events, which is more commonly used in diffraction studies of this type. This was because the nature of the experiment made it extremely important that each sample be deformed over the same period of time, due to the time dependence of the various deformation and relaxation mechanisms involved. For example, if beam current were to be lost during a test it would be preferable to have the deformation of the sample continue and simply miss a few diffraction data points than to hold the sample at the current increment for several extra minutes while waiting
for the beam to be restored.

4.3 Results

4.3.1 Macroscopic Stress-Strain Data

The macroscopic flow curves for the tests performed in ND and RD are shown in Figures 4.2(a) and 4.2(b), respectively. All data points shown in the plot correspond to the time averaged stress and strain of the sample during a single neutron diffraction measurement. Note that all curves that stop short of 10% strain represent tests where the samples buckled during compression, resulting in the test being stopped prematurely. The fact that buckeling occurred in some samples will not have impacted the results as the samples that buckeled did so at strains above 5%, which is well into the plastic zone and allows for ample comparison of the data before that point. All data collected after buckeling occurred has been omitted from the results.

In both ND and RD, the macroscopic flow curves show little variation in behavior between the different test modes. This agreement between the various tests was important for the purpose of comparing the lattice strain data between samples. In addition to the overall agreement between curves, care was taken to choose increments in stress, position, or strain that were as appropriately spaced as possible so that the individual data points occur at similar average values of macroscopic stress and strain across the different tests. Due to relaxation in stress and/or strain during the incrementally loaded samples, this agreement grows worse with increasing plastic
Figure 4.2: Macroscopic stress-strain curves for samples loaded along ND (a) and RD (b).
strain, and it was not possible to achieve perfect agreement. The relaxation phenomenon is shown in Figure 4.3, where the complete macroscopic flow curve of the strain controlled sample compressed in ND is shown with no averaging of the data points.

The differences in stress at a given strain amongst samples in Figures 4.2(a) and 4.2(b) can be explained by examining the strain rates of the samples during the neutron measurement. For the samples deformed continuously at a constant strain rate, this is of course trivial. For the incrementally loaded samples, however, one must look at the change in strain that occurs during the times at which the samples are being held at a constant stress, strain, or actuator position. This strain rate during measurement may change for different measurements throughout the experiment, for example the stress controlled samples tend to creep more during measurements taken at high plastic strain as opposed to lower ones, so for the purposes of easy comparison an average for each sample is taken over all measurement periods. Table 4.1 lists the strain rates during measurement for all samples deformed in both ND and RD. Comparing the rates to the stress strain curves in Figure 4.2 shows that higher strain rates during neutron measurement correlates with a higher macroscopic stress. This makes sense, as the material is known to have a high strain rate exponent (in the range of 0.025-0.030 [93]) meaning that it is very rate dependent.
Table 4.1: Strain rates during neutron measurement.

<table>
<thead>
<tr>
<th>Test</th>
<th>Rate ($10^{-6}s^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Incremental Stress (ND)</td>
<td>9.65</td>
</tr>
<tr>
<td>Continuous (ND)</td>
<td>7.34</td>
</tr>
<tr>
<td>Slow Continuous (ND)</td>
<td>2.32</td>
</tr>
<tr>
<td>Incremental Position (ND)</td>
<td>1.53</td>
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<tr>
<td>Incremental Strain (ND)</td>
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</tr>
<tr>
<td>Continuous (RD)</td>
<td>7.34</td>
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<tr>
<td>Incremental Stress</td>
<td>5.55</td>
</tr>
<tr>
<td>Slow Continuous (RD)</td>
<td>2.32</td>
</tr>
<tr>
<td>Incremental Position (RD)</td>
<td>1.50</td>
</tr>
<tr>
<td>Incremental Strain (RD)</td>
<td>0.000842</td>
</tr>
</tbody>
</table>

4.3.2 Lattice Strain Development

Compression Along ND

The evolution of lattice strains in Zircaloy-2 under both tension and compression has been investigated in great detail previously [9, 2] and will be discussed here only as it relates to the main issue being explored. Figures 4.4 and 4.5 show the evolution of lattice strain for the $\{10\bar{1}2\}$ and $\{10\bar{1}1\}$ oriented grains measured parallel to loading in the samples compressed in the ND direction. Most of the elastic portion of the data has been truncated in order to better show the material response at and after yielding. It can be seen in both cases that all lattice curves follow a similar trend, but that two of the samples exhibited different initial yielding behavior. The sample loaded in strain control supports a slightly lower load after yielding than the others, while the sample compressed at a constant rate comparable to the average strain rates of the incremental tests was able to support a higher stress after yielding. The remaining tests performed under stress control, position control, and at a reduced strain rate, displayed yielding behavior very similar to one another. It should be
noted that one difference observed between the \{10\bar{1}2\} and \{10\bar{1}1\} oriented grains is the behavior at high stress; specifically the fact that the lattice strain data for the different tests converges at high stress in the \{10\bar{1}2\} oriented grains, but not in the case of the \{10\bar{1}1\} oriented grains.

Strains measured in the \{0002\} direction are shown in Figure 4.6 and were not found to vary significantly between samples. As will be seen in the rest of the experimental results, the grain orientations showing the greatest dependence on loading mode are those whose lattice strains deviate significantly from linearity when plotted against stress. Considering things qualitatively for all of the grain orientations that show this dependence, we see that differences among samples occur when there is a sharply defined inflection away from linearity near the yield stress (usually due to the activation of a slip system). The strains shown in Figure 4.6, however, show a much
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Figure 4.4: Lattice strain curves for the \{10\bar{1}2\} oriented grains plotted against macroscopic stress (a) and macroscopic strain (b) for samples loaded along ND.
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Figure 4.5: Lattice strain curves for the \{10\bar{1}1\} oriented grains plotted against macroscopic stress (a) and macroscopic strain (b) for samples loaded along ND.
smoother and more gradual divergence from linearity, and no variation is observed based on loading mode.

**Compression Along RD**

The grain orientations showing the most sensitivity to loading mode in their lattice strain development were the \{11\bar{2}0\} oriented grains measured along RD while the samples were being compressed in RD, as shown in Figure 4.7. The \{11\bar{2}0\} lattice strains go through two inflection points that occur at varying loads depending on the type of incremental loading and (in the case of continuous loading) the strain rate. With the exception of the stress controlled test, all of the samples showed the same general trend of a sharply defined inflection point at the point of yielding during which the slope of the curve goes from positive to negative, followed by a second inflection resulting in what appears to be a return to elastic deformation. In the stress controlled case, however, the inflection points are not as sharp and the curve maintains a positive slope at all times. This indicates that in the stress controlled test, the amount of elastic strain continues to increase, as opposed to the other cases in which it relaxed slightly with the onset of plasticity. As the deviation of the \{11\bar{2}0\} lattice strain curve from linearity is associated with the activation of prism \(< a >\) slip, this implies that the deformation mechanics of the various samples differ from one another.

The \{10\bar{1}0\} lattice strains measured parallel to loading (Figure 4.8) show results similar to that of the \{11\bar{2}0\} curves, though the differences among samples are less well defined. Still, it can be observed that the lattice strain evolution of the stress
Figure 4.6: Lattice strain curves for the \{0002\} oriented grains plotted against macroscopic stress (a) and macroscopic strain (b) for samples loaded along ND.
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Figure 4.7: Lattice strain curves for the \{11\overline{2}0\} oriented grains plotted against macroscopic stress (a) and macroscopic strain (b) for samples loaded along RD.

(a) Lattice Strain (microstrain)

(b) True Stress (%)
controlled sample differs from that of the position and strain controlled sample in a manner similar to what is observed in the case of the \{11\overline{2}0\} oriented grains.

Looking in a crystallographic direction outside of the basal plane we see different results from those observed in the \{10\overline{1}0\} and \{11\overline{2}0\} orientations. In the case of the \{10\overline{1}1\} strains measured parallel to loading (Figure 4.9), the trend and magnitude of the lattice strains show little variation between samples, indicating that these grain orientations are much less sensitive to loading mode. The amount of deviation from linearity in the plots is also lower. The \{0002\} oriented grains measured in this orientation, shown in Figure 4.10, represent grains that have twinned and thus been reoriented to have their \{0002\} poles shifted from either the transverse or normal direction into the rolling direction. For this reason, there is no data available for \{0002\} until after the point of yielding. In order to calculate the lattice strains for these grain orientations, the initial \(d\) spacing of the \{0002\} planes had to be found. The lack of \{0002\} poles initially in the rolling direction made this non-trivial, since there was no \{0002\} peak to measure the position of in the undeformed samples. This problem was solved by performing a Rietveld refinement on the undeformed spectrum, fitting all of the existing peaks and obtaining the values for the \(a\) and \(c\) lattice spacings. The spacing between \{0002\} planes was then equal to half the \(c\) value. The behavior of twinned grains appears very similar for all samples, with each showing the same linear relationship between applied stress and lattice strain.
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Figure 4.8: Lattice strain curves for the \{10\bar{1}0\} oriented grains plotted against macroscopic stress (a) and macroscopic strain (b) for samples loaded along RD.

(a) Stress (MPa) vs. Lattice Strain (microstrain)

(b) True Strain (%) vs. Lattice Strain (microstrain)
Figure 4.9: Lattice strain curves for the \{10\bar{1}1\} oriented grains plotted against macroscopic stress (a) and macroscopic strain (b) for samples loaded along RD.
Figure 4.10: Lattice strain curves for the \{0002\} oriented grains plotted against macroscopic stress (a) and macroscopic strain (b) for samples loaded along RD.
4.3.3 Peak Intensity

The fact that the loading mode dependence of the lattice strain curves is greater in samples loaded along RD than ND suggests that the texture may be playing a role. The main difference in the micromechanical behavior of Zircaloy-2 when compressed in RD as opposed to ND is the activity of tensile twinning, which is practically non-existent when the material is compressed along ND. To this end, the evolution of the texture of the various samples was investigated by measuring the relative intensities of certain diffraction peaks throughout deformation. By correcting for factors that vary with different lattice reflections such as multiplicity, structure factor, and wavelength, these intensities may be converted into the approximate volume fraction the material occupied by a given grain orientation [94]. Similarly, this technique may also be used to calculate the texture coefficients of the various grain families by dividing this volume fraction by what the volume fraction would be if the material were divided equally amongst all measured grain orientations. These calculations assume that there are no grains within the gauge volume which are not detected via diffraction.

Figure 4.11 shows the texture evolution of the \{10\bar{1}0\} and \{0002\} grain families for all tests performed under compression in RD. The plots have been normalized so that all tests show the same initial texture, even though measurements indicate different initial texture coefficients due to slight sample to sample texture variation (see Table 4.2). This was done to make the differences in texture development due to loading more obvious. Although such plots do not give as much texture information as proper pole figures, some general inferences about the texture development of the
material under stress can be made. There appears to be little variation in the overall behavior of the texture evolution, with all the samples following similar trends in Figures 4.11 (a) and (b). There is, however, some variation in the magnitude of the texture coefficient amongst different samples at a given level of stress. Differences in texture begin to appear only after the point of yielding and are most clearly visible in Figure 4.11(a). For example, the texture coefficients of the \{10\bar{1}0\} oriented grains all show that there is a clear critical stress at which the grains begin to reorient themselves; this critical stress shows some variability between samples, but other than that the trend is the same for all samples. Compare this to the \{0002\} texture coefficients plotted in the same figure; here there is a clear difference in the rate of change in the texture coefficient with stress, rather than the same process simply starting at a different stress as with the \{10\bar{1}0\} oriented grains.

Table 4.2: Initial texture coefficients of samples compressed in RD.

<table>
<thead>
<tr>
<th>Test Type</th>
<th>RD</th>
<th>ND</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0002</td>
<td>10\bar{1}0</td>
</tr>
<tr>
<td>Stress</td>
<td>0.028</td>
<td>2.037</td>
</tr>
<tr>
<td>Strain</td>
<td>0.054</td>
<td>1.995</td>
</tr>
<tr>
<td>Position</td>
<td>0.022</td>
<td>1.913</td>
</tr>
<tr>
<td>Continuous</td>
<td>0.031</td>
<td>1.929</td>
</tr>
<tr>
<td>Slow Continuous</td>
<td>0.024</td>
<td>2.047</td>
</tr>
</tbody>
</table>

Since there are initially no \{0002\} poles oriented in the rolling direction, the volume fraction of twins can be estimated by calculating the volume fraction of \{0002\} oriented grains detected in that orientation as the sample is deformed. Although the calculations performed showed a very small (~0.1\%) initial volume fraction of \{0002\} oriented grains in the undeformed samples, the data was simply offset by this amount to give an initial twin volume fraction of zero. The growth of the total twin
volume fraction with increasing stress and strain is shown in Figures 4.12(a) and 4.12(b), respectively. The fact that there is less divergence among samples when the volume fraction is plotted against true strain suggests that the amount of twinning is controlled by the plastic strain in the sample and not the applied stress. This contradicts the results of a recent study done on Beryllium [95] in which it was observed that twin volume fraction was a function of the applied stress and not the total plastic strain.

4.4 Discussion

The results of experiments performed show evidence of deformation mode activity being dependent on loading mode to some extent. Because the investigation of deformation mechanics is a major application of diffraction studies in metals [86, 87, 96, 64], it is important for this phenomenon to be properly quantified. Although strain rate effects are often taken into account when performing in situ diffraction studies of samples under uniaxial deformation, the effects of the various relaxation behaviors of stress, strain, and position controlled loading modes are often assumed to be negligible. This assumption gives a valid first order approximation when considering lattice strain evolution qualitatively, but when looking at things quantitatively differences can be found. There are certain circumstances when even qualitative agreement is not reached, specifically in cases of stress relaxation.

The onset of certain inflection points in the plots of stress vs lattice strain were observed to occur at significantly different values of stress for the various tests. Since these inflection points are thought to correspond to the critically resolved shear
stresses of different deformation modes being reached, this indicates that the deformation mechanics vary from test to test. For example, in the \{11\bar{2}0\} lattice strains plotted in Figure 4.7 there is a spread of 51MPa in the applied stress at which the first inflection occurs under strain control and the stress at which it occurs during continuous deformation at the slower strain rate, with all the other tests falling somewhere between these two extremes. In the same plot, the applied stress at which the second inflection point occurs varies by 40MPa amongst the tests. This particular grain family also demonstrates the instance in which even qualitative differences can be seen amongst the tests, since all of the tests except for the one performed in stress control show stress relaxation in the \{11\bar{2}0\} oriented grains.

The sample compressed in RD under stress control shows different behavior in the evolution of lattice strain in the \{11\bar{2}0\} and \{10\bar{1}0\} grain families. While all of the other grain orientations show a clear relaxation in lattice strain with increasing macroscopic stress past the initial inflection point in the plots, in the stress controlled sample the lattice strain is always increasing. The reason for this stems from the different relaxation processes occurring during the “hold” phase of the experiment, which occurs when the sample is being held at a constant stress, strain, or position while neutron counting is done. The sample held at a constant stress will continue to accumulate plastic strain until enough dislocations have been generated to harden the material to the point where it can support the stress being exerted without further plastic strain. The driving force created by the constantly maintained external stress ensures that dislocations are being generated more quickly than they can annihilate with each other. In strain or position control, however, the driving
force for dislocation motion is not constant. Since the motion of dislocations around obstacles (and each other) is a thermally activated process, over time more and more dislocations will move into a configuration that allows them to annihilate or reach a stable configuration. The important distinction is that unlike what occurs under stress control, as the dislocations begin to annihilate and rearrange the driving force to create new ones decreases. This results in the dislocation density within the grain dropping during each hold phase, rather than continuing to increase or reaching a steady state as it does while the sample is in stress control.

4.5 Conclusions

Distinctions were found in the lattice strain evolution of cylindrical compression samples of Zircaloy-2 during in situ neutron diffraction experiments subjected to different loading modes. Incremental loading experiments in steps of constant stress, strain, and position were carried out, as well as continuous loading at two different strain rates. The variations in lattice strain were found to be texture dependent and occurred most noticeably in samples compressed along the rolling direction, which contained the smallest concentration of basal poles.

Grain orientations most prone to showing dependence on loading mode were those that showed large amounts of plasticity, such as \{11\overline{2}0\} and \{10\overline{1}0\}. Plastically harder grain orientations which showed relatively little plastic behavior, such as \{0002\} were found to be unaffected by loading mode. The observed variations in micromechanical behavior are thought to be due to thermally activated dislocation motion during times
CHAPTER 4. EFFECT OF LOADING MODE ON LATTICE STRAIN MEASUREMENT

when the material is being held at a constant point during the incremental loading tests. During this time the material relaxes in either stress, strain, or both (for strain control, stress control, and position control, respectively), and the different relaxation behavior is thought to result in differing dislocation mobility.

The results suggest that caution must be exercised when drawing comparisons between material data obtained from tests using different loading methodologies and strain rates.
Figure 4.11: Texture coefficients of \{10\bar{1}0\} and \{0002\} oriented grains measured in RD (a) and ND (b), during loading in RD.
Figure 4.12: Plot of total twin volume fraction with increasing stress (a) and strain (b).
Chapter 5

Self-Consistent Modeling of Loading Mode Effects

5.1 Introduction

Elastoplastic self-consistent (EPSC) models have been widely used to calculate internal stresses of polycrystalline materials during thermomechanical loading [45, 87, 50, 10]. Unfortunately, such models are rate-independent and thus cannot account for dynamic changes in the loading conditions imposed upon the material. Conversely, viscoplastic self-consistent (VPSC) models use a rate-dependent formulation, but are unable to calculate internal stresses since they lack the ability to account for elastic effects. The benefits of the two approaches have been unified with elasto-viscoplastic self-consistent (EVPSC) models which are capable of calculating internal stresses and accounting for time dependent effects [43, 97, 47, 98].

The ability of EVPSC models to calculate internal stresses on a dynamically loaded
sample makes it an ideal model for the purpose of investigating *in situ* neutron diffraction experiments involving uniaxial deformation. One such set of experiments was recently conducted in order to investigate the effects of different loading modes on Zircaloy-2 [99]. Specifically, the impact on lattice strain development was studied for samples compressed incrementally under stress control, strain control, and position control. These results were also compared to samples that had been deformed continuously. Lattice strain development was found to be partially dependent on the type of loading the samples were subjected to. In this paper we attempt to fit model results from EVPSC [47] and EPSC [45] to the experimentally measured lattice strains measured for samples under stress control, strain control, and continuous deformation. The data gathered under position control in [99] was not included because there is no convenient way to simulate actuator position using the EVPSC model.

Zircaloy-2, like many zirconium based alloys, is used extensively in the nuclear industry. It is made up primarily of zirconium (>98 wt%) with a hexagonal close packed (HCP) structure which results in anisotropic thermal, plastic and elastic properties. During plastic deformation of zirconium, prism $<a>$ slip $\{10\bar{1}0\} <11\bar{2}0>$ is the most easily activated deformation mode [17, 21, 20], followed by basal $<a>$ slip $\{0001\} <11\bar{2}0>$, pyramidal $<c+a>$ slip $\{10\bar{1}1\} <11\bar{2}3>$, tensile twinning $\{10\bar{1}2\} <10\bar{1}1>$ and then finally compressive twinning $\{11\bar{2}2\} <11\bar{2}3>$. Although prism $<a>$ slip can accomodate strains in only the $a$ direction, both pyramidal $<c+a>$ and twinning are able to accomodate strains in both the $a$ and $c$ directions.
5.2 Summary of Experiments

The experiments were a series of in situ uniaxial compression tests carried out at the Spectrometer for Materials Research at Temperature and Stress (SMARTS) apparatus at the Los Alamos Neutron Science Center (LANSCE). The full details of the experiments have been reported elsewhere [99] and will only be described in brief here.

The material used was a warm rolled plate of Zircaloy-2. Previously performed texture measurements [8] have shown that the basal plane normals are oriented primarily in the normal direction with a spread of $\pm 50^\circ$ towards the transverse direction (TD) and $\pm 30^\circ$ towards the rolling direction (RD). This pattern implies that more basal normals can be observed in the TD than the RD. The $\{10\overline{1}0\}$ normals are weakly clustered at approximately $30^\circ$ away from the RD. Pole figures of the undeformed material have been published previously [8] and will not be reproduced here. Basal texture was found to vary significantly through the thickness of the plate, but was uniform for $\tilde{2}0$mm in the mid-thickness. Therefore care was taken to cut the samples from this region of the plate.

Cylindrical compression samples were machined from this material in two different orientations. One set of samples was cut with the rolling direction of the slap oriented axially in the cylinder, and another set was cut with the normal direction oriented along the cylinder’s axis. This allowed for the influence of texture on the results to be taken into account, since the RD and ND contain the lowest and highest concentrations of basal poles, respectively.
The experiments themselves involved compressing the samples to approximately 10% strain. In some cases the samples buckled before this level of macroscopic strain could be reached; for these tests, all data after the point of buckling was discarded in any analysis. Experiments were carried out compressing the samples both continuously at a constant strain rate, and by incremental deformation under stress control and strain control. This was done for both the RD and ND samples. In both sample orientations, the strain controlled test was performed first, and the stress data collected during that test was used to determine the equivalent increments in stress for use in the subsequent tests, such that data points would be taken at similar increments along the stress-strain curve.

All data was collected by neutron diffraction with the gauge volume centered at the midpoint of the sample. Lattice strains were calculated using equation 5.1 to find the lattice strains with the measured peak shifts; where \( \epsilon_{hkit} \) is the lattice strain of the grain family \( hkit \); \( d_{hkit}^{ref} \) and \( d_{hkit} \) are the plane spacing before and after deformation; and \( \theta_{hkit}^{ref} \) and \( \theta_{hkit} \) are the diffraction angles before and after deformation.

\[
\epsilon_{hkit} = \frac{d_{hkit} - d_{hkit}^{ref}}{d_{ref}}
\]  

(5.1)

All plotted lattice strains are calculated with respect to the start of each test, and thus represent only the increment produced by loading. It should also be noted that the lattice strain is the average response of all grain orientations that satisfy Bragg’s law for a particular \( hkit \) in the gauge volume while neutron data is being collected.
5.3 Self-Consistent Modeling

Simulations were performed using EVPSC [47] and EPSC [45] in an attempt to recreate the data observed in the experiments performed at a constant strain rate, stress control, and strain control. This was accomplished easily for EVPSC, since it allows for the user to select what the control variable will be for a given thermomechanical process, as well as the amount of time said process will take. By simply examining the data collected during the experiments, profiles of the loading mode for each experiment were programmed into EVPSC so that the loading conditions imposed upon the simulation matched those of the physical samples exactly. For the simulations involving EPSC, the sample deformation was simulated from zero to 10% strain, since following the actual dynamic loading mode was not possible using the time-independent EPSC code and no difference is obtained for stress or strain control. For both models, an initial step was included before deformation to simulate cooling from 898K to 298K in order to account for thermal strains present in the samples due to material processing [46].

5.3.1 EVPSC Hardening Equations

EVPSC uses a dislocation-based hardening model to control the work hardening behavior (the derivative of CRSS with respect to accumulated shear) of the slip and twinning systems in the material. A thorough discussion of the hardening equations is given in [47] and so only a brief description will be given here.

The hardening of plastic slip systems is considered to receive contributions from
resistance due to stored dislocations, and from the Hall-Petch effect created by twin and grain boundaries. Thus, the critically resolved shear stress (CRSS) of a given slip system can be written as shown in equation 5.2; where $g$ is the index of a given slip system, $\tau_c^g$ is the critically resolved shear stress, $\tau_{dis}^g$ is the contribution due to stored dislocations, and $\tau_{hp}^g$ is the contribution of Hall-Petch type hardening.

\[
\tau_c^g = \tau_{dis}^g + \tau_{hp}^g \quad (5.2)
\]

The contribution of dislocation hardening to the critically resolved shear stress is calculated using equation 5.3. $\tau_0^g$ is the initial shear stress and represents the resistance due to solute atoms, $\chi$ is a material constant, $\mu$ is the effective shear modulus, $b^g$ is the magnitude of the Burgers vector of the dislocations of the $g$th system, $h$ is the matrix that describes the interactions between the different slip systems, and $\rho$ is the total dislocation density.

\[
\tau_{dis}^g = \tau_0^g + \chi \mu b^g \sqrt{h^{gh} \rho^h} \quad (5.3)
\]

The changes in dislocation density are a result of the competing mechanisms of storage and dynamic recovery and can be calculated using equation 5.4. The coefficients $k_1^g$ and $k_2^g$ are material parameters, and $\dot{\gamma}$ is the plastic strain rate in the material.
\[ \dot{\rho}^g = (k_1^g \sqrt{\rho^g} - k_2^g \rho^g) \left| \dot{\gamma}^g \right| \]  

(5.4)

The hardening contribution from the Hall-Petch effect is calculated using equation 5.5, where \( k_{hp} \) is the classical Hall Petch parameter defined for each deformation mode, and \( L^g \) is the directional mean free path of dislocations.

\[ \tau_{hp}^g = \frac{k_{hp}}{\sqrt{L^g}} \]  

(5.5)

Hardening due to tensile twinning is handled differently by the model than slip is. The critical shear stress is initially set to a reference shear stress, \( \tau_0 \), and from there the changes in it are calculated using equation 5.6. The terms \( k_{tw}^{lat} \), \( k_{tw}^{self} \), and \( a \) are the twin hardening parameters, and \( f \) is the twin volume fraction.

\[ \dot{\tau}_c^g = k_{tw}^{lat} \left( \sum_{h=1}^{N_{tw}} f^h a^h \right) \sum_{h \neq g} \dot{\gamma}^h + k_{tw}^{self} \left( \sum_{h=1}^{N_{tw}} f^h \right) \dot{\gamma}^g \]  

(5.6)

### 5.3.2 EPSC Hardening Equations

The hardening equations for EPSC use an extended Voce hardening relationship for each deformation mode which describes the evolution of the CRSS of a given deformation mode using equation 5.7 [48]. The index \( s \) refers to a given deformation mode, \( \tau_s \) is the instantaneous CRSS, \( \Gamma \) is the accumulated shear in a grain, \( \tau_0^s \) and \( \tau_1^s \) are the initial and final back-extrapolated CRSS values, and \( \theta_0^s \) and \( \theta_1^s \) are the initial and final hardening rates.
\[ \tau^s = \tau_0^s + (\tau_1^s + \theta_1^s \Gamma)[1 - \exp(-\theta_0^s \Gamma / \tau_1^s)] \] (5.7)

### 5.3.3 Fitting of Modelling Results to Experimental Data

One of the difficulties in using self-consistent modeling to recreate experimentally observed phenomena is the proper selection of hardening parameters. Zirconium based alloys such as Zircaloy-2 pose an especially complex problem due to the existence of four unique deformation modes which are also dependent on the texture of the material. Recently, a method has been developed to automate the selection of hardening parameters of a self-consistent model to attain an acceptable fit to experimental data [1]. A genetic algorithm (GA) is used to refine the hardening parameters until an acceptable fit is reached. The macroscopic flow curves, and lattice strains both parallel to the loading direction, and lattice strains in a single Poisson direction are used as the fitting criteria. For the sake of brevity, the algorithm will not be described here, as a full description of its operation is available elsewhere [1]. The GA was used to separately refine the hardening coefficients used by EVPSC and EPSC.

Several parameters for the three active slip systems in Zircaloy-2 were refined; as well as the twin hardening parameters for tensile twinning. It was discovered that one single set of EVPSC hardening parameters could be used to simulate the entire set of experiments (i.e. strain control, stress control, and continuous loading in both RD and ND for a total of 6 experiments). Tables 5.1 and 5.2 show the final values returned for slip and twinning after refinement was completed.
Table 5.1: Slip hardening parameters selected for modeling.

<table>
<thead>
<tr>
<th></th>
<th>Prism</th>
<th>Basal</th>
<th>Pyramidal</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\tau_0)</td>
<td>0.112</td>
<td>0.156</td>
<td>0.33992</td>
</tr>
<tr>
<td>(k_1)</td>
<td>2.17E8</td>
<td>5.44E8</td>
<td>9.39E8</td>
</tr>
<tr>
<td>(k_2)</td>
<td>193</td>
<td>168</td>
<td>106</td>
</tr>
<tr>
<td>(\dot{\gamma}_0)</td>
<td>0.000351</td>
<td>0.000381</td>
<td>0.000101</td>
</tr>
<tr>
<td>(n)</td>
<td>28</td>
<td>27</td>
<td>20</td>
</tr>
<tr>
<td>(k_{bp})</td>
<td>0.0374</td>
<td>0.0262</td>
<td>0.0128</td>
</tr>
</tbody>
</table>

Table 5.2: Twinning hardening parameters selected for modeling.

<table>
<thead>
<tr>
<th></th>
<th>Tensile Twinning</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\tau_0)</td>
<td>0.223</td>
</tr>
<tr>
<td>(k_{tot}^{tw})</td>
<td>0.907</td>
</tr>
<tr>
<td>(k_{self}^{tw})</td>
<td>0.111</td>
</tr>
<tr>
<td>(a)</td>
<td>0.5</td>
</tr>
<tr>
<td>(\dot{\gamma}_{tw})</td>
<td>0.151</td>
</tr>
<tr>
<td>(f^c)</td>
<td>0.0902</td>
</tr>
<tr>
<td>(\dot{\gamma}_{0})</td>
<td>0.000377</td>
</tr>
<tr>
<td>(n)</td>
<td>28</td>
</tr>
</tbody>
</table>

Likewise, the hardening parameters used by EPSC were refined by the GA (see Table 5.3 for values). Since EPSC has no rate dependence, it was decided to refine three different sets of hardening parameters; one each for continuous deformation, stress control, and strain control. Note that the data for compression along RD and ND was fitted simultaneously for each of these three cases. This was done not only for the sake of obtaining a better fit to each set of experiments, but also to see how well the rate-independent EPSC code could account for time-dependent effects by simply changing the hardening coefficients. Previous work has shown that simplifying the parameter search for EPSC by setting \(\tau_1 = 0\) and \(\theta_0 = \theta_1\) can still result in an acceptable fit and save on computation time [10, 1]. This assumption will therefore
be used here to reduce the already large amounts of time required to refine multiple sets of hardening parameters.

Table 5.3: Hardening parameters obtained while fitting the EPSC model [50] to the results.

<table>
<thead>
<tr>
<th>Test</th>
<th>Prism</th>
<th>Basal</th>
<th>Pyramidal</th>
<th>Tensile Twinning</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stress</td>
<td>0.097</td>
<td>0.168</td>
<td>0.151</td>
<td>0.348</td>
</tr>
<tr>
<td>Strain</td>
<td>0.090</td>
<td>0.108</td>
<td>0.133</td>
<td>0.346</td>
</tr>
<tr>
<td>Continuous</td>
<td>0.103</td>
<td>0.087</td>
<td>0.142</td>
<td>0.347</td>
</tr>
</tbody>
</table>

5.4 Results

5.4.1 Macroscopic Flow Curves

The experimentally measured macroscopic flow curves for the tests performed under stress control, strain control, and continuous deformation are compared to the results from the EVPSC model in Figure 5.1. Although the experimental data is not perfectly reproduced by EVPSC, the ability to approximate the loading behavior of all three loading modes in two principal directions from a single set of material parameters is non-trivial. Overall, EVPSC appears to predict macroscopic behavior more accurately for compression in the rolling direction than the normal direction. The strain controlled compression in the normal direction (Figure 5.1(b)) is particularly problematic, with the model consistently over predicting the amount of stress relaxation in the sample.
Figure 5.1: Experimental data and EVPSC calculations of the macroscopic flow curves in stress control (a), strain control (b), and continuous deformation (c).
The EPSC modeling results were also plotted against the experimentally measured macroscopic flow curves. Due to the fact that EPSC is incapable of reproducing any time-dependent effects such as relaxation, a time average of both stress and strain was taken of each step in the incrementally loaded samples, reducing each loading step into a single data point. The results of fitting EPSC to the data are shown in Figure 5.2. The EPSC model results fit the experimental data quite well, especially in the case of the samples compressed along RD. Note that unlike the EVPSC results, a different set of hardening parameters was used to obtain the model data presented in Figures 5.2 (a), (b), and (c).

5.4.2 Lattice Strain Evolution

The first lattice strains examined after fitting were those of the \{11\bar{2}0\} family of grains oriented in RD while the sample was compressed in RD. This was the plot in which the experimental data showed the greatest amount of variation between the different tests [99] and offers some insight into how well the model is able to capture these differences. Figure 5.3 shows the results of the fitting process for both EVPSC and EPSC.

The EVPSC model fails to reproduce any difference in the \{11\bar{2}0\} oriented grains between the stress controlled and continuously deformed samples. The strain controlled sample is well represented qualitatively, showing the same inflection points visible in the experimental data, but at the incorrect magnitude. Furthermore, the EVPSC data for the continuously deformed sample lacks the inflection point visible
Figure 5.2: Experimental data and EPSC calculations of the macroscopic flow curves in stress control (a), strain control (b), and continuous deformation (c).
in the experimental curve and is nearly indistinguishable in behavior from the stress
controlled sample. This is in stark contrast to the experimental data, which shows
the continuously deformed sample and the strain controlled sample to show qualita-
tively similar behavior, and the stress controlled sample being drastically different
from both. For all its faults, the EVPSC model still performs better than the rate-
independent EPSC model, which fails to predict any significant difference between
the three tests.

![Figure 5.3: Comparison of experimental data and model fitting results for the \{11\overline{2}0\} grain family measured along RD during compression in RD.]

Similarly, the \{10\overline{1}0\} oriented grains show differences in the experimental data
that are represented well qualitatively, but not quantitatively (Figure 5.4). Unlike
the results for \{11\bar{2}0\}, here we see that the differences between the tests in the EVPSC data closely resembles the experimental results. Specifically, the continuously deformed and stress controlled tests produced nearly identical data, while the strain controlled sample showed a less negative lattice strain measurement at a given stress level than the other two tests. Although this variation amongst the different tests is exaggerated in the EVPSC data, the general trend is preserved.

The EPSC model, on the other hand, still fails to account for the differences in lattice strain introduced by the variation in loading mode. The EPSC results do show a slight separation between the three tests just past the point of yielding, but it is much smaller than what is shown in the experimental results, and the simulated results from the three tests all seem to follow the trend of the continuous and stress controlled experimental data.

Looking outside of the basal plane at the \{10\bar{1}1\} strains, we see that EVPSC once again predicts that the strain controlled test should result in a significantly different lattice strain state than the continuously deformed or stress controlled tests (Figure 5.5). Unlike the results seen with \{10\bar{1}0\}, however, here the strain controlled sample’s behavior is best captured by the model, while the other two are less accurately reproduced. The results from EPSC, meanwhile, capture the small difference in lattice strain evolution well. EPSC correctly predicts that the strain controlled sample will have a slightly lower lattice strain value for a given stress past the point of yielding, but the magnitudes of the lattices strains are over predicted for all three tests by EPSC.
Figure 5.4: Comparison of experimental data and model fitting results for the \{1010\} grain family measured along RD during compression in RD.
Another important set of grains to consider during deformation are the newly reoriented grains that have undergone twinning. Due to the texture of the samples, there are initially no measurable grains oriented with their \{0002\} poles along the rolling direction. As twinning occurs, however, the associated grain reorientation results in more and more \{0002\} oriented grains aligning in this direction. Figure 5.6 shows the lattice strain development for these newly reoriented grains, along with the predictions from EVPSC and EPSC. As shown, there is no significant variation between the experimentally observed results for the various tests, but the predictions made by the models for the tests show good agreement only in the case of the EVPSC
simulation of strain control, with all the other modeling results overestimating the magnitude of the lattice strain in the grains.

Figure 5.6: Comparison of experimental data and model fitting results for the \{0002\} grain family measured along RD during compression in RD.

Looking at tests conducted under compression in ND, we see that the models provide roughly the same level of agreement as in RD. For example, Figure 5.7 shows the lattice strain development of the \{0002\} oriented grains in samples deformed in the normal direction. The experimental data shows that the loading mode has virtually no impact on the lattice strain evolution of these grains, but EVPSC once again shows different behavior in strain control than in the other two tests. The results obtained
from EPSC show results similar to the stress controlled and continuous EVPSC results, which match the general trend of the experiments, but are unable to predict the correct magnitude of the lattice strains.

Figure 5.7: Comparison of experimental data and model fitting results for the \{0002\} grain family measured along ND during compression in ND.

The \{10\overline{1}1\} oriented grains in the samples compressed along ND show poor agreement with the model predictions at stresses higher than -400MPa (Figure 5.8). The relaxation in lattice strain displayed in the experimental data for all three tests is not accounted for by EPSC or EVPSC, causing the predicted curves to begin diverging from the observed trend at this point. At stresses beyond approximately -550MPa, the relaxation has ended and the grains begin accumulating lattice strain once again.
From this point on, EVPSC provides a reasonable approximation of the experimental observations; although the magnitudes of the lattice strains in this region are over predicted, the slope and overall trend of the curves mirror those demonstrated by the experiments. Interestingly, the EPSC results for stress control and continuous deformation are closer in magnitude to the experimentally measured lattice strains, but at the same time do a poorer job of reproducing the shape of the curves. This is especially noticeable at high compressive loading where the concavity of the EVPSC curves matches that of the experimental data very well, but the EPSC results do not. This result makes sense if one considers that EVPSC is attempting to model the actual loading mode of the material, while EPSC is using non-rate dependent hardening parameters to model deformation occurring at a non-constant rate.

Grains oriented with their \{10\overline{1}2\} poles along the normal direction showed minor variation in lattice strain development during compression along ND. EVPSC adequately predicts the behavior of the strain controlled sample, although it demonstrates yielding sooner than is observed experimentally. The results from EPSC are less accurate in predicting the behavior of the strain controlled sample, but are on par with the EVPSC results up until the applied stress reaches approximately -50MPa. Beyond stresses of -500MP, the EVPSC results give a much better fit to the strain controlled data, as well as slightly better agreement with the other two tests.
Figure 5.8: Comparison of experimental data and model fitting results for the \{1011\} grain family measured along ND during compression in ND.
Figure 5.9: Comparison of experimental data and model fitting results for the \{10\bar{1}2\} grain family measured along ND during compression in ND.
CHAPTER 5. SELF-CONSISTENT MODELING OF LOADING MODE EFFECTS

5.5 Discussion

The overall trend observed in the model results of the lattice strain evolution is that EVPSC consistently shows the strain controlled tests developing lower magnitudes of lattice strain for a given applied stress. In some cases, this phenomenon is seen in the experimental data as well, though usually not to the same extent demonstrated by EVPSC. Unfortunately, the model also shows this segregation in situations where it is certainly not observed experimentally. EPSC also shows this relationship between the tests, but to a much smaller extent than is observed experimentally. It also fails to reproduce certain features and inflection points that EVPSC is able to at least partially account for, such as in the \{11\overline{2}0\} oriented grains compressed in RD.

5.5.1 Deformation Mode Activities

In order to investigate the possible mechanisms responsible for the differences in observed lattice strain behavior amongst the different tests, the relative deformation mode activities predicted by EVPSC were examined. Since EPSC was less successful in reproducing the distinctions in lattice strain behavior amongst the different samples, it was not used to analyze deformation mode activity.

The relative activities of all three slip systems as well as tensile twinning were plotted against applied stress in order to look for any changes in activity that correlated with regions where the lattice strain curves for the different samples were predicted to diverge from one another. For example, Figure 5.3 shows the strain controlled sample undergoing a sharp inflection point at approximately -300MPa. This was found to
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correspond to a sudden increase in the activity of tensile twinning (Figure 5.10) and a decrease in the prismatic slip activity (Figure 5.11), both of which occurred only during the strain controlled simulations. These findings were consistent with peak intensity measurements [99] that showed the strain controlled sample had a consistently higher twin volume fraction than the other samples at a given level of stress.

For the samples compressed in ND, there was also a point at which the strain controlled sample underwent an inflection point that was sharper than the ones demonstrated than the other samples. This is seen in Figure 5.8 at a stress of approximately -425MPa. At this level of applied stress in ND we see that pyramidal slip begins to become much more active at the expense of Basal slip, shown in Figures 5.12 and
Figure 5.11: Relative activity of the prismatic slip system during compression along RD
5.13, respectively.

![Figure 5.12: Relative activity of pyramidal $<c+a>$ slip during compression along ND](image)

**5.6 Conclusions**

Model results from both EPSC and EVPSC were fitted to experiments measuring the lattice strain evolution of Zircaloy-2 under uniaxial compression. Three different loading modes were considered in both the ND and RD directions: incremental loading in stress control and strain control, as well as continuous loading at a constant
Figure 5.13: Relative activity of the basal slip system during compression along ND
strain rate. It was found that EVPSC was able to fit all of the data simultaneously with a single set of hardening parameters, although the differences in lattice strain development between the continuous and stress controlled samples were not accurately described during modeling. This suggests that a dislocation evolution law that more appropriately describes the thermal/athermal division of dislocations is required. Since the EPSC model cannot simulate time-dependent loading, different hardening parameters had to be found for each loading mode applied to the samples, one each for stress control, strain control, and continuous loading.

Overall, the single set of EVPSC hardening parameters gave a better fit to the experimental data than the three separate EPSC simulations. Although the results from EVPSC were not perfect and failed to predict many differences in lattice strain development between the stress controlled and continuously deformed samples, it did a better job of predicting the overall behavior of the material. In particular, EVPSC was able to account for cases such as those seen in Figures 5.3 and 5.4, where the strain controlled sample shows an inflection point at -300MPa.

Deformation mode activity from the EVPSC simulations suggests a possible mechanism behind the experimentally observed lattice strain curves. Specifically, in the case of the strain controlled sample under compression in RD, there is a dramatic increase in the activity of tensile twinning and decrease in the activity of prism \(< a >\) slip at the level of stress at which the strain controlled sample’s \{11\bar{2}0\} and \{10\bar{1}0\}
lattice strain curves diverge from those of the stress controlled and continuously de-
formed samples. Under strain control in ND, it was found that an increase in pyra-
midal $< c+a >$ slip and decrease in basal slip gave similar results, though to a lesser
degree.
Chapter 6

Conclusions and Future Work

6.1 Conclusions

A genetic algorithm was successfully used to optimize the selection of hardening coefficients for use in self-consistent modeling of Zircaloy-2 under uniaxial tension and compression. Tensile and compressive tests in three material directions (rolling, transverse, and normal) were all simulated using a single set of hardening parameters, and the results were found to be comparable in accuracy to those obtained through manual refinement of the parameters using trial and error. The high parallelization available in the use of the genetic algorithm means that it can reach a solution within a small fraction of the time that it would take a human being to manually search the parameter space. The process is entirely automated once begun, and is not limited to any particular type of model, since it sees the system it is optimizing as a “black box” and is only concerned with the input and output. This numerical approach for parameter selection is therefore recommended to any researchers attempting to fit model results to experimentally measured lattice strains or macroscopic flow curves.
The relatively fast, automated nature of the approach has the potential to reduce the turnaround time on such studies significantly and to allow those involved in analyzing the data to focus more of their attention on interpreting the results of their work, and less on fine-tuning the model parameters.

The loading mode dependence of lattice strain measurement via neutron diffraction was investigated in Zircaloy-2. It was found that certain grain orientations show significantly varied lattice strain evolution based on whether the sample is held constant through stress control, strain control, or position control. The differences tend to arise near the point of initial yielding in the material, which is often a region of great interest during lattice strain studies, since it is associated with the activation of various slip and twinning modes. The main cause behind the variations in lattice strain amongst the different loading schemes is thought to be the increased amount of dislocations generated during stress control. Based on the results obtained here, it is recommended that any future studies in lattice strain evolution of Zr alloys make use of the same loading mode for all specimens that are intended to be directly compared to one another.

The EPSC and EVPSC models were both applied to the data comparing the lattice strain evolution of Zircaloy-2 under different loading modes. It was found that the time-dependent nature of EVPSC made it a much more viable candidate for predicting the relaxation behavior of the material during the experiment. The data obtained from EVPSC offered new insight into how these different forms of incremental loading can affect the relative activities of the various deformation modes.
Overall, the two goals of the research presented in this thesis have been met. The Genetic Algorithm has been successfully tested and implemented as a model optimization technique that can greatly reduce the time needed to obtain hardening parameters from experimental lattice strain measurements. Simultaneously, the neutron studies performed in Chapter 4 have shown that the lattice strain measurements obtained through neutron diffraction can be influenced to some degree by the type of incremental loading used, which is something researchers should be aware of when comparing datasets obtained through different experimental methods. Finally, the work done in Chapters 4 and 5 resulted in a greater understanding of how Zircaloy-2 behaves under incremental loading conditions as compared to continuous loading.

6.2 Future Work

There are certain areas in which the results of this work could be expanded upon. For example, the genetic algorithm currently does not directly include texture information in its assessment of the goodness of fit between the model and experimental data. In principle it should be possible to compare the measured twin volume fraction to the results generated by the model. It may even be feasible to compare measured pole figures to their model-derived counterparts, although this would likely require some major revisions to the code of the genetic algorithm software (possibly the self-consistent modeling software as well).

The material relaxation observed in Zircaloy-2 under the various loading schemes
seen in Chapter 4 have not been observed dynamically. In other words, each lattice strain data point captured represents the time averaged value of the continuously relaxing state of the material. It should be possible to repeat these same experiments at a synchrotron x-ray facility in order to capture the lattice strain relaxation dynamically, since such facilities typically have measurement times on the order of seconds.

Although Zircaloy-2 was found to have some dependence of its lattice strain evolution on loading mode, other materials may or may not share this quality. Repeating the experiments with a multi-phase material, such as Zr 2.5wt% Nb could provide interesting data, since the two phases would likely undergo different amounts of relaxation due to experiencing different degrees of total plasticity during deformation.
Bibliography


1 PID Settings Used During Uniaxial Deformation Tests

The uniaxial deformation tests carried out in Chapter 4 required the compression rig to be able to control the stress and strain applied to the sample as well as the position
of the actuator. In order to ensure adequate control, a sample was placed in the rig and tests were performed at low loads/strains/displacement to ensure that the rig could respond well enough to hold the relevant control variable steady. Sinusoidal waveforms were used to test the smoothness of the control and the proportional-integral-derivative (PID) settings were adjusted until a satisfactory response was observed. It should be noted that although the PID settings listed here functioned very well during the experiment, any attempt to reproduce the experiment should include its own PID tuning process, especially if a different sample material or compression rig is used. The PID values for position, stress, and strain control are listed in Table 1.

Table 1: PID settings

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