The effect of microstructural defect evolution during elastic fatigue loading on subsequent mechanical properties

Joseph Indeck
THE EFFECT OF MICROSTRUCTURAL DEFECT EVOLUTION DURING ELASTIC FATIGUE LOADING ON SUBSEQUENT MECHANICAL PROPERTIES

by

JOSEPH INDECK

A DISSERTATION

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The Department of Mechanical and Aerospace Engineering to
The School of Graduate Studies of
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ABSTRACT

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Title The effect of microstructural defect evolution during elastic fatigue loading on subsequent mechanical properties

Evolution of mechanical properties due to fatigue loading is often overlooked in the design process. Material properties in the as-received condition from a vendor are characterized and then these properties become the basis of design considerations. The effect of expected fatigue loading during operation and its potential to degrade mechanical performance is frequently accounted for with appropriate safety factors. Most notably, the aerospace industry does not have the luxury of large safety factors due to the detrimental effect of weight on cost and performance. Therefore, it becomes necessary to quantify not only the change in mechanical properties as a function of its fatigue life, but also understand what microstructural mechanisms are responsible for any property changes in order to optimize component design. Research into material evolution at both the macro and micro length scale has become even more relevant in the past few years with the advent of reusable launch systems.

This research investigates the microstructural evolution during elastic fatigue loading and the mechanisms driving subsequent mechanical property change. Two different material systems are analyzed, pure α-iron and 7075-T6 aluminum alloy. A
unique experimental approach was implemented in which multiple sub-tensile specimens are obtained from the interior of a fatigue specimen. Pre-fatigued sub-tensile specimens are tested under quasi-static and dynamic strain rates. Subsurface material for microscopy characterization is also obtained from the fatigue specimens.

It was found that the evolution of mechanical properties is dependent on both the subsequent loading strain rate and the amount of dislocation reversibility (controlled by the fatigue stress ratio) in each fatigue loading cycle. In-depth microscopy characterization revealed that microscopic fatigue-induced voids are the primary factor in subsequent strength degradation. Results from the research advance our current understanding of microstructural defect accumulation during fatigue loading and their effect on subsequent mechanical properties. Additionally, the dependence of post-fatigue material behavior on both strain rate and dislocation reversibility highlight the need to investigate additional materials and different fatigue conditions. Lastly, several analysis techniques utilizing statistical analysis and machine learning have been applied to microscopy datasets in order to identify underlying microscopic mechanisms that contribute to changes in subsequent mechanical response.

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I next want to thank my family. Seeing my siblings, John and Kate, succeed in challenging circumstances confirmed that my choice to seek a PhD was the correct one. To my parents, Debbie and Ron, your influence and encouragement has certainly changed my life for the better. You are some of the best role models one could hope for and I am extremely lucky to have you two as parents.

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<tr>
<td>$a$</td>
<td>lower data truncation limit</td>
</tr>
<tr>
<td>$A$</td>
<td>area</td>
</tr>
<tr>
<td>$A_b$</td>
<td>cross-sectional area of the Kolsky bar</td>
</tr>
<tr>
<td>$A_s$</td>
<td>cross-sectional area of the test specimen</td>
</tr>
<tr>
<td>$b$</td>
<td>Burgers vector magnitude</td>
</tr>
<tr>
<td>$b$</td>
<td>slip direction</td>
</tr>
<tr>
<td>$b_r$</td>
<td>residual Burgers vector</td>
</tr>
<tr>
<td>$C_L$</td>
<td>longitudinal wave speed</td>
</tr>
<tr>
<td>$C_0$</td>
<td>bulk wave speed</td>
</tr>
<tr>
<td>$d$</td>
<td>KNN point-to-point distance</td>
</tr>
<tr>
<td>$d$</td>
<td>average grain diameter</td>
</tr>
<tr>
<td>$e$</td>
<td>specific internal energy</td>
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<tr>
<td>$E_b$</td>
<td>Young’s modulus of the Kolsky bar</td>
</tr>
<tr>
<td>$F$</td>
<td>force</td>
</tr>
<tr>
<td>$F_1$</td>
<td>scoring metric for machine learning classification</td>
</tr>
<tr>
<td>$F_{1\text{norm}}$</td>
<td>scoring metric normalized to a coin flip model</td>
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\( F \)  \( \)  PCA scores

\( h \)  \( \)  KDE kernel bandwidth

\( k \)  \( \)  KNN number of cross-validation folds

\( k_T \)  \( \)  Zerilli-Armstrong material constant

\( k_y \)  \( \)  Hall-Petch material constant

\( K \)  \( \)  KNN number of neighbors

\( l_0 \)  \( \)  initial specimen length

\( m \)  \( \)  empirical constant for Walker’s effective stress measure

\( M \)  \( \)  Schmid factor

\( n \)  \( \)  number dataset points

\( \mathbf{n} \)  \( \)  normal vector to a plane

\( N \)  \( \)  number of fatigue cycles

\( N_f \)  \( \)  number of fatigue cycles to failure

\( N/N_f \)  \( \)  fatigue life ratio

\( P \)  \( \)  perimeter

\( r \)  \( \)  scan resolution

\( R \)  \( \)  fatigue stress ratio

\( s \)  \( \)  \( U_S-u_p \) empirical constant

\( u_{fs} \)  \( \)  free surface velocity
$u_p$  particle velocity

$U_S$  shock velocity

$U$  left singular vectors of $X$

$v$  average dislocation velocity

$V$  specific volume

$v$  line of intersection vector

$V$  PCA loading matrix; right singular vectors of $X$

$W$  KNN weighted distance

$X$  covariance or correlation matrix

$\Delta u_{fs}$  spall pullback velocity

$\epsilon$  strain

$\epsilon$  DBSCAN neighborhood size

$\epsilon_r$  reflected strain in the Kolsky incident bar

$\epsilon_t$  transmitted strain in the Kolsky transmission bar

$\epsilon_{eng}$  engineering strain

$\epsilon_{lag}$  Lagrangian strain

$\dot{\epsilon}$  strain rate

$\eta$  MgZn$_2$ precipitate formed in 7075 aluminum alloy

$\eta'$  MgZn$_2$ precipitate formed in 7075 aluminum alloy
θ  angle between the lines of intersection for two slip planes with the grain boundary
λ  diagonalized eigenvalues of $XX^T$
μ  PDF mean
$μ_0$  calculated mean
ρ  material density
$ρ_0$  dislocation density in line length per unit volume
σ  engineering stress
$σ_0$  stress level at onset of constant strain in dynamic tensile test
$σ_0$  Hall-Petch material constant
$σ_0$  calculated standard deviation
$σ_{HEL}$  Hugoniot Elastic Limit
$σ_m$  mean stress in fatigue loading
$σ_{max}$  maximum cyclic stress in fatigue loading
$σ_{min}$  minimum cyclic stress in fatigue loading
$σ_{sp}$  spall strength
$σ_{T0}$  Zerilli-Armstrong material constant
$σ_T$  flow stress required for twinning
$σ_y$  yield stress
$\sigma^2$ PDF variance

$\bar{\sigma}$ effective stress measure for non-reversible fatigue

$\Sigma$ singular value matrix
CHAPTER 1

INTRODUCTION

1.1 Research statement

The scope of this dissertation research is to investigate the effect of globally elastic fatigue loading on a material’s microstructural evolution and its impact on the subsequent mechanical properties. Elastic loading is commonly considered to be completely reversible, but this definition only applies to a continuum level length scale—thus the term globally elastic. Microstructural plasticity, e.g. dislocation irreversibility during fatigue loading, will still occur due to local phenomena. Material failure during globally elastic fatigue loading in crystalline metals is a localized, progressive process that involves the development and formation of microstructural features at different length scales. Certain microstructural features (such as specific dislocation arrangements or fatigue-induced voids) develop over time, accumulate, and eventually generate changes on a macroscopic length scale, like measurable crack propagation. Measurement of instantaneous material properties during the fatigue process is often overlooked in favor of studies that focus on fatigue crack initiation, crack growth mechanisms, and fatigue life prediction. Fatigue-induced microstructural changes during the fatigue process have the potential to change material properties compared
to the pristine condition. This subset of research can consider the important questions of whether or not a component still meets design criteria after accumulating fatigue loading during service, and what mechanisms on the microstructural level are accumulating that contribute to changes in the mechanical response.

1.2 Motivation

In general, many characterization tests and engineering designs typically rely on as-received material properties as supplied by vendors. However, there are situations when the instantaneous properties of the material are desired. Therefore, performance changes during the in-service life that might occur due to fatigue loading should not be overlooked. If a large factor of safety is used during the design process it is justifiable to ignore any material changes since a review of existing experimental results on the topic does not show strength decreases below a factor of two over a majority of the fatigue life. Considering that some standard safety factors can be five or above, changes due to elastic fatigue loading are understandably ignored. However, in industries that can be sensitive to over-design, such as too much weight in an aerospace application, understanding the minutia and intricacies of a material’s mechanical performance evolution over time could lead to improved design. For example, if strength decreases due to fatigue (e.g. through defect accumulation) a different measure of fatigue life can be defined using the amount of strength degradation instead of using fracture from crack propagation. On the other hand, if strength increases due to fatigue (e.g. through strain hardening) weight could be saved by redesigning the part, or the component can safely be reused. Therefore, evolution
of fatigue-induced defects and their effect on the material’s mechanical properties, is an important topic to research both from a fundamental microstructural perspective and an application perspective.

1.3 Research objective

The objective of this research is to quantify microstructural changes in materials as a result of fatigue loading; specifically to address how subsequent mechanical properties, tested at different strain rates, are affected. Advances in understanding what mechanisms caused subsequent property changes was enabled by employing a combination of microscopy characterization techniques, including one that has not been widely used before in this area of research. Volumetric analysis of fatigued material allowed insights into the microstructural evolution which might otherwise be overshadowed by free-surface effects. Therefore, this dissertation research improves upon previous studies through the application of particular characterization and analysis techniques to show the mechanisms causing defect accumulation throughout the bulk material and its effect on subsequent mechanical properties. Improvements to engineering component design can be attained by implementing the results of this research, specifically by moving away from large empirical safety factors and singular values for fatigue life. Savings in both component weight and cost could be achieved if material evolution is taken into consideration during design. Eliminating over-engineered designs is especially critical in the aerospace industry since extra weight leads to larger cost increases relative to other industries.

Organization of the dissertation is as follows:
• Chapter 2 reviews some of the pertinent background literature on the topic. Discussed are fundamental studies relating to dislocation behavior during fatigue and its role in driving crack initiation and eventual failure. In addition, a review of previous studies reporting mechanical behavior after interrupting fatigue is reported.

• Chapter 3 discusses the methodology developed to investigate material evolution throughout fatigue loading. Unique aspects include the use of real-time strain monitoring to assess fatigue life, micro X-ray computed tomography (µXCT) to quantify volumetric material defect accumulation, and the use of crystal plasticity modeling to create training datasets for machine learning clustering and classification models.

• Chapter 4 presents results for pure α-iron including volumetric µXCT scans quantifying the increase of fatigue-induced voids and electron backscatter diffraction data of the grain structure at different intervals of the fatigue life.

• Chapter 5 presents results for 7075-T6 aluminum alloy including identification of neighboring grain combinations that are susceptible to becoming locations of void nucleation and quantification of shape parameters for iron-rich inclusions that result in cracking.

• Chapter 6 summarizes the completed research and highlights the knowledge learned for both the problem statement in general and for the two specific materials.
• Chapter 7 lists several avenues of future research that could be pursued considering the results of the completed research.
CHAPTER 2

BACKGROUND AND LITERATURE REVIEW

Stress-life approaches to fatigue behavior, where cyclic loading is performed at a constant stress amplitude until failure, were first reported on in detail around the 1860s by August Wöhler who studied repeated bending of railway car axles [1, 2]. Commonly, stress-life approaches to fatigue behavior involve low stress amplitude loading cycles below the yield stress. This approach has been termed high cycle fatigue (HCF) since the low stress amplitudes lead to a long fatigue life (i.e. a high number of cycles to failure). During HCF permanent changes occur on a microscopic length scale due to the globally elastic loading conditions. The purpose of this Chapter is to review microscopic (namely dislocation) behavior during fatigue loading and how that affects subsequent mechanical behavior.

2.1 Irreversibility during fatigue loading

It can be assumed that for the majority of globally elastic loading conditions, microscopic behavior is rarely ever purely elastic. For example, microstructural

\(^1\)The original works of Wöhler are written in German and the author cannot verify what is actually reported. Details regarding the original references are taken from the extensive history of fatigue—which includes Wöhler’s work—by Schütz, [3], which summarizes [1] being where design of finite fatigue life was introduced and [2] being the first tabulated fatigue test results.
changes occur during fatigue loading even at very low stress amplitudes [4], and materials still fail during high cycle fatigue (HCF) and very-high cycle fatigue (VHCF) even though far-field loads are well below the yield stress. Therefore, one can infer there must be–at some length scale–irreversible effects that accumulate over time and eventually lead to material failure after millions or even hundreds-of-millions of loading cycles. Dislocation motion is one source of microscopic irreversibility. The applied stress required to move a dislocation, commonly referred to as the “Peierls stress” is a small fraction, sometimes orders of magnitude below the typically measured bulk yield stress, especially for pure metals [5].

The relative ease of edge dislocation motion at low stresses is not a new observation [6]. Dislocation motion has been observed in pure copper under bending stresses as small as 5.2 kPa and dislocation multiplication at 18 kPa using an etch-pit technique [7]. Similarly, nonzero dislocation velocities in pure aluminum were measured at applied shear stresses below 2 MPa [8]. Further, the numerically calculated Peierls stress in aluminum for both screw and edge dislocation components with a dissociated core are below 10 MPa [9]. Formation of fine 0.1–0.3 μm subgrains formed near a fatigue crack in 7075-T6 aluminum alloy that were independent of crack tip deformation shows that dislocation motion during fatigue loading also occurs in alloys despite precipitate obstacles [10].

Similar assumptions about dislocation motion at low applied stresses can also be made for body-centered cubic (BCC) materials, specifically in iron. Ikeda [11] loaded foil crystals in an electron microscope and by analyzing recorded images concluded that edge components of dislocations move at a resolved stress below the
macroscopic yield stress. Furthermore, when there was no long range movement of screw dislocations Ikeda [11] calculated a total strain of $3 \times 10^{-5}$ due to the applied stress by the swept area of dislocations between two images. This strain calculation implies dislocation motion at a stress of approximately 6 MPa, when using Hooke’s Law and a Young’s modulus of 200 GPa, which is much lower than other reported frictional stress values [12]. In addition, Turner and Vreeland [13] measured edge dislocation velocities in pure iron between approximately $10^{-2}$ mm/s and 10 mm/s for resolved shear stresses between 5 MPa and 50 MPa, respectively, by monitoring dislocations in a slip band with the Berg-Barret x-ray method. Furthermore, Caillard [14] used a transmission electron microscope (TEM) to monitor in-situ dislocation motion in pure iron specimens and observed through intensity changes between images that non-screw components of the dislocation experienced a negligible frictional stress.

The fact that dislocations move readily at low applied stresses is inadequate to fully explain–on a microstructural length scale–what might occur throughout fatigue loading. Cyclic slip irreversibility and dislocation irreversibilities must also be coupled with dislocation mobility when considering fatigue loading. Woods (in 1973) proposed an estimation of irreversible dislocation motion being related to the break down of short-range order in the crystal to help explain a decrease of hardness [15]. Short-range order being five to ten interatomic distances [16]. Newer definitions of irreversibility include a description by Essmann (in 1982) as the proportion of dislocation motion which is different upon reverse loading and consists of the plastic strain of randomly distributed slip steps along a slip band that are not completely canceled
during reverse glide [17]. Also, a similar definition is given as the ratio of microscopic irreversible plastic shear strain to total bulk cumulative plastic shear strain [18].

Persistent slip bands (PSBs) are a prevalent dislocation structure developed during fatigue loading and are the primary locations where irreversibility occurs. There are several approaches to predict PSB behavior and crack initiation during fatigue loading and each treat irreversibility slightly different [19]. Figures 2.1a and 2.1b shows a schematic illustrating how irreversibility occurs in a PSB. First, a vacancy-dipole model in which if a static dislocation density is assumed a plastic resolved shear strain below a certain level can be accommodated entirely through reversible back-and-forth motion of dislocations [20]. The level of accommodated plastic resolved shear strain is dependent on the local dislocation density, Burgers vector, slip-line length, and fraction of strain accommodated by the dislocation type located along the PSB. However, an equilibrium between dislocation multiplication and annihilation must occur if the plastic shear strain is greater than this value in order to keep a constant dislocation density. In this case, annihilation takes place through climb for edge dislocations and cross-slip for screw dislocations [20]. Since dislocations have a tendency to move along different slip planes depending on the loading sense during fatigue, [17,21,22], reverse glide generally cannot occur on the same slip plane due to the vacancy created by cross-slip annihilation of a screw dislocation [18]. Irreversibilities originate from this dislocation annihilation and continue to accumulate throughout the bulk material during fatigue [23]. Annihilation of dislocations leads to vacancy formation that in turn is the main physical reason for irreversibility in the dislocation glide process. For instance, screw dislocation pairs in room temperature
Figure 2.1: Dislocation configuration and irreversibility in a PSB for the vacancy-dipole model, (a) and (b), and the micromechanical model, (c). (a) A simplified dislocation arrangement of a PSB formed during fatigue showing the high dislocation density walls inside the slip band. Adopted from [23]. (b) As dislocations annihilate in the PSB walls and create vacancy point defects, the glide plane is shifted. A dislocation will travel from A to A’ during a loading cycle and B to B’ in reverse loading following a stepped pattern. Vacancies in the walls and internal stresses from the PSB-matrix interface do not allow dislocations to return to their original position causing irreversibility. Adopted from [23]. (c) Dislocations travel along the two PSB layers and pile-up at a rigid obstacle, typically a grain boundary. The stress from dislocations on layer II creates a back stress on the dislocations along layer I pinning them in place during reverse loading causing irreversibility. Adopted from [26]. Extrusions at the surface created by the PSB are not drawn.

copper annihilate each other as long as the glide planes are separated by less than \( \approx 50 \text{ nm} \) [24], and edge dislocations less than \( \approx 1.6 \text{ nm} \) [17]. For iron single crystals, Li et al. reported a critical annihilation radius of 375 nm for two mobile edge or screw dislocations [25]. Hence, it could be stated that irreversibility is due to the vacancy formation created during dislocation annihilation blocking reverse slip along the same plane.
Another approach to PSB irreversibility, as depicted in Fig. 2.1c, entails accumulation of dislocations at an obstacle, such as a grain boundary, and being pinned there by the back stress of the dislocation stress field on the opposing PSB layer. This concept of PSB evolution and crack initiation is incorporated in a micromechanical model [26,27,28,29,30]. Consider a single loading cycle to highlight this irreversibility concept. During loading in one direction dislocations will move along one glide plane of a PSB accumulating at an obstacle. During reverse loading dislocation motion will occur in the opposite direction, but the glide plane is not necessarily perfectly reversed. Thus, these dislocations cause an effective back stress on the dislocations that moved during the first half loading cycle and not all of these dislocations move back to their original location remaining pinned at the obstacle. While the motion of dislocations are largely reversible at lower stress amplitudes, a small percentage of irreversible ones gradually accumulate with continued fatigue. Using energy considerations of the dislocation stress field, crack formation, and PSB free surface energy, a critical dislocation density along the PSB that leads to crack initiation has been formulated [26]. Furthermore, the spacing between PSB’s can also be estimated [31]. Therefore, irreversibility is due to the accumulation of dislocations at an obstacle that remain pinned during reverse loading.

Both approaches and irreversibility definitions eventually lead to the conclusion that cracks and micro-voids will form due to dislocation motion and interactions. Potential mechanisms that drive irreversibility are introduced because this research examines if non-trivial changes to the mechanical properties result throughout the bulk material as a result of these mechanisms occurring. Furthermore, it is of interest
to quantify any bulk changes and determine their effect on subsequent mechanical behavior.

2.2 Dislocation features of fatigued material

The purpose of this section is to review a prevalent form of dislocation structure formed during fatigue loading, the persistent slip band (PSB). Dislocations move due to local stress concentrations, interact, and can eventually produce structures, such as PSBs even during globally elastic loading [18]. Defects that accumulate due to the irreversibilities of dislocation motion are located in the PSB dislocation structure. As will be discussed, crack initiation and eventual failure of the material in many fatigue conditions, takes place at a PSB highlighting the importance of understanding microstructural evolution during fatigue. A further overview on approaches to predict PSB behavior and crack initiation during fatigue loading is provided by Man et al. [19].

2.2.1 Persistent slip bands

A 1903 study by Ewing and Humfrey is likely the first ever to show the formation of slip bands during fatigue loading [32]. Their original schematic for how a slip band emerges and rises above surrounding material is shown in Figs. 2.2a and 2.2b and is strikingly similar to recent schematics with more detailed observations from recent literature, cf. Fig. 2.3 [33]. Figures 2.2a and 2.2b represent what Ewing and Humfrey call a “heaving-up” of each slip band from the surface and then the effect of reverse loading on the edge of the slip band. Growth of slip bands from faint lines at 1000 cycles to broad, extended lines at 20 000 cycles is also shown, Figs. 2.2c
Figure 2.2: (a) Schematic from Ewing and Humfrey showing how observed slip bands protrude upward from the surface and surrounding material. (b) The effect of reverse loading on the shape of protruded slip bands. (c) Microscope image of Swedish iron after 1000 cycles showing the faint lines of multiple slip bands on the right-hand side of the grain. (d) Microscope image of the same grain at 20,000 cycles showing the growth and broadening of slip bands during fatigue. All images taken from [32].

and 2.2d. The interesting fact that this phenomena occurred at low stresses was not lost on Ewing and Humfrey as they described, “[i]t is remarkable that these actions are brought about by stresses much below what is ordinarily understood by the elastic limit of the material.”
Figure 2.3: (a) Schematic showing characteristic surface relief due to a PSB formed during fatigue. Fully developed slip markings show ribbon-like extrusions and an intrusion on either side. (b) An SEM-FEG micrograph of an individual PSB extrusion in 316L steel. Images taken from [33].

Approximately fifty years later Thompson et al. showed that fatigue failure in copper originated in a slip band [34]. This work also likely contained the first use of the phrase “persistent slip band” to describe slip markings, similar to those shown by Ewing and Humfrey, that remained visible even after etching away roughly 2 µm of material. The fact some slip lines remained after etching indicated these particular bands were something more than surface steps—a bulk phenomena. Another important conclusion from Thompson et al. was the fact that continuous removal of these slip bands through polishing the surface increased the fatigue life by more than 200% helping form the understanding of fatigue failure originating at the surface due to these bands.

However, it might be incorrectly concluded from Thompson et al., [34], that PSBs are only located on the surface since it was reported that removal of 34 µm of
material resulted in the disappearance of all slip marks, even those termed persistent slip bands. As the importance of PSBs on fatigue failure became understood, further research has investigated whether or not they are isolated at the surface. Initial indications of PSBs as a bulk phenomena came, in part, from work by Winter, [35], who showed the same PSB dislocation structure existed throughout the entire volume of a copper single crystal. It was postulated that PSBs might also occur throughout a polycrystalline material as well, since a PSB can traverse through an entire single crystal.

Winter et al., [36], pursued the question of bulk PSBs and observed them in polycrystalline copper up to 3 mm from the surface at a similar frequency and with little difference in dislocation structure. An electron channeling contrast (ECC)/scanning electron microscope (SEM) image of a PSB in an interior copper grain is shown in Fig. 2.4, however the grain depth from the surface is not specified. Pohl et al., [37], also observed PSBs 2 mm from the surface in low carbon steel (0.1 wt.% C) albeit at a lower frequency than material near the surface. Furthermore, it was qualitatively predicted by Lukas and Kunz, [38], that the frequency of PSB occurrence for interior grains 1–2 mm from the surface is an order of magnitude less than surface grains due to the constraints of surrounding grains on the plastic deformation of interior grains. Being constrained by surrounding material means it is likely that a grain will experience more than a single slip system making the development of a PSB less common than a surface grain.

However, in more recent studies Weidner and Skrotzki, [40], found that in pure polycrystalline nickel PSBs can develop in the bulk of the material, have the same
Figure 2.4: Electron channeling contrast imaging of PSB in an interior grain of pure polycrystalline copper. The stress amplitude for this experiment was 54 MPa which was previously considered as below a PSB formation threshold. Image taken from [39].

dislocation structure to what is observed in surface grains, and occur with the same frequency as surface grains. Most notable was the observation that bulk PSBs can lead to grain boundary displacements, instead of the surface steps that have more commonly been studied and reported. Figure 2.5 shows how a bulk PSB creates “waves” along the grain boundary and voids originating at the interface of the PSB and grain boundary. It was found that formation of internal cracks, at least in the case of copper, was due to grain boundaries and not impurities [41]. Therefore, it is likely that the interaction of PSBs with grain boundaries—not other microstructural inhomogeneities—lead to internal void formation. Figure 2.6 show PSB termination at a grain boundary leading to an internal void nucleation. It should be noted that
Figure 2.5: Electron channeling contrast imaging of bulk PSBs in pure polycrystalline nickel. The direction of the Burgers vector and slip planes of the PSBs are denoted. (a) Arrows show the grain boundary displacement at the end of each PSB. The PSB indicated on the far left has displaced enough to create what might be a visible void. (b) Void creation due to a PSB terminating at a grain boundary. Images taken from [40].

even though internal voids can form at low stress amplitudes, 30 MPa for the crack pictured in Fig. 2.6, it does not necessarily mean the stress is large enough to cause further crack propagation and failure. In general, interaction of PSBs with the free surface creates intrusions and extrusions that become the site of crack initiation and eventual failure. It is now accepted that PSBs accommodate plastic strain developed during fatigue and are the sites for crack initiation in ductile single-phase metals [42].

2.2.2 Dislocation arrangement of persistent slip bands

Dislocation structures in copper single crystals formed at low plastic strain amplitudes were investigated by Lukáš et al. [43]. Material was fatigued at approximately 64 MPa up to an estimated 20% of its fatigue life ($2 \times 10^5$ out of $1 \times 10^6$ cycles). Figure 2.7 shows transmission electron microscope images of the dislocation...
Figure 2.6: Electron channeling contrast image showing crack formation at a grain boundary 50µm below the surface of pure copper. A ladder-like PSB structure can faintly be seen. The purity of copper tested, 99.999%, show that grain boundaries are responsible for internal crack formation. Image taken from [41].

structures after fatigue loading. Perpendicular to the primary slip plane, Fig. 2.7a, bands of dislocations separated by dislocation-free channels were observed. These bands of dislocations are also sometimes referred to as veins, cf. [44]. Formation of a PSB with a well defined ladder structure of dislocation walls, Fig. 2.7b, takes place after dislocations throughout the bulk volume of the material have arranged into the dislocation bands. In fact, only a small percentage of the overall material volume will form the ladder-like PSB structure [43]. As previously discussed, in Section 2.2.1, this type of PSB structure can occur throughout the entirety of the material volume. However immediately next to the surface (≈10µm) PSBs can be formed from rows of dislocation cells, instead of the dislocation walls in the ladder structure, as seen in Fig. 2.7c. These cells form more readily when the fatigue stress amplitude
Figure 2.7: Single crystal copper fatigued to approximately 20% of the fatigue life. (a) Image showing the dislocation bands that form throughout the entire volume of material after fatigue loading. There are relatively few dislocations located in the channels between the dark dislocation bands. (b) A typical ladder-like PSB structure, formed along the primary slip direction, alongside the bands of dislocations which do not form into a PSB. (c) Rows of dislocation cells in a PSB, formed along the primary slip direction, alongside a well defined ladder-like PSB structure. Dislocation cells in a PSB will only occur in material near the free surface. All images are taken along the (1 2 1) plane which is perpendicular to the primary slip plane and the black lines indicate the primary slip direction [T 0 1]. Images taken from [43].

is higher and the activation of non-coplanar slip systems. The stress field around a dislocation located next to a free surface is greater than a dislocation in an infinite medium [45,46], accounting for the observation of cell-like PSBs only near the surface and not in the bulk.

In general, dislocations can form a stable configuration of edge dislocations in a checkerboard arrangement called a Talyor lattice [47]. Stable referring to zero net force on each dislocation under no external loading. However, the arrangements of dislocations determined by Taylor are only stable in an infinitely repeating pattern. Identifying stable configurations of finite Taylor lattices and the formation of dislocation walls (occurring in a ladder-like PSB) was done by Neumann using a numerical technique to determine stable dislocation structures in a relaxed configuration, i.e.
Figure 2.8: (a) Rectangular dislocation array. When $h = 2d$ then the arrangement is considered a Taylor lattice. Highlighted in red is a dislocation quadrupole, the building block for stable dislocation arrangements and the initial dislocation structure of fatigued material. (b-d) Collections of dislocation quadrupoles stable at zero applied stress. Images taken from [44].

with no applied stress [44]. Considering single dislocations, dipoles (two dislocations of opposite signs), and quadrupoles (four dislocations making up the smallest group in a Taylor lattice) only groups of quadrupoles are stable under zero applied stress while being surrounded by dislocation-free material. Figure 2.8 shows both a Taylor lattice arrangement of dislocations and stable quadrupole configurations as calculated by Neumann.
If a group of quadrupoles is stressed the dislocations become more and more polarized eventually reaching a point at which the configuration separates and transforms into dipole walls instead. This process is shown in Fig. 2.9a with the resulting dipolar walls being more stable than the original quadrupole arrangement. These dipole walls are stable because they form during application of an applied stress, whereas at zero applied stress only the quadrupole arrangement will stably relax. As cyclic loading continues and the material hardens, spacing between the walls in each vein will decrease down to a minimum distance. At this minimum wall spacing the dislocations can no longer rearrange into additional walls with smaller spacings within the vein so they instead transform into the dislocation walls seen in a ladder-like PSB. Ladder-like PSB dislocation walls are thicker than the dipole walls in the veins and more stable. A possible dislocation wall configuration was calculated by Neumann and is shown in Fig. 2.9b. An important assumption with this work is that the dislocation veins (Fig. 2.7a) are arranged in a regular manner in blocks of dislocation quadrupoles, e.g. Fig. 2.9a. While the work of Neumann provides tremendous insight into the formation of dislocation walls with a well defined ladder configuration, there still remain questions as to how the initial dislocation veins are formed [48].

Observations from Neumann’s numerical work were experimentally observed—almost twenty years later—in copper single crystals by Li et al. [49]. The experimental work was carried out using SEM and ECC at regular intervals during fatigue loading at a plastic shear strain of $1 \times 10^{-3}$. After only 20 cycles there were no dislocation structure observable in the bulk material, but at 40 cycles the beginning of vein formation was witnessed. As cycling continued, up to approximately 700 cycles, the
Figure 2.9: Illustrations of stable dislocation quadrupole configurations during fatigue loading. (a) The top layer shows the dislocation arrangement, at zero applied stress, of the dislocation veins located throughout the entire material. The bottom layer shows the dislocation arrangement, at maximum applied stress before transformation into dipole walls. Separation of the dislocations into distinct dipole walls is shown in the illustration. (b) Schematic of stable dislocation arrangement in ladder-like PSB walls. Images taken from [44].

vein structure (e.g. height, width, and spacing) evolved. Experimental measurements showed that during this portion of cycling the spacing between veins decreased as predicted by Neumann, see Fig. 2.10. The first experimental observations of PSBs by Li et al. occurred at approximately 800 cycles, when spacing of the dipole walls reached a minimum. At this point during fatigue the exact transition into the ladder-like PSB is not well characterized, but Li et al. [49], proposed the following qualitative process:

- A dipole wall collapse in the vein, due to applied stresses causing dislocation curvature, will cause adjacent wall collapse when the wall spacing decreases to a critical value.

- Wall collapses take place along the slip direction and a persistent slip line (PSL).
Dislocation walls in ladder-like PSBs then emerge from the PSL, see Fig. 2.11, due to easier operation of screw dislocation segments along the PSL.

Observation of dislocation walls appearing on either side of a PSL indicates the requirement of PSL formation, from dipole wall collapse in the vein, as a prerequisite to ladder-like PSB formation.

![Graph showing experimental measurements of spacing between dislocation dipole walls as a function of fatigue loading.](image)

**Figure 2.10**: Graph showing experimental measurements of spacing between dislocation dipole walls as a function of fatigue loading. References from the plot are Pedersen and Winter [50], and Basinski et al. [51]. Image taken from [49].

### 2.2.3 Other fatigue-induced dislocation arrangements

Various microstructural features form during fatigue depending on the material, amplitude, stress ratio, and amount of cyclic loading, even though PSBs are the most widely referenced and reported dislocation structure formed during fatigue. For example, a diagram of the cyclic saturation stress-strain regions correlates to the evolution of different dislocation structures a material might experience, see Fig. 2.12.
**Figure 2.11**: Arrow pointing out a PSL development after 2250 cycles. Dislocation walls for a ladder-like PSB are seen emerging from both sides of the PSL implying the requirement of PSL formation before a ladder-like PSB formation. Image taken from [49].

The dark regions in the structures are groups of edge dislocation dipoles formed during reversed cyclic loading separated by relatively dislocation free regions. Vein networks, shown on the bottom left of Fig. 2.12, are unique in the fact they do not produce long-range internal stresses similar to those caused by dislocation pile-ups during monotonic loading. Persistent slip bands, shown in the bottom middle of Fig. 2.12, are the regions where dislocation walls span between two glide planes and appear like rungs on a ladder. Lastly, dislocation cells, shown on the bottom right of Fig. 2.12, can form due to secondary slip between the PSB and matrix. However, microstructure evolution during fatigue is not exclusively defined by the formation of dislocation substructures. For example, 7075 aluminum alloy will experience fatigue crack initiation from cracked inclusion particles in lieu of prominent strain localization from dislocation structures [52,53].
To summarize, fatigue-induced dislocation structures are bands (or veins) of quadrupoles throughout the volume of the material. How these structures form is still not completely understood. Transition from these bands (veins) to the typically seen ladder-like PSB with walls of dislocation quadrupoles takes place when the dislocations in the bands (veins) separate and form dipolar walls. Once the spacing of
these walls reaches a minimum distance they will breakdown along a PSL where a
transition to the thicker walls of the PSB takes place. Dislocations bowing out from
the walls into the adjacent walls has been proposed as the mechanism for how surface
slip steps form. If this process occurs in an interior grain of a polycrystalline material,
then the flow of dislocations from wall-to-wall can eventually cause grain boundary
displacements and internal void nucleation. However, not all fatigue loading will
produce this specific ladder-like PSB. Under conditions of higher stress amplitudes a
band of dislocation cells will form instead.

2.3 Effect of pre-fatigue loading on mechanical properties

This section presents a review of prior research on the effect of pre-fatigue
loading on mechanical properties. In 1933 Herbert French used the term “probable
damage lines” to describe a region of overstress where cumulative damage will affect
the material’s fatigue strength [56]. His original stress vs. number of cycles to failure
(S-N curve) including this probable damage line is shown in Fig. 2.13. In one instance
after 10000 loading cycles of an overstress above the fatigue strength, a specimen
failed in 22000 cycles at a stress below the fatigue strength where it should have
had an infinite life. French’s work was one of the initial studies investigating how
pre-fatigue loading altered subsequent mechanical properties.

Another one of the first set of studies was performed by MacGregor and Gross-
man and involved the investigation of prior fatigue loading on the ductile-to-brittle
transition temperature of 1020 steel alloy, 4130 steel alloy, 75S-T6 aluminum alloy,
and 24S-T4 aluminum alloy [57, 58]. The amount of energy required for fracture in
the aluminum was measured as an indication of prior fatigue damage instead of a ductile-to-brittle transition. An increase in the transition temperature as the number of fatigue cycles increased was observed. For example, the transition temperature increased 46°C in one specimen cycled to 73% of the fatigue life. Furthermore, increases in the transition temperature of the 1020 steel alloy still took place even at fatigue stress amplitudes below the expected endurance limit. A curve predicting ductile-to-brittle transition temperature vs. the number of fatigue cycles was made to quantify the effects of prior fatigue loading. It was also found that the energy absorption decreased as the number of fatigue cycles increased for both aluminum alloys. These two studies along with the work of French were some of the first published data proving that globally elastic fatigue loading can change bulk material properties in a non-trivial fashion.

The works by Lopez et al. [59,60] closely resemble the experimental procedure of what is used for this dissertation research. Specimens of Ti-6Al-4V were electri-
cal discharge machined (EDM) to remove any surface cracks and examine the bulk material post-fatigue tensile behavior under quasi-static and dynamic strain rates. Removing surface effects through EDM is an important step to investigate whether any microscopic changes in the bulk material affect subsequent behavior. Fatigue tests were performed under load controlled cyclic loading with a stress amplitude 70–90% of \( \sigma_y \) to three different levels of \( N/N_f \), approximately 0.02, 0.30, and 0.59. Results showed that fracture strain, energy absorption, and UTS were unaffected by the prior fatigue loading for both quasi-static and dynamic strain rates.

Quasi-static and dynamic tensile behavior of 6061-T6 aluminum alloy and AISI 4140T steel alloy after cyclic loading with a mean stress of approximately 28% of \( \sigma_y \) and a stress ratio of \( R = 0.2 \) was investigated by Sanchez-Santana et al. [61,62,63]. It was found that the steel alloy was more sensitive to previously fatigue-induced defects than the aluminum alloy. Quasi-static yield stress decreased by approximately 27% in the steel alloy, but only 5% in the aluminum alloy after 75% of the fatigue life. Similarly, quasi-static ultimate tensile strengths (UTS) decreased by approximately 21% in the steel alloy, and again by a smaller 7% in the aluminum alloy. The steel alloy exhibited a greater reduction in elongation and the aluminum alloy exhibited an increased microhardness when cycled to under strain controlled fatigue versus load controlled fatigue cycled to the same percentage of fatigue life. Furthermore, no strength changes were measured in the 6061-T6 aluminum alloy under quasi-static strain rates, \( \approx 10^{-3} \text{s}^{-1} \) due to prior fatigue loading, but there was a noticeable decrease in strength under dynamic strain rates, \( \approx 10^{3} \text{s}^{-1} \). It needs to be noted that the dynamic tension data from the Kolsky bar testing in these studies was analyzed.
with the standard 3-wave assumption typically reserved for compression testing and known to be inaccurate for tension testing [64, 65]. Nevertheless, these observations show that both the type of fatigue loading and type of subsequent tensile loading affect how the mechanical properties evolved—conclusions also proven with the results of this dissertation research.

Quasi-static fracture toughness of the same two materials, 6061-T6 aluminum alloy and AISI 4140T steel alloy, were investigated after load controlled fatigue conditions below the yield stress [66]. Fracture toughness of the steel alloy did not change due to the prior fatigue loading, however there was a decrease of approximately 20% in the aluminum alloy samples. Conversely, under dynamic strain rates, the fracture toughness of the aluminum alloy remained constant whereas the steel alloy decreased up to 55% relative to the pristine material. These results show that subsequent mechanical behavior is material dependent, in addition to being dependent on the type of fatigue and subsequent loading conditions.

Three different aluminum alloys were tested under quasi-static and dynamic tension after undergoing load controlled fatigue: 2219-T87, 6061-T6, and 7075-T7351 [67]. Pre-fatigue loading was performed at 78% and 89% of the UTS to either 0.6 or 0.8 of the fatigue life. It was found none of the aluminum alloys had changes in UTS, total strain, or energy absorption during subsequent quasi-static loading. However, both the 2219-T87 and 7075-T7351 aluminum alloys experienced decreases in UTS and energy absorption during the post-fatigue dynamic tension. The UTS of the 2219-T87 aluminum alloy decreased by a maximum of 20% and the 7075-T7351 aluminum alloy decreased between approximately 5%–20% depending on the stress amplitude.
of the fatigue loading. Two of these aluminum alloys, 2219-T87 and 6061-T6, were investigated using SEM to discern if the pre-fatigue loading lead to an increase in cracks greater than 10µm after dynamic tension [68]. The 2219-T87 aluminum alloy had an increase of approximately 25% in the total number of visible cracks between the pristine and pre-fatigued specimens, whereas the 6061-T6 aluminum alloy had an average increase of 50%. These microscopic observations show that prior fatigue loading can alter both the subsequent macroscopic properties of a material (i.e. the yield stress or UTS), and the subsequent microscopic properties (in this case a higher number of microcracks).

Criteria to define acceptable levels of fatigue loading that do not adversely affect impact toughness were developed for another aluminum alloy, 2017-A T3 [69,70]. A specialized damage indicator using mixed sound emission and numerical simulation modal analysis was developed to predict the subsequent mechanical response for combined fatigue-impact loading. Three different post-fatigue behaviors were identified:

1. Subsequent mechanical properties that were equivalent to the pristine material.
   This behavior occurred in 90% of the samples that were fatigued with a stress amplitude half of the yield stress ($\sigma_a = 0.50\sigma_y$) regardless of the amount of fatigue loading.

2. Subsequent mechanical properties that were weaker than the pristine material, specifically yield stress and decreased ductility. This behavior occurred in 95% of the samples that were fatigued with a stress amplitude 70% of the yield stress ($\sigma_a = 0.70\sigma_y$) to 50% or more of the fatigue life ($N/N_f > 0.50$).
3. Subsequent mechanical properties that were stronger than the pristine material, specifically an increase of 50% in both yield stress and UTS. This behavior occurred in 60% of the samples that were fatigued with a stress amplitude 70% of the yield stress ($\sigma_a = 0.70\sigma_y$) to 10% or less of the fatigue life ($N/N_f < 0.10$).

A similar analysis was performed on 5454-O aluminum alloy, however no changes in the macroscopic material response due to the fatigue loading were observed [71]. Nonetheless, the authors do comment on the difficulty of imparting the same amount of fatigue-induced defects between test specimens due to scatter in fatigue testing. Two actions were taken in this dissertation research to address this concern. First, multiple post-fatigue test specimens were obtained from each fatigue test. Second, post-fatigue specimens were obtained from the bulk material to remove surface defects that might be responsible for potential scatter in the number of cycles to failure. Both of these approaches, in addition to using real-time strain evolution monitoring, were employed for this dissertation research as described in Chapter 3.

There are also several studies focusing on the behavior and subsequent mechanical properties of different steel alloys after fatigue loading. Medium carbon steel 45# displayed separate trends in yield stress, UTS, and ductility as a function of fatigue loading at 80% of the yield stress [72]. Yield stress experienced an initial 25% decrease and then fluctuated throughout the rest of the fatigue life. Ultimate tensile stress remained constant until near the end of the fatigue life ($N/N_f > 0.90$) when it experienced a 40% decrease. Finally, ductility continually decreased throughout the entire fatigue life. Evolution of mechanical properties due to fatigue are not always
negative. Strength of 304 stainless steel was shown to increase for both yield stress and UTS as a result of strain controlled fatigue loading [73]. Changes were not insignificant either, with the yield stress increasing from 260 MPa to 740 MPa and the UTS increasing from 750 MPa to 935 MPa. As expected, these increases also resulted in a loss of ductility, upwards of 80% in the worst case scenario at the end of the fatigue life. Strength of AISI 1022 steel was also found to undergo strength increases due to prior fatigue loading [74]. Yield stress increased approximately 18% and UTS approximately 5% compared to the pristine material. This study also demonstrated that different degrees of non-reversible fatigue loading have effects on subsequent mechanical properties. Fatiguing with higher levels of mean strain decreased the rise in UTS.

Subsequent loading strain rate also determines if a material will experience post-fatigue mechanical property changes along with the fatigue mean stress. In other words accumulated defects that develop throughout fatigue loading are strain rate dependent. For instance AISI 1045 steel tested at strain rates between $1 \times 10^{-4}$ s$^{-1}$ to $1 \times 10^{0}$ s$^{-1}$ had different amounts of yield stress changes [75]. Yield stress increases were as high as 26% during quasi-static loading ($1 \times 10^{-4}$ s$^{-1}$), but were less than half that during intermediate strain rate loading ($1 \times 10^{0}$ s$^{-1}$). Another study on two different steel alloys, AISI 1215 and 12L14 steel, also showed strain rate dependency in the subsequent mechanical properties [76]. Changes in the strength and toughness during dynamic loading ($1 \times 10^{3}$ s$^{-1}$) were twice as much as the changes during quasi-static loading ($1 \times 10^{-3}$ s$^{-1}$). Subsequent strength changes during quasi-static loading were then mitigated by decreasing the fatigue stress amplitude [77].
Several research studies studying the effect of pre-fatigue loading on subsequent mechanical properties for materials with different stacking fault energies (SFE) have been published by Yan et al. [78,79,80,81,82,83]. Investigated materials include pure aluminum [79,80,83], pure copper [78,81], and an aluminum-copper alloy [82]. Fatiguing at a stress amplitude of 30 MPa to only 5% of the fatigue life caused the creation of dislocation cells in different thickness of pure aluminum sheets [80]. These dislocation cells resulted in an increase of UTS due to their promotion of dynamic recovery and sub-grain formation during the subsequent tensile deformation. Pure aluminum processed with equal-channel angular (ECAP) pressing to an average grain size of approximately 1 µm was fatigued with a stress amplitude approximately half of the yield stress, $\sigma_a \approx 0.50\sigma_y$ [83]. An initial decrease of about 15% in both the yield stress and UTS was measured at only 5% of the fatigue life, $N/N_f = 0.05$. The high residual stresses and dislocation densities created during the ECAP processing are relieved during the initial fatigue loading accounting for the measured strength decreases. An aluminum alloy with 16 at.% copper was fatigued under strain controlled conditions until a specific amount of cumulative plastic strain developed [82]. Fatiguing at a low strain amplitude, $1.0 \times 10^{-3}$, resulted in a 24% increase in UTS without a corresponding loss of elongation. More planar slip dislocations arising during the fatigue loading was thought to be responsible for the UTS increase. On the other hand, fatiguing at a higher strain amplitude of $2.3 \times 10^{-3}$ and $3.7 \times 10^{-3}$, resulted in a decrease of both the UTS and the elongation, approximately 10% and 20%, respectively. The following observations relating the SFE and effect of pre-fatigue loading can be made by comparing the results of all these works. Pure aluminum, with a rel-
atively high SFE of 200 mJ/m², experienced dynamic recovery of the microstructure during subsequent tension testing that led to finer sub-grains but not an increase in UTS [79, 80]. Pure copper, with an intermediate SFE of 45 mJ/m², can experience a UTS increase, but total elongation will decrease because of the PSB conversion to elongated dislocation cells during tensile testing [78, 81]. Finally, the Cu-16 at.% Al alloy with a low SFE, 6 mJ/m², can experience increases in both UTS and elongation due to twinning during tensile loading and the formation of mobile planar slip dislocations during fatigue respectively [82]. In general, lower SFE materials are more likely to experience increases in mechanical behavior due to fatigue.

Multiple other studies report on the subsequent mechanical behavior of additional materials [84, 85, 86, 87, 88, 89, 90, 91]. Strength changes of various materials after elastic fatigue loading for both quasi-static and dynamic loading are shown in Figs. 2.14 and 2.15, respectively. Data trends highlight that it is common for strength to change due to fatigue (even when the maximum stress is below the yield stress) whether it increases or decreases. It should be clear, based on the preceding literature review, that the subsequent mechanical behavior of a material that has undergone fatigue loading varies greatly depending on the material, fatigue parameters, property in question (e.g. strength or ductility), and the subsequent loading strain rate. This fact is underscored by the vastly different responses highlighted in Figs. 2.14 and 2.15. Therefore, a generalized conclusion describing the subsequent mechanical response based solely on fatigue cycles cannot be made. Instead, a detailed analysis must be performed on pre-fatigued material in order to know how and why mechanical properties evolve as a function of fatigue life. Researching and reporting on only
the global behavior of subsequent mechanical properties—while important, is not a
unique research approach. Therefore, the novel aspects of this dissertation research
come from the quantification of microscopic changes and their correlation to the evo-
lution of subsequent mechanical properties. As a result, mechanisms producing the
measured material changes are identified.
Figure 2.14: Results for various materials showing trends in the change of strength during post-fatigue quasi-static deformation, $\dot{\varepsilon} \approx 10^{-3} \text{s}^{-1}$. Only data consisting of load controlled fatigue at, or below, the yield stress is shown. (a) Yield stress values as a function of fatigue life. (b) Percent change in the yield stress as a function of fatigue life. (c) Ultimate tensile stress values as a function of fatigue life. (d) Percent change in the UTS as a function of fatigue life.
Figure 2.15: Results for various materials showing trends in the change of strength during post-fatigue dynamic deformation, $\dot{\epsilon} > 10^2 \text{s}^{-1}$. Only data consisting of load controlled fatigue at, or below, the yield stress is shown. (a) Maximum dynamic stress as a function of fatigue life. (b) Percent change in maximum stress as a function of fatigue life.
CHAPTER 3

EXPERIMENTAL METHODOLOGY AND ANALYSIS APPROACHES

An original experimental and analysis approach was developed in order to link the bulk microstructural evolution to subsequent mechanical property changes throughout a material’s fatigue life. This chapter is devoted to describing the experimental approach and analysis methods. The testing process consisted of first fatiguing a specimen, second obtaining sub-tensile specimens from the bulk of the fatigued material, third characterizing the microstructure of the bulk fatigued material, and lastly testing the sub-tensile specimens under both quasi-static and dynamic strain rates. Addressed during development of the research process were concerns about fatigue to failure uncertainty, the ability to perform microstructure characterization, reproducibility in test results, and the ability to use different equipment for mechanical testing at varying strain rates. The analysis methods include the use of several machine learning algorithms and creation of a statistical approach to determine practical resolution limits from datasets following an assumed distribution.
3.1 Materials and specimen preparation

This section describes the stock material, its processing and machining into the test specimens, and details about the design and dimensions of the fatigue and sub-tensile specimens.

3.1.1 α-Iron

Round as drawn bar stock of 99.8% pure iron with a 25.4 mm diameter and 1 m length manufactured by Goodfellow Corporation was used for this dissertation research. Each rod was produced from an initially cast billet, then hot rolled and finished by cold drawing. Pure α-iron was chosen as a material of interest because it is the base constituent in steel, the most widely used structural material. Note that Turner and Vreeland reported little change in dislocation motion for specimens of iron with a carbon impurity content of 45 ppm by weight compared to higher purity iron [92]. Thus, it is reasonable to assume that the small amount of impurity content for the iron used in this research will not significantly affect the dislocation mobility conclusions from studies on either higher purity iron or single crystal iron [11,13,14,93]. This assumption means there should still be ample dislocation motion in the material at applied loads below the yield stress. Average grain diameter of the stock iron was 63.5 μm corresponding to an ASTM grain size number of 5.0, [94], calculated by a line-intercept method at multiple locations from images taken on an optical microscope. Surface finishes were measured using a Taylor-Hobson
surface contact profilometer with a 60 mm long stylus and 0.002 mm diamond tip. The average measured $R_a$ value for the $\alpha$-iron fatigue samples was 0.040 $\mu$m.

### 3.1.2 7075-T6 aluminum alloy

Cold drawn bar stock of 7075-T6 aluminum alloy with an average initial diameter of 25 mm was purchased from McMaster Carr. The 7075 aluminum alloy was developed in 1943 to improve stress-corrosion issues of an existing aluminum alloy by adding chromium [95]. It is strengthened through the formation of $\text{MgZn}_2$ $\eta$ and $\eta'$ precipitates which inhibit dislocation movement [96, 97, 98, 99]. Specific -T6 heat treatment steps are as follows:

- Heat in an air furnace between 460°C to 500°C ($\pm 6$°C) for a soak time specified by the part thickness. For parts 12.70 mm to 25.40 mm thick, soak for a minimum of 90 min, however, ASTM warns about possible melting occurring at temperatures greater than 482°C [100].
- Quench in room temperature water within 15 s after removal from the air furnace. The water bath needs to be large enough to remain below 43°C during the quench.
- Soak at 121°C ($\pm 6$°C) for 24 h. A recommended heating rate is approximately 15°C/h.
- Air cool to room temperature.

Strength of the 7xxx series of aluminum alloys relative to pure aluminum and other aluminum alloys can be seen in Fig. 3.1. This increased strength compared to other
aluminum alloys is one reason 7075 is attractive for use in the aerospace industry. Surface finishes were again measured using the Taylor-Hobson surface contact profilometer described in Section 3.1.1. The average measured \( R_a \) value for the 7075-T6 aluminum alloy fatigue samples was 0.095\( \mu \)m.

**Figure 3.1**: Strength values for aluminum alloys with various tempers. Data referenced from ASTM standards B211 [101] and B221 [102].

3.1.3 Specimen preparation

Round fatigue samples, for both materials, were machined from the stock rods according ASTM E466 [103]. Sub-tensile specimens were cut from the interior of the fatigue sample using wire-cut electrical discharge machining (EDM) once the fatigue tests were interrupted. Wire-cut EDM was chosen in order to minimize effects from conventional machining such as strain hardening or temperature increases that might alter the microstructure of the fatigued sample. It was desirable to obtain multiple
sub-tensile specimens from one fatigue sample in order to assume that each sub-tensile specimen had relatively the same amount of fatigue induced microstructural defects. In this fashion, a direct correlation could be made regarding strain rate on the material behavior by testing sub-tensile specimens which came from the same fatigue sample under different loading conditions. This approach and specimen design was followed to avoid generating only one sample apiece for quasi-static and dynamic loading, then assuming they reached the same $\frac{N}{N_f}$ level. Assuming the same $\frac{N}{N_f}$ level based only on the number of cycles can sometimes be unreliable due to inherent variability and scatter involved with fatigue loading. As a result, design of the sub-tensile test specimens had to ensure the ability to get multiple specimens out of a single fatigue sample, be compatible for both the quasi-static load frame and the dynamic loading apparatus, and follow as close as possible the subsize specimen dimensions recommended by ASTM E8 [104]. The final design also guaranteed the gauge section of the sub-tensile specimen came completely from the gauge section of the fatigued sample. Dimensions for both the round fatigue samples and sub-tensile dogbone specimens are specified in Fig. 3.2 with the sub-tensile specimens having a thickness of 2.00 mm.

### 3.2 Experimental hardware and testing

This section describes the setup and hardware for the three types of experimental testing performed on both materials for this dissertation research: fatigue testing, quasi-static tension testing, and dynamic tension testing. Plate impact shock
Figure 3.2: Dimensions of the round fatigue samples, location of the sub-tensile dogbone specimens inside the fatigue sample, and dimensions of the post-fatigue sub-tensile test specimens. Thickness of the sub-tensile dogbone specimens was 2 mm. The same sub-tensile dogbones are used in both quasi-static and dynamic experiments. All dimensions are in mm.

experiments was also performed, but only on the α-iron material samples. Therefore, details of the plate impact shock experiments are instead described in Section 4.2.4.

3.2.1 Fatigue testing

Load controlled fatigue experiments were performed on a servo-hydraulic MTS 370 load frame equipped with a 100 kN load cell. Grips were aligned to minimize lateral and shear strain prior to each fatigue test by using a dummy specimen and measuring strains induced during clamping. Strains were measured using the digital image correlation (DIC) software VIC2D from Correlated Solutions. Figure 3.3 shows
a dummy specimen in the unclamped and clamped states with the strain contours listing the maximum and minimum values of lateral strain. It can be seen that the amount of induced strain is minimal when the grips are properly aligned with each other. Alignment of the MTS grips was performed prior to every fatigue and quasi-static tension test. All fatigue experiments used a ramp loading profile, Peak-Valley Compensation (PVC), and had a maximum fatigue stress, $\sigma_{\text{max}}$, of 70% of the yield stress ($0.70 \sigma_y$). Macroscopic strain evolution during fatigue was measured using a 12 mm gauge length extensometer with a $\pm 1.08$ mm travel length. The extensometer was secured in the center of the gauge section using $3.175 \text{ mm } \times 0.406 \text{ mm } \times 19.05 \text{ mm}$ springs. Other fatigue test details that differed between the two materials are specified in their respective experimental results chapter; Chapter 4 for $\alpha$-iron and Chapter 5 for 7075-T6 aluminum alloy.

3.2.2 Quasi-static tensile testing

Quasi-static tension experiments on the sub-tensile specimens were performed using the same servo-hydraulic MTS machine with a different set of wedges installed and gripped between 500 psi and 750 psi. Tests were displacement controlled at a rate of $1 \text{ mm/min}$ leading to a strain rate of approximately $\dot{\varepsilon} = 1 \times 10^{-3} \text{ s}^{-1}$. Longitudinal strain was measured using DIC and speckle patterns were applied to the specimens first painting the specimen white with an airbrush equipped with a 0.3 mm needle, then speckling with matte black spray paint. Engineering stress, Eq. (3.1), was calculated using the MTS load cell output ($F$) and specimen cross-sectional area.
Figure 3.3: Lateral strain contours measured with DIC of a dummy specimen (a) before clamping the MTS grips and (b) after clamping the MTS grips. The amount of strain in the sample should not significantly increase due to holding it in place with the grips.

Longitudinal strain was gotten as the average strain from the DIC data points. A common type of strain calculation in DIC (and VIC2D) uses the Lagrangian strain tensor, which was converted to engineering strain using Eq. (3.2).

\[
\epsilon_{\text{eng}} = \sqrt{2\epsilon_{\text{lag}} + 1} - 1
\]
3.2.3 Dynamic tensile testing

Dynamic tension experiments were performed on the sub-tensile specimens using a modified Kolsky bar (Split-Hopkinson Pressure Bar). Kolsky bar systems have been comprehensively covered and a number of excellent references document the theory, history, concept, and operation of the technique to measure dynamic deformation cf. [64, 65, 105, 106, 107, 108, 109]. Sub-tensile specimens were press-fit into collars, with a pressure of 20 MPa to 28 MPa, which thread onto the incident and transmission bars. During testing, either a 300 mm or 610 mm long striker sleeve was launched into a threaded cap on one end of the incident bar to create a tensile wave. Pulse shapers between 18 to 22 American Wire Gauge (AWG) copper wires were wrapped around the incident bar next to the threaded end cap to remove oscillations in the loading wave and help maintain a constant strain rate during the loading pulse. The incident and transmission bars are 19 mm in diameter and 2.4 m and 1.8 m long, respectively. All bars and strikers are made from C350 maraging steel with strain gauges located approximately 900 mm from the bar-sample interface. Details of the apparatus are shown in Fig. 3.4. Stress in the sample is calculated using Eq. (3.3), which is derived from the typical Kolsky bar assumptions of one-dimensional elastic wave propagation and force equilibrium of the specimen, where $E_b$ is Young’s modulus of the incident and transmission bars, $A_b$ is the cross-sectional area of the incident and transmission bars, $A_s$ is the initial cross-sectional area of the test specimen, and
\( \epsilon_t \) is the measured transmission strain in the transmission bar.

\[
\sigma = \frac{E_b A_b}{A_s} \epsilon_T
\]  

(3.3)

Strain was measured using DIC on images collected from a high-speed Shimadzu HPV-2 camera. Dynamic compression testing using a Kolsky bar will normally use Eq. (3.4) to calculate the specimen strain rate as a function of time: where \( C_L \) is the longitudinal wave speed of the bars, \( l_0 \) is the initial length of the specimen, and \( \epsilon_r \) is the measured reflected strain in the incident bar.

\[
\dot{\epsilon}(t) = -\frac{2C_L}{l_0} \epsilon_r(t)
\]  

(3.4)

Specimen strain is then obtained by integrating Eq. (3.4).

\[
\epsilon(t) = \int_0^t \dot{\epsilon}(t) dt.
\]  

(3.5)

An important point to make is that Eqs. (3.4) and (3.5) assume that the displacement of the bar faces and the sample faces are equal. This assumption is valid during compression since the bar and sample faces remain in contact during the entire test. However, during a tension test the gauge section and grip section of the dogbone specimen deform non-uniformly invalidating this assumption. Therefore, a different method to measure strain must be used, necessitating the use of DIC in the strain measurements. Analysis software from REL Inc. was used to temporally synchronize...
the stress data obtained from the transmission bar strain gauge and the strain data obtained from DIC [110].

![Figure 3.4: Modified Kolsky Bar configuration for the dynamic tension experiments.](image)

### 3.3 Microscopy characterization

This section describes the microscopy equipment used for microstructural characterization.

#### 3.3.1 Scanning electron microscopy and electron backscatter diffraction

Material preparation for scanning electron microscope (SEM) included grinding with increasing grit SiC papers, polishing using 9μm to 1μm diamond suspension fluid, final polishing with a 0.05μm colloidal silica suspension to achieve a mirror-like finish, and finally etching with Kellers etchant. Microscope images were taken on a Hitachi S-3700N SEM operating with a power of 15kV, a probe current of 60mA, and a working distance 10mm–10.5mm from the sample. Additionally, an Oxford
X-MaxN energy dispersive spectroscopy (EDS) detector was used to obtain elemental composition information of observable features. Finally, an OXFORD Instruments NordlysMax3 electron backscatter diffraction (EBSD) detector with an electron column operating at 20 kV and 6.1 spot size was used to collect microstructure information. The MTEX toolbox, [111], in MATLAB® R2019a was used to analyze the EBSD data.

3.3.2 Micro X-ray computed tomography

Only the α-iron material utilized micro X-ray computed tomography (μXCT) in this dissertation research. Nevertheless, in-situ characterization of iron inclusion particles in the 7075 aluminum alloy can be performed with μXCT as demonstrated by [112, 113, 114, 115, 116]. In brief, μXCT measures the emission volume through a sample as the sample (or X-ray) rotates. The emission values are plotted as a function of the rotation angle to create a 2D plot called a sinogram. Additional filtering and processing of the sinogram data can be performed before back projecting and reconstructing the image from the filtered sinogram data. This process is repeated layer-by-layer to create a 3D image mapping of the part.

The post-fatigue sub-tensile α-iron samples were analyzed for voids using a Zeiss Xradia 510 Versa, a commercial μXCT system. This lab-based μXCT system consists of a Nordson DAGE transmissive x-ray tube with a tungsten target anode, which operates from 30–160 kV with up to 10 W of power, a dynamic spot size from 2 to 4 μm full width at half maximum (FWHM), and a maximum 34° full cone beam angle. For the α-iron, the microCT system was operated at maximum voltage and
power, i.e. 160 kV and 10 W, and using a filter designed by the manufacturer containing mostly tungsten (i.e. HE#3). The transmitted x-rays were absorbed by using a lens-coupled scintillator that has three objectives 0.4X, 4X, and 20X and then acquired by a 2.5 × 2.5 k charge-coupled device (CCD) with a physical pixel size of 13.5 µm × 13.5 µm. Based on the required resolution and sample size, 4X was selected with some geometric magnification and binning resulting in an approximately 3 µm × 3 µm detector element size. Image binning was adjusted on the $N/N_f = 0.83$ α-iron specimen giving a higher voxel resolution of approximately 1.5 µm × 1.5 µm. The reconstructed voxel resolution was isotropic, i.e. identical in all three dimensions. Two µXCT scans were acquired in order to cover a total volume of approximately $3 \times 3 \times 2$ mm$^3$. Reconstruction of tomography data used Zeiss algorithms which included some beam hardening correction and smoothing. Sub-tensile specimens at fatigue life ratios of $N/N_f = 0, 0.31, 0.62, 0.83, \text{ and } 0.94$ were 3D imaged. Images were processed in an academic version of the commercial software Dragonfly by Object Research Systems (ORS) Inc. Edges were cropped out from each image stack so only the interior of the sub-tensile specimen was analyzed. Images were thresholded in Dragonfly by allowing the software to use a gray scale cutoff value based on a selected void.

### 3.4 Data analytic approaches

This section describes the machine learning approaches utilized in the study, in addition to a novel statistical analysis methodology developed by the author.
3.4.1 Principal component analysis

One machine learning approach and analysis tool to describe correlations and trends between variables within a dataset is principal component analysis (PCA). Anything beyond three-dimensions becomes difficult to visualize and therefore more challenging to detect changes and interpret the data. The concept of PCA is to reduce dimensionality in a dataset while maintaining as much variance as possible [117]. While different types of PCA analysis exist, such as those where variables change over time (functional PCA [118,119]), those that are not as sensitive to data outliers (robust PCA [120,121]), and those that work on other types of data structures such as histograms (symbolic PCA [122,123]), standard PCA can still be broadly applied to produce meaningful insights.

In general, singular value decomposition is used to break the matrix of data into three matrices such that:

\[ X = U \Sigma V^T \]  

(3.6)

Where \( X \) is either the covariance or correlation matrix, \( U \) is the left singular vectors of \( X \), \( V \) is the right singular vectors of \( X \), and \( \Sigma \) are the singular values. The decision to use either a covariance or correlation matrix for \( X \) depends on the measurements themselves. It is common to use the correlation matrix when variables have different measurement units (for example comparing the price of a house in \( $ \) to its size in \( \text{ft}^2 \)) or for measurements that have different relative scales (such as comparing velocity of celestial objects in \( \text{km/s} \) to velocity of vehicles in \( \text{km/h} \)). Checking that the input data for each variable is normalized to a mean of zero and standard deviation of one
(known as the z-score) prior to computing the principal components is important to remember in these situations. The matrices $U$ and $V$ are the eigenvectors of the matrix $XX^T$ and $X^TX$, respectively. They have the properties of $U^TU = I$ and $V^TV = I$. In addition, $\Sigma = \lambda^{1/2}$ where $\lambda$ are the diagonalized eigenvalues of the matrix $XX^T$. Further mathematical descriptions of PCA, such as calculation of a covariance matrix, can be found in [124,125,126].

There is no one right way to interpret PCA results, but a common technique is to use a biplot and look at both the principle component scores and loadings together. Principle component scores refers to the original data matrix values transformed into principle component space and can be denoted as $F$. The matrix $V$ from Eq. (3.6) is also called the loading matrix and represents the vectors of each variable in the principle component plane. Therefore, the transformation of the original data to the principle component planes is expressed as:

$$F = XV \quad (3.7)$$

### 3.4.2 Density-based spatial clustering of applications with noise

Density-based spatial clustering of applications with noise (DBSCAN) is an unsupervised machine learning algorithm that automatically groups data into clusters. Benefits of the DBSCAN algorithm include requiring no prior knowledge about the data, its ability to cluster data into arbitrary shapes, and it is computationally efficient when scaling up to large datasets [127]. Each point in the dataset is analyzed based on two input parameters to group it into one of three different types of points. The
two input parameters are a neighborhood size, $\epsilon$, and a minimum number of points, $MinPts$. The three types of points are:

1. Core point–a point that has at least $MinPts$ in its $\epsilon$ neighborhood.

2. Border point–a point that has less than $MinPts$ in its $\epsilon$ neighborhood, but resides in a core point’s neighborhood.

3. Noise point–a point that is either a core point or a border point and will not belong to any cluster.

Figure 3.5 shows an illustration of each type of point. It is easy to identify, and if necessary remove, outlier points and noise in datasets when using the DBSCAN algorithm. In addition, finding the border between two overlapping clusters is straightforward, at which point other fuzzy methods might then be used to estimate probabilities of belonging to either group. A process to objectively select the input parameters, $\epsilon$ and $MinPts$, is discussed in Section 5.2.3.3 of the results.

3.4.3 K-nearest neighbor

A popular supervised machine learning method for classification or regression prediction is the K-nearest neighbors (KNN) algorithm. The approach to using nearest neighbors for predicting responses (classification for the purpose of this dissertation research) dates back to the early 1950s [128] and has the benefit of not requiring information about the data distribution. A KNN classifier is created using known data called a training dataset. Any arbitrary query point can then be classified
Figure 3.5: Illustration of the three point types determined from DBSCAN clustering. The green point has a total of seven other points in its neighborhood, larger than the $MinPts = 4$ criterion, making it a core point. The blue point has only three other data points in its neighborhood (less than $MinPts = 4$) but it resides inside the green circle making it a border point. The red point has less than $MinPts = 4$ in its neighborhood and does not reside inside a core neighborhood making it a noise point.

by analyzing a set number, K, of neighboring points in the training dataset. Point-to-point distances between the query point and each value in the training dataset are calculated using a specified distance metric. The Euclidean distance, $d$ calculated from Eq. (3.8), between points is frequently used.

$$d = \sqrt{(x_1 - x_2)^2 + (y_1 - y_2)^2}$$ (3.8)

Classification of a query point is decided on by using either a majority voting or weighted distance method. Majority voting classifies a query point to the class
with the highest estimated probability among the K-nearest neighbors. For example, if \( K = 5 \), and there are three occurrences of “Group A” and two occurrences of “Group B” among the five nearest neighbors, then the query point will be classified as “Group A”. Issues may arise when there is an equal frequency of occurrence among groups. Such as three occurrences of both “Group A” and “Group B” if \( K = 6 \), or two occurrences of “Group A” two occurrences of “Group B” and one occurrence of “Group C” for \( K = 5 \) when more than two classifications exist. A specified weighting function can be applied to each distance to avoid this situation. For instance, an inverse weighting function, Eq. (3.9), can be applied to each distance and the query point will then be classified to the group with the largest weighted distance total out of the K-nearest neighbors. Thus, majority voting classification weights each neighbor equally whereas closer neighbors have a stronger influence on the outcome in weighted distance classification.

\[
W_i = \frac{1}{d_i} \tag{3.9}
\]

### 3.4.3.1 Repeated k-fold cross-validation

It should be apparent that the selection of K will have an influence on the final classification outcome, so it is prudent to use an informed process to pick the K value opposed to a subjective selection. Cross-validation is a technique which uses the training dataset to optimize the input parameters into a function. It works by removing a chunk of data from the training dataset, then creating the classifier, and finally testing the predictive capability on the chunk of data that was removed. For
example, if a training dataset has $n$ number of total data points and $p$ data points were removed, the classifier would be created using the $n - p$ dataset, and finally tested on the removed $p$ points. Scoring metrics for the classifier’s performance are calculated by comparing the predicted classification of the $p$ points to the actual classification as a function of K. The K value that results in the best performance score is chosen as the input parameter when classifying new data points. Two common performance metrics include the accuracy and $F_1$ score. Accuracy is simply the number of correct classifications divided by the total number of classifications. However, accuracy scoring for a dataset that has a large imbalance between two classes can be misleading [129]. A more appropriate metric in these situations is the $F_1$ score. In a classification problem there are four outcomes: true positive (TP), true negative (TN), false positive (FP), and false negative (FN). Defined in Eq. (3.10), the $F_1$ score is the harmonic mean of the two terms, precision and recall.

$$F_1 = \frac{2(\text{Precision} \times \text{Recall})}{\text{Precision} + \text{Recall}} \quad (3.10)$$

where

$$\text{Precision} = \frac{\text{TP}}{\text{TP} + \text{FP}}$$

$$\text{Recall} = \frac{\text{TP}}{\text{TP} + \text{FN}}$$

An extension of cross-validation is a method called k-fold cross-validation that breaks the original training dataset into k chunks of approximately equal size. Each chunk then takes a turn being the tested dataset. Figure 3.6 depicts the k-fold cross-
validation process where an initial training dataset is divided into five different parts. Five different models are trained using the four parts colored in blue and tested using the one part colored in orange. A performance score is calculated for each of the five models and then averaged to end up with an overall score for the model. A value of \( k = 5 \) or \( k = 10 \) is commonly selected for k-fold cross-validation [126,130,131].

**Figure 3.6:** Depiction of data division in k-fold cross-validation for \( k = 5 \). The blue colored folds represent the new training dataset used to create the model and the orange colored folds represent the withheld test points. A performance metric can be calculated for each model and averaged to produce a total model score.

A further extension is repeated k-fold cross-validation (sometimes called Monte Carlo cross-validation) where the random assignment of data into each fold is repeated over multiple iterations. The k-fold cross-validation process depicted in Fig. 3.6 is repeated over a specified number of iterations. Each iteration performs a different
random data assignment into each fold as shown in Fig. 3.7. An average of the model’s performance metric (in this case the $F_1$ score) can be calculated just like it was with k-fold cross-validation. Increasing the number of iterations will decrease uncertainty in the estimated performance, but can substantially increase the computational requirement.

**Repeated k-fold cross-validation**

Data from the original dataset is randomly assigned to the folds over multiple iterations.

**Figure 3.7:** Depiction of repeated k-fold cross-validation. Data from the original dataset is randomly assigned to the folds over multiple iterations.

### 3.4.4 Statistical analysis of datasets

Image resolution (i.e. pixel or voxel size) of microscopy techniques is sometimes confused with the size of features that are able to be determined from that specific technique. However, image processed data containing only a single pixel/voxel cannot accurately represent shape parameters of the feature and in some cases the single pixel is noise and not representative of a feature at all. In many cases, a minimum number of pixels/voxels is selected that a feature must contain in order to be ana-
lyzed, yet selecting that number of minimum pixels in a non-subjective way is less straightforward. Therefore, a data analysis methodology was developed to determine the feature resolution of physically relevant metrics of data obtained from segmented image sets to solve this problem [132]. The developed method determines the best-fit distribution curve of a dataset by analyzing a truncated portion of the data. An effective resolvable size to know the frequency of occurrence for the metric of interest is established when including parts of the truncated dataset results in exceeding a specified error tolerance. As such, this method allows for the determination of the feature resolution regardless of the processing parameters or imaging instrumentation. Additionally, the number of missing objects that exist below the resolution of the instrumentation may be estimated. Application of the developed method was demonstrated on data obtained via 2D SEM and from 3D $\mu$XCT. It was shown that the minimum number of pixels/voxels required for accurate determination of a physically relevant metric was dependent on the metric of interest. This methodology of utilizing the prior knowledge of the distribution of metrics of interest was found to be well suited to determine the feature resolution in applications where large datasets can be achieved. Further details and an in-depth discussion of the developed statistical methodology is presented in Appendix A.
CHAPTER 4

α-IRON EXPERIMENTAL RESULTS

This chapter summarizes experimental results for the α-iron material. First, subsequent microscopy characterization including micro X-ray computed tomography (µXCT), optical imaging, and electron backscatter diffraction (EBSD) is shown and the observed microstructural evolution is described. Second, subsequent mechanical testing results including quasi-static tension and dynamic tension experiments are presented. Effects of observed microstructural changes on the subsequent mechanical behavior under each type of loading is discussed. Finally, results from a series of plate impact shock experiments to measure spall strength as a function of fatigue life are presented.

4.1 Microscopy observations

4.1.1 Micro X-ray computed tomography

Sub-tensile specimens at fatigue life ratios of $N/N_f = 0, 0.31, 0.62, 0.83,$ and 0.94 were 3D imaged to analyze development of voids throughout the material volume. Image stacks from the µXCT scans were transformed for the void analysis in 2D slices moving through the material thickness from front-to-back through the sub-tensile
specimen gauge section. The image orientation relative to the sub-tensile specimen is detailed in Fig. 4.1, and all $\mu$XCT analyses are oriented in the same fashion. Any identified void with a zero aspect ratio or smaller than $300 \mu m^3$ was removed from the analysis. Aspect ratio refers to the ratio of minor to major diameter of voids and a zero aspect ratio identifies those where the minor diameter was small enough to be considered erroneous, e.g. voids appearing in only one plane. Any remaining voids were then manually checked in the corresponding X-ray images and only included in the analysis if there was a clear visible discontinuity in the images at the void location. Manual verification of each detected void eliminated potential errors, such as streak artifacts, that can be created during image reconstruction.

Figure 4.1: Orientation of sub-tensile specimen (left) relative to the fatigue loading direction and X-ray scanning. In this dissertation research, all X-ray scans were reconstructed to create images (right) going through the entire thickness of the sub-tensile specimen along the z-axis, front-to-back.
Defect analysis in Dragonfly involved selecting a global threshold for surface determination to extract the volume inspected. The defect analysis was processed using proprietary algorithms for the extracted volume by defining a gray scale value representative of a defect and then filtering the results by aspect ratio, individual void volume, and finally manual verification. Histograms showing the distributions of the calculated void volumes and aspect ratios were similar between all specimens. Distributions for the volume and aspect ratio at each fatigue life are shown in Figs. 4.2 and 4.3, respectively. Each histogram was binned using the same equally spaced intervals for the bin edges in each dataset. The volume distributions of the voids showed a much higher occurrence of smaller sized voids and an approximately even distribution of voids above 2000 µm$^3$. The aspect ratio distributions showed that most of the voids were elliptical in shape and there were no spherically shaped voids. Each sample displayed approximately a log-normal size distribution and there was a fairly even occurrence of larger sized voids after the initial drop-off from the highest frequency at the smallest sizes. Average void volume was determined by using the peak of a log-normal distribution fit to the histogram data. There was an increase of about 50% in the average void volume between the first and final fatigue life ratio, 31% and 94%, respectively. Statistics from the void analysis are summarized in Table 4.1 and shows both the average void volume and total void ratio increased from the beginning to the end of the fatigue life. Void ratio is defined as the total volume of voids divided by the total volume of scanned material. Also of interest, was the notable amount of detected defects in the as-received material. There is confidence that the manufacturing process induced a non-negligible amount of defects since each
void making up the statistics in Table 4.1 was manually verified. The aspect ratio
distribution indicated the shape of the voids were more elliptical than spherical. A
rendering of the largest void at each fatigue life ratio is presented in Fig. 4.4 and shows
both the volume increase and morphology of the void as the fatigue life progressed.

Table 4.1: Detected void statistics from the image analysis on the sub-tensile X-
ray computed tomography scans. Voxel resolutions in the tomography scans were
between 1.5\(\mu m\) to 3\(\mu m\).

<table>
<thead>
<tr>
<th>Fatigue ratio ((N/N_t))</th>
<th>Average void volume ((\mu m^3))</th>
<th>Largest void volume ((\mu m^3))</th>
<th>Voids &gt; 300 (\mu m^3)</th>
<th>Total void ratio (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1070</td>
<td>5400</td>
<td>53</td>
<td>0.00048</td>
</tr>
<tr>
<td>0.31</td>
<td>650</td>
<td>3100</td>
<td>46</td>
<td>0.00021</td>
</tr>
<tr>
<td>0.62</td>
<td>740</td>
<td>5400</td>
<td>51</td>
<td>0.00032</td>
</tr>
<tr>
<td>0.73</td>
<td>910</td>
<td>8800</td>
<td>46</td>
<td>0.00026</td>
</tr>
<tr>
<td>0.83</td>
<td>705</td>
<td>10300</td>
<td>101</td>
<td>0.00043</td>
</tr>
<tr>
<td>0.94</td>
<td>1000</td>
<td>9700</td>
<td>126</td>
<td>0.00057</td>
</tr>
</tbody>
</table>

Figure 4.2: Volume distribution for all of the detected voids in the sub-tensile specimen at each fatigue life ratio. (a) \(N/N_t = 0\), (b) \(N/N_t = 0.31\), (c) \(N/N_t = 0.62\), (d) \(N/N_t = 0.73\), (e) \(N/N_t = 0.83\), and (f) \(N/N_t = 0.94\). Note the logarithmic x-axis.
Figure 4.3: Aspect ratio distribution for all of the detected voids in the sub-tensile specimen at each fatigue life ratio. (a) $N/N_f = 0$, (b) $N/N_f = 0.31$, (c) $N/N_f = 0.62$, (d) $N/N_f = 0.73$, (e) $N/N_f = 0.83$, and (f) $N/N_f = 0.94$.

The overall distribution of voids for a sub-tensile specimen at each portion of the fatigue life is shown in Figs. 4.5 to 4.10. Each figure contains X-ray images of the highlighted voids to show their morphology and positioning to neighboring voids. Coordinates of the voids are also shown to illustrate that the observed defects happen in the bulk material and are not surface flaws. Bulk dislocation activity is believed to be responsible for changes in the void ratio, first a decrease from 0.00048% to 0.00021% then an increase back up from 0.00021% to 0.00057% at the end of the fatigue life. Studies of fatigue in nickel found that PSBs also form in the bulk material, not just the surface, and that bulk PSBs can lead to grain boundary displacements [40]. Therefore, it is reasonable to assume that dislocation activity, irreversibility,
and accumulation during fatigue loading caused the internal void changes observed in the μXCT scans.

There was a clear progression in the void characteristics throughout the fatigue life that can be explained through the measured macroscopic strain evolution response. Processing defects, in the form of internal voids, in the as-received material were detected with the largest void being approximately $3000 \mu m^3$, see Fig. 4.5 or Fig. 4.4. Defining an initial reference state for the as-received material meant defects due to preparation were not a major concern, even though the amount of defects observed was greater than expected. After 31% of the fatigue life both the average void volume and total void ratio was cut in half going from $1470 \mu m$ to $780 \mu m$ and...
0.00048% to 0.00021%, respectively. In addition, there was little change in the morphology and grouping of the voids. This portion of the fatigue life experienced no significant accumulation of macroscopic strain. Linking observations from the \( \mu \)XCT imaging, fatigue testing, and post-fatigue sub-tensile testing (presented in Section 4.2) shows that microscopic deformation mechanisms which are activated during elastic fatigue loading have the potential to alter bulk properties.

Changes in the void arrangement were noticed when advancing to 62% and 73% of the fatigue life. Not only did the average void volume and total percentage of voids increase, there were several collections of voids that laid along the same plane parallel to the loading direction. The sequence of X-rays in Figs. 4.7 and 4.8 show that multiple voids appear next to each other, whereas prior analyses showed only singular voids. For the fatigue life ratios of \( N/N_f = 0.31, 0.62 \) and \( 0.73 \) the accumulation of macroscopic compressive strain helps explain the decrease in average void volume and total void ratio relative to the as-received material. The buildup of residual compressive strain with the number of fatigue cycles works to “squeeze” some of the processing defects out. On the other hand, increases in the average void volume and largest void size between 31% and 73% of the fatigue life could be attributed to the continued activation and accumulation of dislocations along grain boundaries. This assertion is supported by the optical microscope images of Fig. 4.11 showing that voids and cracks in the fatigued material occurred along grain boundaries.

Furthermore, grouping of voids along the loading axis was noticed immediately after the first indication of accumulated macroscopic strain. Void growth has been attributed to plastic deformation of the surrounding grains [133]. However, plastic
Figure 4.5: Locations of voids greater than 300µm³ in the $N/N_f = 0$ sub-tensile specimen. Observed voids were elliptical in shape and several were detected in the bulk material. Images from the µXCT scans were taken in the X-Y plane with each step occurring in the Z-direction with the specified step size of 3µm. Approximate location of the voids are listed in mm from the indicated origin in the 3D figure. All pixels encompassed by the void in each x-ray image have been colored black for clarity.

deformation in a majority of grains is not necessarily happening during the fatigue loading portion of this study because imposed global stresses are a fraction of the yield stress. It is more likely that plastic deformation is limited to specific grains, such as those favorably oriented for a high resolved shear stress and PSB formation. Therefore, it might be inferred that void nucleation in these specific grains might
Figure 4.6: Locations of voids greater than 300 $\mu$m$^3$ in the $N/N_f = 0.31$ sub-tensile specimen. Observed voids were similar in nature to the as-received material. Images from the $\mu$XCT scans were taken in the X-Y plane with each step occurring in the Z-direction with the specified step size of 3 $\mu$m. Approximate location of the voids are listed in mm from the indicated origin in the 3D figure. All pixels encompassed by the void in each x-ray image have been colored black for clarity.

be more likely to occur than void growth since surrounding grains do not experience plastic deformation on account of the elastic loading conditions. This argument could perhaps explain the increase in overall void ratio but not an increase in the number of voids greater than 300 $\mu$m$^3$ between 0 % and 62 % of the fatigue life.

At 83 % of the fatigue life, changes to the internal voids became more distinct. The total number of voids and total void ratio increased from the 73 % fatigue life increment. On the other hand, the average void volume saw a decrease that could be due to the binning of images mentioned in Section 3.3.2. The smaller voxel size meant that there was a higher frequency of smaller voids near the 300 $\mu$m$^3$ limit that will shift the log-normal distribution fit to the left. The largest void shown in Fig. 4.4 for
Figure 4.7: Locations of voids greater than 300µm³ in the $N/N_f = 0.62$ sub-tensile specimen. A set of voids grouped along a line parallel to the loading direction is now observed that were not present in the previous specimens. Voids had similar shapes but larger average sizes compared to the sub-tensile specimen earlier in the fatigue life. Images from the XCT scans were taken in the X-Y plane with each step occurring in the Z-direction with the specified step size of 3µm. Approximate location of the voids are listed in mm from the indicated origin in the 3D figure. All pixels encompassed by the void in each x-ray image have been colored black for clarity.

$N/N_f = 0.83$ was analyzed independently because the thresholding values to capture the entire void resulted in a large amount of erroneous void detections. Edges of the largest void had higher gray scale values (i.e. the edges were less black than the bulk of the void) resulting in a higher threshold cutoff value to encompass the entire void. As a result of the higher threshold cutoff value, thousands of erroneous voids (i.e. noise) were detected if those thresholding parameters were used throughout
Figure 4.8: Locations of voids greater than 300 $\mu$m$^3$ in the $N/N_f = 0.73$ sub-tensile specimen. Results of the $\mu$XCT scan were similar to the $N/N_f = 0.62$ fatigued material in the case that multiple sets of voids could be seen grouped parallel to the loading direction. However, both the average void volume and maximum void size increased from the previous fatigue life ratio. Images from the $\mu$XCT scans were taken in the X-Y plane with each step occurring in the Z-direction with the specified step size of 3$\mu$m. Approximate location of the voids are listed in mm from the indicated origin in the 3D figure. All pixels encompassed by the void in each x-ray image have been colored black for clarity.

the entire volume—an unmanageable number of voids to manually check. Finer voxel resolutions at this fatigue life ratio, 1.5$\mu$m compared to 3$\mu$m for the other $\mu$XCT scans, was thought to be the cause of the widespread noise when thresholding and segmenting to include the entirety of the largest void. Therefore, this fatigue life ratio was the only sub-tensile specimen that underwent a separate analyses of the
largest sized void. An independent analysis of the largest void explains the perceived discrepancy in maximum void volume between Fig. 4.4 and Fig. 4.9.

Most notable at this portion of the fatigue life was the increase in voids grouped together along lines parallel to the loading direction. There were more than thirty collections of vertical voids counted throughout the scanned volume, whereas the previous fatigue life measurement had only two. Figure 4.9 denotes collections of voids that were grouped together along the loading axis and it is seen that they occur throughout the entire bulk material and are not localized in one section. Furthermore, the X-ray images in Fig. 4.9 highlight the typical length of the void groupings. It can be surmised that accumulation of macroscopic tensile strain led to two pronounced changes in the void characteristics, since this specimen corresponded to the region of strain evolution immediately after the tensile response became dominate. First, a marked increase in the number of voids grouped together parallel to the loading axis, and second an increase in the overall number of voids.

Finally, at 94% of the fatigue life, groups of voids collected along the loading axis were again prevalent throughout the volume. An increase in the length of the void groupings was also noticed. Pictured in Fig. 4.10 are two of these vertical collections that extended approximately 2 mm along the loading axis, much longer than the 800 μm maximum length at 83%. Both the average void volume and total void ratio continued to increase as would be expected since there was a greater net tensile strain accumulation in the material at this portion in the fatigue life. The largest void size nearly doubled, the average void volume increased after an initial decline, and the total void ratio increased as a result of elastic fatigue loading.
$N/N_f = 0.83$  Fatigue loading direction

**Figure 4.9:** Collections of voids along the same axis, parallel to the fatigue loading direction, in the $N/N_f = 0.83$ sub-tensile specimen. Each black line in the 3D volume represents a collection of voids that are aligned in the Y-axis parallel to loading. Six of these collections are boxed and the corresponding x-ray images are shown as a representation of the length over which voids are grouped together. An increase in the grouping of voids parallel to the loading direction was observed at this fatigue life ratio. All pixels encompassed by the void in each x-ray image have been colored black for clarity.
This step-by-step $\mu$XCT analysis over the entire fatigue life demonstrates the evolution of microscopic voids throughout the material’s volume. How these observed void characteristics are related to the macroscopic strain behavior and subsequent mechanical property evolution is discussed in Section 4.2. In summary, the initial fatigue loading showed a decline in the overall number of voids and void volume. Voids began to form along lines parallel to the loading direction at the onset of accumulated macroscopic plastic strain. Then, a significant increase in the number of voids collected along lines parallel to the fatigue loading direction occurred once tension dominated the macroscopic strain response. Finally, the overall length of void groups along the loading direction grew with further accumulation of tensile strain.

4.1.2 Optical microscopy

Material at $N/N_f = 0.73$, 0.83 and 0.94 was imaged with a Zeiss Axio light microscope and 503 Axiocam camera in order to visualize the location of internal voids relative to the microstructure. It was observed that voids induced during fatigue, which were visible after polishing, always corresponded to displacements between different grains and not an intragranular crack. Optical microscope images shown in Fig. 4.11 were taken from material at $N/N_f = 0.73$ and 0.94 and show that void locations predominantly occurred along grain boundaries. The direction of fatigue loading for the images in Fig. 4.11 was vertical. Similar observations were made for material at $N/N_f = 0.83$. 

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Figure 4.10: Collections of voids along the same axis, parallel to the fatigue loading direction, in the $N/N_f = 0.94$ sub-tensile specimen. Several sets of void groups appear over a greater length compared to those detected in the $N/N_f = 0.83$ material. In this instance the x-ray images move through the x-direction for a total of approximately 30µm. Images between those shown have been omitted for clarity. All pixels encompassed by the void in each x-ray image have been colored black for clarity.
Figure 4.11: Optical microscope images showing the location of internal voids after loading to a certain percentage of the fatigue life: (a) 73% and (b) 94%. Material was located approximately 3 mm below the surface. In each case voids, denoted by the arrows, occurred along grain boundaries. The fatigue loading direction was vertical.

4.1.3 Electron backscatter diffraction

Material from different stages of the fatigue life, 0%, 31%, 83% and 94%, were imaged with EBSD to ascertain changes in the grain structure. Figure 4.12 shows the α-iron grain structure in the as-received material and at the beginning of the fatigue life. The as-received material exhibited an initial substructure in the grains likely due to the manufacturing process. Both the grains and substructure, from a zoomed in EBSD scan, are shown in Figs. 4.12a and 4.12b, respectively. On the other hand, Fig. 4.12c (taken at \( N/N_f = 0.31 \)) displays more homogeneous grain interiors than the as-received material and elimination of the substructure. Distribution of the grain area for the EBSD image at each fatigue condition is shown in Fig. 4.13. A misorientation limit of 1° was used in the EBSD processing, so some artificial
noise is included in the analysis, but the difference in the number of small grains is obvious. The as-received material has an order of magnitude more smaller sized grains than the pre-fatigued material indicating that the application of cyclic loading is responsible for removal of the substructure. As discussed in Section 2.1, fatigue loading causes dislocation motion even when the applied stresses are well below the macroscopic yield stress. Bulk dislocation motion has the ability to eliminate some of the substructure grain boundaries. Elimination of this substructure is thought to be the reason for initial strength decreases during both quasi-static and dynamic loading that is unexplained by the observed volumetric void configuration.

Additional EBSD scans on material from late in the fatigue life, 83% and 94%, were also obtained. It was known, from the $\mu$XCT characterization, that introduction of voids throughout the bulk material occurred at these fatigue life ratios. Therefore, the EBSD scans focused on material surrounding these voids. Figure 4.14 shows a representative EBSD scan containing a fatigue-induced void at both 83% and 94% of the fatigue life. Voids were found by continually removing material through grinding until a void became visible under an optical microscope.

4.2 Mechanical testing results

4.2.1 Fatigue testing and strain evolution

Load controlled fatigue tests on the $\alpha$-iron were performed at a frequency of 1 Hz and were fully reversible (i.e. zero mean stress and a stress ratio of $R = -1$). Material response during fatigue was quantified by measuring macroscopic strain
Figure 4.12: Electron backscatter diffraction images of the as-received and 31% fatigued \(\alpha\)-iron material. Images show there is an initial substructure in the as-received material that is eliminated after initial fatigue loading. (a) Wide field of view EBSD image of the as-received material (b) Close up view of a single grain showing in greater detail the substructure in the as-received material. (c) Wide field of view EBSD image of material fatigued to 31% of the fatigue life showing elimination of the substructure. (d) Inverse pole figure map.

accumulation throughout the fatigue life via the attached extensometer. Macroscopic strain evolution during fatigue of \(\alpha\)-iron is shown in Fig. 4.15. A representative strain evolution curve as a function of fatigue life is shown in Fig. 4.15a. The test was
Figure 4.13: Grain size distribution of the (a) as-received and (b) 31% fatigued α-iron material. Analysis of the EBSD scans show a much larger number of smaller sized grains in the as-received material indicating elimination of the fine grain substructure. Elimination of the substructure caused a subsequent drop in strength of mechanical properties.

Figure 4.14: Electron backscatter diffraction images of the (a) 83% and (b) 94% fatigued α-iron material. Each EBSD scan contains a fatigue-induced void. The grain orientations follow the inverse pole figure map of Fig. 4.12d.
normalized to the number of cycles to failure. Four distinct regions of macroscopic strain behavior were observed. Region 1 had minimal changes in strain under both tension and compression loads. This initial region was expected because the fatigue stress amplitude remained below the elastic limit. Region 2 refers to the cycles during which measured strain increased under compression but remained constant under tension. Region 3 refers to the portion of fatigue life when measured strain changed during both tension and compression, but in different directions. Finally, region 4 refers to the last portion of the fatigue life where tension dominated the strain response of the specimen and overall net strain eventually becomes positive even at the peak compressive load. The amount of fatigue life spent in Region 3 is thought to be small due to the dominance of the tensile strain in the material response after it starts accumulating. These four characteristic regions in the strain evolution are exclusive to α-iron and can change depending on the deformation mechanisms activated during the fatigue loading.

The characteristic features observed in the strain evolution was repeatable and used as a real-time indication of where in the fatigue life the sample was. Strain evolution, and the four notable regions, can be seen for a majority of the completed tests in Fig. 4.15b. Monitoring and stopping the fatigue tests based on instantaneous strain measurements alleviated any issues arising from fatigue scatter and a different number of cycles to failure each specimen. Finally, these results also show a distinct tension-compression asymmetry in the material behavior that would be expected for a body-centered cubic (BCC) metal cf. [134, 135, 136, 137]. It also indicates that whatever deformation mechanisms and microstructural irreversibilities are activated
Figure 4.15: Macroscopic strain evolution during load controlled fatigue of $\alpha$-iron as measured by attached extensometer. (a) Detailed strain evolution of a fatigue specimen tested until failure. Dashed lines indicate the points where fatigue tests were interrupted for subsequent mechanical testing and microscopic characterization. (b) Strain evolution for a majority of the completed tests. Strain evolution is consistent regardless of the number of cycles required to fail the specimen.

under tension eventually dominate material behavior during fatigue. The number of specimens tested and characterized at each fatigue condition is shown in Table 4.2.

Table 4.2: Overall test matrix detailing the total number of post-fatigue sub-tensile specimens characterized and tested.

<table>
<thead>
<tr>
<th>$N/N_f$</th>
<th>Fatigue specimens</th>
<th>$\mu$XCT specimens</th>
<th>Quasi-static specimens</th>
<th>Dynamic specimens</th>
<th>Shock specimens</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
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<td>1</td>
<td>1</td>
<td>2</td>
<td>3</td>
</tr>
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<td>Failure</td>
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4.2.2 Quasi-static testing and effect of microstructural evolution

Quasi-static engineering stress-strain curves for each fatigue level are shown in Fig. 4.16, graphical results in Fig. 4.17, and tabulated results in Table 4.4. Measurement uncertainties were propagated through the analysis following the process outlined by Kline and McClintock [138]. Parameters for the DIC instrumentation (for both the quasi-static and dynamic experiments) are given in Table 4.3. These parameters are the minimum suggested by Reu to report in scientific publications [139]. A continual decrease of ductility due to the pre-fatigue loading until the very end of the fatigue life was observed. At $N/N_f = 0.94$ the softening behavior changed and the total amount of strain increased. However, strength of the pre-fatigued material did not exhibit a similar increase at the end of fatigue life. Instead there was a decrease in both the yield stress and UTS. More specifically, yield strength was shown to decrease continually as a function of fatigue life, and up to 20% overall. On the other hand UTS remained within experimental uncertainty, after an initial drop-off, until $N/N_f = 0.73$ at which point it eventually decreased by approximately 50 MPa, or about 10%. It was also found that the strain value at UTS remained constant regardless of any prior fatigue loading even though the strength value changed. Thus, the toughness of the material, calculated as the integral of the stress strain curve and accounts for both strength and ductility, also decreased until the end of the material’s fatigue life.

Observed microstructural evolution and detected void changes in the bulk of the material can help explain the measured changes in mechanical behavior. The initial drop-off in the strength properties is likely due to the elimination of a fine
Table 4.3: DIC parameters for quasi-static and [dynamic] experiments.

<table>
<thead>
<tr>
<th>Device and Measurement Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
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<td>Camera</td>
<td>Point Grey Grasshopper 3 [Shimadzu HPV-2]</td>
</tr>
<tr>
<td>Image Size</td>
<td>2448x2048 pixels [312x260 pixels]</td>
</tr>
<tr>
<td>Lens</td>
<td>Tamron 180 mm macro lens [Tamron 180 mm macro lens]</td>
</tr>
<tr>
<td>Image Scale</td>
<td>68 pixels/mm [13 pixels/mm]</td>
</tr>
<tr>
<td>Images (Frame Rate)</td>
<td>225 (1 frame/sec) [102 (250k frames/sec)]</td>
</tr>
<tr>
<td>DIC Software</td>
<td>Correlated Solutions VIC-2D</td>
</tr>
<tr>
<td>Correlation Algorithm</td>
<td>Normalized Square Differences</td>
</tr>
<tr>
<td>Interpolation</td>
<td>Optimized 8-tap spline</td>
</tr>
<tr>
<td>Subset Size</td>
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<td>Step Size</td>
<td>5 pixels [4 pixels]</td>
</tr>
<tr>
<td>Measurement Points</td>
<td>8,000 [450]</td>
</tr>
<tr>
<td>Strain Window</td>
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</tr>
<tr>
<td>Smoothing Method</td>
<td>90% center weighted Gauss filter</td>
</tr>
<tr>
<td>Spatial Resolution</td>
<td>57 pixels [27 pixels]</td>
</tr>
<tr>
<td>Displacement Resolution</td>
<td>0.035 pixels [0.005 pixels]</td>
</tr>
<tr>
<td>Strain Resolution</td>
<td>440 $\mu$\epsilon$ (Lagrange)$ [470 $\mu$\epsilon$ (Lagrange)$]</td>
</tr>
</tbody>
</table>

grained substructure that was present in the material likely due to the manufacturing process and not the void evolution. Electron backscatter scans of the as-received material, Figs. 4.12a and 4.12b, clearly display an initial substructure within each grain that is no longer present in the $N/N_f = 0.31$ material, Fig. 4.12c. The classic Hall-Petch relationship dictates that a smaller grain size for the same material will have an increased strength due to additional barriers to dislocation motion. Equation (4.1) shows the Hall-Petch relationship where $\sigma_y$ is the yield stress, $\sigma_0$ is a material constant.
Figure 4.16: Quasi-static engineering stress-strain curves of $\alpha$-iron after the introduction of microstructural defects due to different amounts of pre-fatigue loading.

(a)  
(b)  

Figure 4.17: Mechanical properties of $\alpha$-iron under quasi-static uniaxial tension after the introduction of microstructural defects due to different amounts of pre-fatigue loading. (a) Strength and toughness as a function of the fatigue life. (b) Strength and void ratio as a function of fatigue life.
representing the initial stress required for dislocation motion, \( k_y \) is a material constant representing a strengthening coefficient, and \( d \) is the average grain diameter:

\[
\sigma_y = \sigma_0 + \frac{k_y}{\sqrt{d}} \tag{4.1}
\]

Therefore, the substructure in each grain acts as a strengthening mechanism accounting for the initial decrease in strength as it is eliminated during the onset of fatigue loading. As the fatigue progressed between \( N/N_f = 0.31 \) to 0.73 the yield stress decreased whereas the UTS remained constant. This portion of the fatigue life when the number of smaller voids, below 500\( \mu \)m\(^3\), increased but had yet to form into lines of voids parallel to the loading axis. Then, at \( N/N_f = 0.83 \) and continuing to \( N/N_f = 0.94 \) there was a pronounced drop in UTS, while yield stress continued to decrease at a similar rate. It is surmised from these results that the UTS decreases are due to an increase in the amount of void grouping parallel to loading. Furthermore, there were no observed changes in either the UTS or the propensity of voids to appear along the same vertical axis between \( N/N_f = 0.61 \) to 0.73. This observation is evidence that large amounts of vertical void groupings only appear in regions 3 and 4 of the strain evolution and are the likely cause of accumulated tensile strain. Therefore, it is possible that the drop in UTS was due to pre-existing voids along the loading direction resulting in an easier occurrence of intervoid shearing, i.e. the coalescence of voids localized on a single plane, one potential ductile fracture mechanisms detailed by Noell et al. [140].
In summary, the following observations were made regarding the microstructural evolution during fatigue and its effect on subsequent quasi-static mechanical properties. Removal of the process-induced, fine grained substructure resulted in an initial strength decrease. Yield stress decreases are associated with an increased number of small voids and UTS decreases are associated with the prevalent network of vertical void lines beyond the initial fatigue loading. This network of voids, grouped together parallel to the loading direction, is the probable cause of accumulated measured macroscopic tensile strain.

4.2.3 Dynamic testing and effect of microstructural evolution

Dynamic stress-strain curves at each fatigue life ratio are shown in Fig. 4.18, graphical results in Fig. 4.19, and once again the tabulated results in Table 4.4. Results differed from the quasi-static loading as the material exhibited a noticeable strain rate dependence. For one, the flow stress was substantially larger during the dynamic loading. A flow stress and strength increase at higher strain rates has been known and studied extensively in metals [141, 142, 143]. Second, the approximately 15% increase of maximum dynamic strength, after an initial decrease, of the material with progressively larger amounts of fatigue-induced voids was another trend that stood out from these results. The exception being one outlier at $N/N_f = 0.62$.

Increases in strength, after the initial drop-off, are somewhat counterintuitive when considering the number voids increased especially near the end of the fatigue life. However, these strength increases might be explained due to the activation of twinning under high strain rates. Activation of twinning can cause increases in
Table 4.4: Summary of results for quasi-static and [dynamic] tensile tests of $\alpha$-iron throughout the fatigue life.

<table>
<thead>
<tr>
<th>$N/N_t$</th>
<th>$0.2%\sigma_y$ (MPa)</th>
<th>$\sigma_{UTS}$ (MPa)</th>
<th>Toughness (MJ/m$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>dynamic</td>
<td>dynamic</td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>394 ± 9</td>
<td>441 ± 10</td>
<td>101 ± 3</td>
</tr>
<tr>
<td></td>
<td>[802 ± 30]</td>
<td>[134 ± 7]</td>
<td></td>
</tr>
<tr>
<td></td>
<td>[763 ± 26]</td>
<td>[129 ± 6]</td>
<td></td>
</tr>
<tr>
<td>0.31</td>
<td>380 ± 9</td>
<td>406 ± 9</td>
<td>97 ± 3</td>
</tr>
<tr>
<td></td>
<td>[639 ± 30]</td>
<td>[95 ± 5]</td>
<td></td>
</tr>
<tr>
<td></td>
<td>[612 ± 22]</td>
<td>[80 ± 4]</td>
<td></td>
</tr>
<tr>
<td>0.52</td>
<td>368 ± 9</td>
<td>408 ± 10</td>
<td>94 ± 3</td>
</tr>
<tr>
<td></td>
<td>[621 ± 21]</td>
<td>[108 ± 4]</td>
<td></td>
</tr>
<tr>
<td>0.62</td>
<td>349 ± 9</td>
<td>401 ± 9</td>
<td>91 ± 3</td>
</tr>
<tr>
<td></td>
<td>[664 ± 30]</td>
<td>[103 ± 5]</td>
<td></td>
</tr>
<tr>
<td></td>
<td>[753 ± 25]</td>
<td>[103 ± 5]</td>
<td></td>
</tr>
<tr>
<td>0.73</td>
<td>351 ± 9</td>
<td>405 ± 10</td>
<td>88 ± 3</td>
</tr>
<tr>
<td></td>
<td>[662 ± 29]</td>
<td>[102 ± 5]</td>
<td></td>
</tr>
<tr>
<td></td>
<td>[636 ± 24]</td>
<td>[111 ± 5]</td>
<td></td>
</tr>
<tr>
<td>0.83</td>
<td>326 ± 9</td>
<td>387 ± 9</td>
<td>78 ± 2</td>
</tr>
<tr>
<td></td>
<td>[645 ± 30]</td>
<td>[65 ± 4]</td>
<td></td>
</tr>
<tr>
<td></td>
<td>[671 ± 28]</td>
<td>[105 ± 6]</td>
<td></td>
</tr>
<tr>
<td>0.94</td>
<td>312 ± 8</td>
<td>358 ± 9</td>
<td>85 ± 3</td>
</tr>
<tr>
<td></td>
<td>[641 ± 22]</td>
<td>[101 ± 4]</td>
<td></td>
</tr>
<tr>
<td></td>
<td>[697 ± 25]</td>
<td>[94 ± 5]</td>
<td></td>
</tr>
</tbody>
</table>

strain hardening either through a decrease of the dislocation free slip distance, a Hall-Petch effect, or through twinned regions transforming glissile dislocations to sessile dislocations and increasing the hardness compared to the original matrix, the Basinski mechanism [144]. Zerilli and Armstrong developed a constitutive relationship for the flow stress required for twinning in BCC metals as a function of grain size, Eq. (4.2) [142]:

$$\sigma_T = \sigma_{T_0} + k_T d^{-\frac{1}{2}}$$

(4.2)
where $\sigma_T$ is the flow stress required for twinning, $d$ is the average grain diameter, and $\sigma_{T0}$ and $k_T$ are material parameters determined by experimentation. Material constants for iron are also given by Zerilli and Armstrong where $\sigma_{T0} = 330$ MPa and $k_T = 2.8$ MPa m$^{1/2}$ [142]. A required flow stress of approximately 690 MPa is calculated for twinning in the specific grain size of the iron used for the dissertation research. This estimation indicates the presence of twinning would be expected during the dynamic tests and helps explain the higher strength values in the dynamic stress-strain curves.

Increases in dynamic strength as a function of fatigue life is thought to be related to the increased amount of voids, which in turn raises the chance of heterogeneous twin nucleation by way of two possible mechanisms. First, the local stress field around a void or group of voids is redistributed [145, 146], resulting in stress concentrations which could assist twin nucleation. Second, a process of dislocation nucleation from the void might lead to slip assisted twinning. Studies have discussed dislocation generation at a void or particle in the surrounding matrix, [147, 148, 149], in addition to the fact slip can preclude twinning [150]. Both of these mechanisms help explain the observation of higher dynamic stresses throughout the fatigue life despite an increase in void size, quantity, and volume.

In summary, the following observations were made regarding the microstructural evolution during fatigue and its effect on subsequent dynamic mechanical properties. Strength increases are attributed to a higher number and larger size of fatigue-induced voids creating more localized stress concentrations that could potentially act as sites for twin nucleation. Extensive twinning leads to increases in strength by
either decreasing the mean free path for dislocation slip, increasing the hardness of the twinned region, or a combination of both. It should be reiterated that void evolution is not the sole contributor to the measured strength evolution in mechanical properties. The initial strength drop between 0% and 0.31% of the fatigue life is associated with the elimination of a fine grained substructure in the material. This finding demonstrates that other microscopic features such as grain geometry, texture, and dislocation density could also evolve during elastic fatigue leading to subsequent mechanical property changes.

**Figure 4.18**: Dynamic engineering stress-strain curves of α-iron after the introduction of microstructural defects due to different amounts of pre-fatigue loading.

### 4.2.4 Plate impact shock experiments and spall strength evolution

A series of plate impact shock experiments were conducted at the Army Research Laboratory (ARL) to measure the Hugoniot elastic limit (HEL) and spall
strength of $\alpha$-iron as a function of the fatigue life and peak shock stress. This section will review how the pre-existing fatigue-induced voids affected material response at strain rates of approximately $10^5 \text{s}^{-1}$. Discussion of the experimental setup is also given in this section since these experiments were only performed on the $\alpha$-iron material and not the 7075-T6 aluminum alloy.

Spall is the dynamic tensile loading of a material during uniaxial strain. Common experimental approaches to measure spall strength include plate impact shock experiments performed in a gas gun or from impact with an explosively launched flyer plate. The flyer plate is driven at high velocities ($>100 \text{ m/sec}$) and impacts the test specimen creating a compressive shock wave in both the test sample and flyer plate. Rarefaction, or release, waves originating from the free surface of both the test sample and flyer plate collide in the test sample. Tensile stresses caused by the overlap of the

Figure 4.19: Mechanical properties of $\alpha$-iron under dynamic uniaxial tension after the introduction of microstructural defects due to different amounts of pre-fatigue loading. (a) Strength and toughness as a function of the fatigue life. (b) Strength and void ratio as a function of fatigue life.
two rarefaction waves have the potential to nucleate, grow, and coalesce voids leading to an internal fracture plane inside the test sample called a spall plane. Experimental design of the test sample and flyer plate thicknesses ensure collision of the rarefaction waves occur in the middle of the test sample. Velocity-time history is often obtained through the use of a Velocity Interferometer System for Any Reflector (VISAR) or Photonic Doppler Velocimetry (PDV) to measure the velocity of the sample’s free surface, $u_{fs}$.

A simplified Lagrangian (X-t) diagram of the loading states and corresponding free surface velocity after impact is shown in Fig. 4.20. The faster running elastic precursor wave, followed by the slower compressive shock wave, propagates through the sample (to the right) and through the flyer plate (to the left). At $t_1$, when the elastic precursor wave arrives at the free surface, the VISAR or PDV velocimetry will begin to measure free surface velocity. The maximum free surface velocity due to the elastic precursor wave is referred to as the HEL and can be thought of as a measure of dynamic yield stress and the point where the material cannot undergo any more elastic deformation under the imposed uniaxial strain loading conditions. At $t_2$ the slower inelastic shock wave arrives at the free surface and reflects back into the material as a release, or rarefaction, wave and starts to unload the material. Unloading from the shocked state begins at $t_3$ when the rarefaction wave from the sample-flyer interface arrives the free surface. However, overlap of the two release waves in the material itself (one from the flyer and one from the sample’s free surface) causes a net tensile stress. Fracture will occur in the material, if this tensile stress is large enough to nucleate, grow, and coalesce voids. Collision of the two tensile waves in the material
occurs at $t_4$ and the resulting drop or “pullback” in the free surface velocity, denoted as $\Delta u_{fs}$, is used to measure the spall strength.

**Figure 4.20**: Simplified diagram of the spall fracture process and representative wave profile. (Left) Lagrangian position-time (X-t) diagram showing the wave propagation in the flyer plate and sample after impact. Blue lines represent elastic waves and black lines represent inelastic (plastic) waves. (Right) Representative plot of the sample’s free surface velocity. Features of the wave profile include the elastic precursor wave to calculate the HEL and the velocity pullback, $\Delta u_{fs}$, to calculate spall strength.

It is important to understand that spall strength is not an intrinsic material property and it changes depending on the loading conditions. The initial shock compression causes various microstructural defects that can alter the subsequent spall strength. For instance, spall strength is dependent on the Hugoniot stress (peak stress), the pulse duration, the unloading rate, and the initial microstructure. Nevertheless, spall strength is a continuum measurement so the relation of microstructure on spall strength helps relate microscale properties with macroscopic properties. Another important consideration is the fact that spall strength is measured under
uniaxial strain and directly comparing the spall strength values to strength results obtained under uniaxial stress conditions (e.g. testing on a load frame or Kolsky bar) is misleading and inaccurate. Additional discussions and references related to spall fracture can be found in [151].

Free surface velocity measurements, through VISAR or PDV, are analyzed and converted into stress using the Rankine-Hugoniot jump equations which relate material properties across a shock wave from the initial state (state 0) to the shocked state (state 1). A common representation of the jump equations in their simplified, 1D form is given in Eqs. (4.3) to (4.5):

\[
\frac{\rho_0}{\rho_1} = \left(1 - \frac{u_{p1}}{U_S}\right) 
\]

(4.3)

\[
\sigma_1 = \rho_0 U_S u_{p1} 
\]

(4.4)

\[
e_1 = \frac{1}{2} \sigma_1 (V_0 - V_1) 
\]

(4.5)

where \(\rho\) is the density, \(u_p\) is the particle velocity, \(U_S\) is the shock velocity, \(\sigma\) is the shock stress, \(e\) is the specific internal energy, and \(V\) is the specific volume. Derivations of the Rankine-Hugoniot jump equations from the conservation equations can be found in [152,153].

Additional material relationships are required to use Eqs. (4.3) to (4.5) since there are more unknowns than equations. One common relationship is the shock velocity versus particle velocity \((U_S-u_p)\) plane. A series of experiments are conducted measuring these quantities to quantify the empirical relationship between the two.
Except for cases where a material undergoes phase transformation, a linear relationship exists in the form:

\[ U_S = C_0 + s u_p \]  \hspace{1cm} (4.6)

where \( C_0 \) is the bulk sound speed and \( s \) is an experimentally determined constant. Values of \( C_0 = 4.460 \text{ km/s} \) and \( s = 1.720 \) for pure \( \alpha \)-iron are referenced from Thomas et al. [154]. Using Eqs. (4.4) and (4.6) the stress in the sample can be calculated as a function of the measured free surface velocity where:

\[ u_p = \frac{1}{2} u_{fs} \]  \hspace{1cm} (4.7)

where \( u_{fs} \) is the measured free surface velocity. The pullback particle velocity can be calculated from the measured free surface velocity using Eq. (4.7). Wave speed during unloading along the isentrope can be approximated using the bulk sound speed. Thus, the spall strength, \( \sigma_{sp} \) can be calculated as:

\[ \sigma_{sp} = \frac{1}{2} \rho_0 C_0 \Delta u_{fs} \]  \hspace{1cm} (4.8)

Similarly, because the elastic precursor wave travels at the longitudinal sound speed of the material, the HEL can be calculated as:

\[ \sigma_{HEL} = \frac{1}{2} \rho_0 C_L u_{fs} \]  \hspace{1cm} (4.9)

where \( C_L \) is the longitudinal sound speed of the target material.
Figure 4.21 shows the experimental setup of the α-iron plate impact shock experiments. Impedance matching 4340 steel alloy momentum trap was used to ensure that potential release waves generated from the interface between the sample holder and edge of the sample did not unload the material prior to spallation. Shock impedance (shown in Eq. (4.10) where \( \rho_0 \) is the initial density and \( U_S \) is the shock velocity) of the α-iron and 4340 steel alloy are closely matched providing wave transmission between the sample and momentum trap without significant reflections.

\[ \rho_0 U_S \quad (4.10) \]

\textbf{Figure 4.21}: Experimental setup for the plate shock impact experiments on α-iron. Flyer plate velocity, \( v_0 \) was measured using PDV and the flyer plate thickness was 3 mm.
α-iron samples at five different fatigue life ratios, 0, 0.31, 0.62, 0.83, and 0.94, were tested at three different impact velocities. Since the spall strength is dependent on the prior loading conditions—due to the passage of the initial compressive shock wave irreversibly changing the material state—it is diligent to test how the pre-existing fatigue defects affect the spall strength at several peak stress states. It was attempted to keep all experiments below 13 GPa at which point the α–ε phase transition occurs in iron [155,156]. Details of the tested α-iron samples and parameters of the plate impact shock experiments are given in Table 4.5.

Table 4.5: Parameters for the plate impact shock experiments on α-iron containing pre-existing fatigue defects. Fyler plate thickness for each experiment was 3 mm.

<table>
<thead>
<tr>
<th>Fatigue life ratio</th>
<th>Diameter (mm)</th>
<th>Thickness (mm)</th>
<th>Initial density (g/cm³)</th>
<th>Impact velocity (km/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>10.03</td>
<td>4.22</td>
<td>7.852</td>
<td>320</td>
</tr>
<tr>
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<td>4.360</td>
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<td>515</td>
</tr>
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<td></td>
<td>4.436</td>
<td>7.846</td>
<td>675</td>
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<tr>
<td>0.31</td>
<td>10.01</td>
<td>4.416</td>
<td>7.869</td>
<td>317</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4.422</td>
<td>7.872</td>
<td>515</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4.376</td>
<td>7.868</td>
<td>677</td>
</tr>
<tr>
<td>0.62</td>
<td>10.04</td>
<td>4.230</td>
<td>7.864</td>
<td>326</td>
</tr>
<tr>
<td></td>
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<td>4.406</td>
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<td>522</td>
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<td></td>
<td></td>
<td>4.544</td>
<td>7.866</td>
<td>681</td>
</tr>
<tr>
<td>0.83</td>
<td>10.01</td>
<td>4.196</td>
<td>7.877</td>
<td>329</td>
</tr>
<tr>
<td></td>
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<td>7.863</td>
<td>676</td>
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<td>0.94</td>
<td>10.01</td>
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<td>7.865</td>
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<td></td>
<td></td>
<td>3.454</td>
<td>7.859</td>
<td>514</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3.558</td>
<td>7.861</td>
<td>683</td>
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</table>

Profiles of the measured free surface velocity were analyzed using Eqs. (4.4), (4.6) and (4.7) to calculate the peak shock stress, Eq. (4.8) to calculate spall strength, and Eq. (4.9) to calculate the HEL. Wave profiles for all of the experiments are shown in Fig. 4.22 and contain the characteristic features discussed above; an elastic
precursor wave, the peak Hugoniot state, and a velocity pullback due to spall fracture. Some of the wave profiles do not exhibit the constant Hugoniot state possibly due to a slight release wave generate by the interface between the $\alpha$-iron sample and 4340 steel alloy momentum trap. Regardless, the peak free surface velocities measured in these experiments match closely with the measured impact velocity, which is expected for an impact between two impedance matched materials, indicating that the measured peak free surface velocity represents the Hugoniot state. Tabulated results from the wave profile analyses are given in Table 4.6.

![Figure 4.22](image.png)

**Figure 4.22:** Free surface particle velocity measurements for different amounts of fatigue loading on $\alpha$-iron samples. Plots have been offset in time for clarity.

Directly plotting the HEL and spall strength as a function of the fatigue life, shown in Fig. 4.23, reveals that the HEL decreased by 20% as a function of the fatigue life, whereas the spall strength remained constant. Since HEL can be thought of as a measure of the dynamic yield stress under uniaxial strain, the observed de-
Table 4.6: Calculated results from the plate impact shock experiments on α-iron at different fatigue life ratios.

<table>
<thead>
<tr>
<th>Fatigue life ratio</th>
<th>Impact velocity (km/s)</th>
<th>HEL (GPa)</th>
<th>Peak stress (GPa)</th>
<th>Spall strength (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>320</td>
<td>1.95</td>
<td>6.31</td>
<td>2.31</td>
</tr>
<tr>
<td></td>
<td>515</td>
<td>1.68</td>
<td>9.83</td>
<td>1.98</td>
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<td></td>
<td>675</td>
<td>1.93</td>
<td>13.22</td>
<td>1.62</td>
</tr>
<tr>
<td>0.31</td>
<td>317</td>
<td>1.78</td>
<td>5.85</td>
<td>2.15</td>
</tr>
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<td></td>
<td>515</td>
<td>1.62</td>
<td>9.48</td>
<td>1.72</td>
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<td></td>
<td>670</td>
<td>1.27</td>
<td>13.27</td>
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<td>522</td>
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<td>9.40</td>
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<td>329</td>
<td>1.45</td>
<td>6.15</td>
<td>2.07</td>
</tr>
<tr>
<td></td>
<td>514</td>
<td>1.67</td>
<td>9.59</td>
<td>1.53</td>
</tr>
<tr>
<td></td>
<td>676</td>
<td>1.41</td>
<td>13.37</td>
<td>2.06</td>
</tr>
<tr>
<td>0.94</td>
<td>312</td>
<td>1.55</td>
<td>6.06</td>
<td>2.30</td>
</tr>
<tr>
<td></td>
<td>514</td>
<td>1.27</td>
<td>9.60</td>
<td>1.74</td>
</tr>
<tr>
<td></td>
<td>682</td>
<td>1.48</td>
<td>13.52</td>
<td>2.04</td>
</tr>
</tbody>
</table>

Increasing trend matches the quasi-static behavior. In both loading situations a greater degree of fatigue-induced voids resulted in yield strength decreases. On the other hand, the spall strength remained constant throughout the fatigue life suggesting that the fatigue-induced voids did not have a substantial effect. It is plausible that the relatively small amount of void percentage (0.00057% at its maximum) were eliminated during the passage of the initial compressive shock wave accounting for the stable spall strength as a function of fatigue life. Additionally, alignment of the fatigue-induced voids was perpendicular to the shock wave and spall fracture plane since they formed groupings parallel to the fatigue loading direction (Fig. 4.21). Void alignment perpendicular to the spall fracture plane meant those voids would not coalesce with each other and might also have contributed to the constant spall strength throughout the entire fatigue life.
Examining the spall strength as a function of the peak stress, instead of fatigue life, shows a different trend. Figure 4.24 shows the spall strength results for each fatigue life ratio as a function of the peak Hugoniot stress. The as-received material experienced a steady decrease in spall strength from low to high peak stress. In other words, a stronger initial compressive shock resulted in a lower subsequent tensile stress required to create a spall fracture plane. However, any pre-fatigue loading of the $\alpha$-iron caused a different material response. While there was still an initial decrease in spall strength, it increased at the highest peak stress level. The higher spall strength could be explained if the material underwent the $\alpha \rightarrow \epsilon$ phase transformation. As listed in Table 4.6, the peak stresses for the highest impact velocity experiments were slightly above the 13 GPa peak stress threshold for the pressure-induced phase
transformation, helping account for the increase in spall strength after the initial decrease.

![Figure 4.24](image)

**Figure 4.24**: Spall strength as a function of peak Hugoniot stress at each fatigue life ratio. It is seen that only the as-received material experienced continual decreases in the spall strength, whereas the pre-fatigued material experienced an increase of spall strength at the highest peak stress.

On the other hand, a continual spall strength decrease in the as-received material might be due to the initial fine grained substructure, see Fig. 4.12. Spall fracture in ductile materials is driven by the nucleation, growth, and coalescence of voids [157,158]. Stress concentrations during deformation frequently occur at grain boundaries due to mismatches in crystal orientation and alignment. Local stress concentrations at grain boundaries mean they become preferential sites for defect nucleation. For example, see Fig. 4.11 where each of the observed fatigued-induced voids nucleated at a grain boundary. Therefore, it can be asserted that materials with smaller grain sizes will experience more void nucleation sites at the onset of tensile
loading— the leading mechanism for spall fracture— producing a lower spall strength. Experimental results have shown that the spall strength decreases as the grain size got smaller in aluminum [160] and tantalum [159]. It is possible that the initial substructure present in the as-received $\alpha$-iron was a factor in the continual decrease of spall strength. If so, this result implies that grain structure was more dominant during the spall response and consequently the spall strength (i.e. the tensile strength under high rate, uniaxial strain loading) than the pre-existing fatigue-induced defects.
CHAPTER 5

7075-T6 ALUMINUM ALLOY EXPERIMENTAL RESULTS

Results and analysis for the 7075-T6 aluminum alloy material are summarized in this chapter. This series of experiments specifically looked at the influence of reversible and non-reversible, macroscopic elastic fatigue on the microstructure and subsequent mechanical properties. Until this dissertation research there had not yet been a comprehensive study on the evolution of subsequent mechanical properties in 7075-T6 aluminum alloy after fatigue loading with varying mean stresses.

First, mechanical testing results including a method to determine fatigue life with tensile mean stresses and subsequent quasi-static and dynamic tension experiments are given. Strength, elongation, and strain hardening at both strain rates are shown and discussed. Second, microscopy characterization using SEM image analysis, microhardness, and EBSD will be shown and discussed. The amount and distribution of inclusion cracking and voids at different interrupted fatigue states was quantified. Finally, use of crystal plasticity modeling, implementation of several machine learning algorithms, and grain boundary slip transmission analysis were utilized to determine a deformation mechanism that is at least partially responsible for the mechanical property evolution.
5.1 Mechanical testing results

5.1.1 Fatigue testing with different mean stress

Fatigue tests on the 7075-T6 aluminum alloy were designed such that the maximum stress in the tensile loading direction was fixed at 70% of the yield stress. With a constant maximum stress in the elastic regime, one can assume the amount of dislocation motion in the tensile loading direction will remain consistent regardless of the stress ratio. Altering the stress ratios will then cause different amounts of dislocation reversibility during the reverse loading steps. Progression from a fully reversible fatigue cycle (\( R = -1 \)) to a fatigue cycle with no load in the opposite sense (\( R = 0 \)) will show how microscopic evolution changes from fully reversed dislocation motion to zero reversed dislocation motion. The discussion and research cited in Section 2.1 highlights that the assumption of dislocation motion in 7075-T6 aluminum alloy at 70% of the yield stress is justifiable.

Typical fatigue S-N curves use the stress amplitude of the loading cycle to predict fatigue life. However, this relationship can become unreliable in predicting the number of cycles to failure when loading is not fully reversed, i.e. \( R \neq -1 \). Therefore, a different approach to predict fatigue life is required. Methods to account for mean stress and strain effects have been used since the late 1800s. Gerber in 1874 [54] and Goodman in 1899 [54, 161] related the mean stress to a material’s tensile stress to account for a positive mean stress. Approaches which more accurately predict the fatigue life with a nonzero mean stress have been subsequently developed. These approaches use an effective stress measure, which considers multiple variables of the
loading cycle instead of only the stress amplitude. For instance, Walker [162] proposed an effective stress measure, $\bar{\sigma}$, of the form:

$$\bar{\sigma} = \sigma_{\text{max}}^{1-m} \Delta \sigma^m$$  \hspace{1cm} (5.1)

where $m$ is an experimentally determined exponent that fits the equation to fatigue test data and allows the prediction of fatigue life for nonzero mean stresses. The process of fitting fatigue life data to calculate $m$ is outlined by Dowling [163]. Equation (5.1) is a generalized case of the Smith Watson Topper (SWT) effective stress when fatigue loading is in the elastic regime and stresses are proportional to strains [164]. The SWT form of effective stress has $m = 0.5$. A modified SWT parameter was determined by Zhao and Jiang for 7075-T651 aluminum alloy to predict fatigue life under non-reversible cyclic loading [165]. However, since the 7075-T6 aluminum alloy used in this study had a different heat treatment and initial mechanical properties than [165], multiple fatigue to failure tests at different stress ratios were performed to calibrate a specific $m$ value to the particular material utilized for this work. A summary of the fatigue to failure tests is outlined in Table 5.1 and includes several strain controlled experiments, to the same maximum stress amplitude, to check for consistency between control modes. The cyclic frequency was 1, 2, or 3 Hz depending on the stress ratio. This method of fitting the fatigue life differed from the real-time monitoring of the strain evolution used with the $\alpha$-iron and described in Section 4.2. Strain evolution during testing of the 7075-T6 aluminum alloy did
not display changes until the end of the fatigue life ($N/N_f > 0.95$) necessitating the approach of using a best-fit line to Eq. (5.1).

**Table 5.1:** Summary of fatigue to failure experiments used to calibrate the Walker coefficient.

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Sample</th>
<th>$\sigma_{\text{max}}$ (MPa)</th>
<th>$\sigma_{\text{min}}$ (MPa)</th>
<th>Stress ratio ($\sigma_{\text{min}}/\sigma_{\text{max}}$)</th>
<th>$N_f$ (cycles)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fatigue to failure</td>
<td>01</td>
<td>387</td>
<td>-387</td>
<td>-1</td>
<td>6922</td>
</tr>
<tr>
<td></td>
<td>02*</td>
<td>385</td>
<td>-380</td>
<td>-1</td>
<td>8336</td>
</tr>
<tr>
<td></td>
<td>03</td>
<td>387</td>
<td>-387</td>
<td>-1</td>
<td>9732</td>
</tr>
<tr>
<td></td>
<td>04</td>
<td>344</td>
<td>-344</td>
<td>-1</td>
<td>22618</td>
</tr>
<tr>
<td></td>
<td>05</td>
<td>344</td>
<td>-344</td>
<td>-1</td>
<td>38846</td>
</tr>
<tr>
<td></td>
<td>06</td>
<td>301</td>
<td>-301</td>
<td>-1</td>
<td>82516</td>
</tr>
<tr>
<td></td>
<td>07</td>
<td>301</td>
<td>-301</td>
<td>-1</td>
<td>123565</td>
</tr>
<tr>
<td></td>
<td>08*</td>
<td>292</td>
<td>-309</td>
<td>-1</td>
<td>190070</td>
</tr>
<tr>
<td></td>
<td>09</td>
<td>301</td>
<td>-301</td>
<td>-1</td>
<td>198552</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>258</td>
<td>-258</td>
<td>-1</td>
<td>1159328</td>
</tr>
<tr>
<td></td>
<td>11*</td>
<td>372</td>
<td>-200</td>
<td>-0.5</td>
<td>51191</td>
</tr>
<tr>
<td></td>
<td>12*</td>
<td>372</td>
<td>-198</td>
<td>-0.5</td>
<td>59280</td>
</tr>
<tr>
<td></td>
<td>13</td>
<td>387</td>
<td>0</td>
<td>0</td>
<td>454818</td>
</tr>
<tr>
<td></td>
<td>14</td>
<td>387</td>
<td>0</td>
<td>0</td>
<td>634752</td>
</tr>
</tbody>
</table>

*Experiments performed under strain control.

Fitting the fatigue to failure experiments with Eq. (5.1) resulted in a value of $m = 0.516$ for the Walker coefficient. Figure 5.1 shows the fatigue to failure tests and calibrated best-fit line. Intervals for the interrupted fatigue tests at 25% and 75% were obtained using the fatigue life calculation provided by Eq. (5.1) and the calculated Walker coefficient. The number of cycles for each interrupted interval and fatigue loading condition is listed in Table 5.2. In addition, using data calibrated to the specific material in this dissertation research was preferred because there can
be scatter of up to two times in the fatigue life of 7075 aluminum alloy due to large impurities appearing near iron inclusions and the location of crack initiation [166].

Table 5.2: Summary of interrupted fatigue experiments. Three sub-tensile specimens are recovered from each test sample for subsequent tension testing.

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Sample</th>
<th>$\sigma_{\text{max}}$ (MPa)</th>
<th>$\sigma_{\text{min}}$ (MPa)</th>
<th>Stress ratio ($\sigma_{\text{min}}/\sigma_{\text{max}}$)</th>
<th>Cycles ($N/N_f$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Interrupted fatigue 01</td>
<td>387</td>
<td>-387</td>
<td>-387</td>
<td>1</td>
<td>1500 0.25</td>
</tr>
<tr>
<td>Interrupted fatigue 02</td>
<td>387</td>
<td>-387</td>
<td>-387</td>
<td>1</td>
<td>1500 0.25</td>
</tr>
<tr>
<td>Interrupted fatigue 03</td>
<td>387</td>
<td>-387</td>
<td>-387</td>
<td>1</td>
<td>4500 0.75</td>
</tr>
<tr>
<td>Interrupted fatigue 04</td>
<td>387</td>
<td>-387</td>
<td>-387</td>
<td>1</td>
<td>4500 0.75</td>
</tr>
<tr>
<td>Interrupted fatigue 05</td>
<td>387</td>
<td>-194</td>
<td>-194</td>
<td>-0.5</td>
<td>10000 0.25</td>
</tr>
<tr>
<td>Interrupted fatigue 06</td>
<td>387</td>
<td>-194</td>
<td>-194</td>
<td>-0.5</td>
<td>10000 0.25</td>
</tr>
<tr>
<td>Interrupted fatigue 07</td>
<td>387</td>
<td>-194</td>
<td>-194</td>
<td>-0.5</td>
<td>30000 0.75</td>
</tr>
<tr>
<td>Interrupted fatigue 08</td>
<td>387</td>
<td>-194</td>
<td>-194</td>
<td>-0.5</td>
<td>30000 0.75</td>
</tr>
<tr>
<td>Interrupted fatigue 09</td>
<td>387</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>150000 0.25</td>
</tr>
<tr>
<td>Interrupted fatigue 10</td>
<td>387</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>150000 0.25</td>
</tr>
<tr>
<td>Interrupted fatigue 11</td>
<td>387</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>450000 0.75</td>
</tr>
<tr>
<td>Interrupted fatigue 12</td>
<td>387</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>450000 0.75</td>
</tr>
</tbody>
</table>

Figure 5.1: Fatigue results at different stress ratios to determine the Walker coefficient, $m$, of 7075-T6 aluminum alloy. A value of $m = 0.516$ was calculated from the best fit linear line of the log-log plot. Effective stress is defined in Eq. (5.1).
5.1.2 Quasi-static tensile testing and subsequent mechanical property evolution

Measured quasi-static tensile properties of the 7075-T6 aluminum alloy are given in Table 5.3. Three sub-tensile specimens were recovered from each of the interrupted fatigue tests shown in Table 5.2 resulting in three post-fatigue tests, at each fatigue condition, under both quasi-static and dynamic strain rates. Parameters for the DIC instrumentation (for both the quasi-static and dynamic experiments) are detailed in Table 5.4. Quasi-static stress-strain curves for all of the fatigue conditions as a function of the fatigue mean stress are shown in Fig. 5.2a. All three tension experiments at identical fatigue conditions were combined into a single representative stress-strain curve using linear interpolation and extrapolation. Strain hardening behavior, shown in Fig. 5.2b, was obtained by converting the representative stress-strain curves at each fatigue condition to true stress-true strain; i.e. strain hardening was calculated after converting the stress-strain results in Fig. 5.2a to true stress-true strain.

Table 5.3: Mechanical properties of as-received 7075-T6 aluminum alloy.

<table>
<thead>
<tr>
<th>Young’s Modulus (GPa)</th>
<th>Yield Stress (MPa)</th>
<th>Ultimate Tensile Stress (MPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>71.4</td>
<td>547</td>
<td>603</td>
<td>14.6</td>
</tr>
</tbody>
</table>

Strength and elongation results for each quasi-static tension test is tabulated in Table 5.5 and shown in Figs. 5.3a and 5.3b. Yield stress was calculated using the 0.2% offset method, ultimate tensile stress (UTS) by finding the maximum engineering stress, and elongation by taking the average gauge section strain at fracture. Pre-
Table 5.4: DIC parameters for quasi-static and [dynamic] experiments.

<table>
<thead>
<tr>
<th>Device and Measurement Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Camera</td>
<td>Point Grey Grasshopper 3 [Shimadzu HPV-2]</td>
</tr>
<tr>
<td>Image Size</td>
<td>2448x2048 pixels [312x260 pixels]</td>
</tr>
<tr>
<td>Lens</td>
<td>Tamron 180 mm macro lens</td>
</tr>
<tr>
<td>Image Scale</td>
<td>91 pixels/mm [12 pixels/mm]</td>
</tr>
<tr>
<td>Images (Frame Rate)</td>
<td>400 (variable) [100 (500k fps)]</td>
</tr>
<tr>
<td>DIC Software</td>
<td>Correlated Solutions VIC-2D</td>
</tr>
<tr>
<td>Correlation Algorithm</td>
<td>Normalized Square Differences</td>
</tr>
<tr>
<td>Interpolation</td>
<td>Optimized 8-tap spline</td>
</tr>
<tr>
<td>Subset Size</td>
<td>49 pixels [13 pixels]</td>
</tr>
<tr>
<td>Step Size</td>
<td>15 pixels [3 pixels]</td>
</tr>
<tr>
<td>Measurement Points</td>
<td>1600 [700]</td>
</tr>
<tr>
<td>Strain Window</td>
<td>5</td>
</tr>
<tr>
<td>Smoothing Method</td>
<td>90% center weighted Gauss filter</td>
</tr>
<tr>
<td>Spatial Resolution</td>
<td>109 pixels [25 pixels]</td>
</tr>
<tr>
<td>Strain Resolution</td>
<td>180 µε [650 µε]</td>
</tr>
</tbody>
</table>

Fatigued material under quasi-static loading experienced an initial decrease in strength at only 25% of the fatigue life regardless of the type of fatigue loading. This drop in strength is similar to the decrease at the beginning of the fatigue life for the α-iron. After initial fatigue loading the only change in strength properties was due to fatigue loading at the highest mean stress of 194 MPa. There was an approximately 7% drop in strength, both yield and ultimate, from the as-received state to this condition.

The surface contours shown in Figs. 5.3c and 5.3d were calculated using cubic interpolation of the data. The best-fit surfaces in Fig. 5.3 clearly show the decrease
Figure 5.2: Quasi-static stress-strain behavior of 7075-T6 aluminum alloy after cyclic loading at different mean stress values to different fatigue life ratios. (a) Average stress-strain curves after fatigue at each condition. (b) Corresponding strain hardening behavior after fatigue.

in strength only at the fatigue condition of greatest mean stress near the end of the fatigue life. Both the yield stress and UTS showed the same decreasing trends. A 7% strength decrease was very similar to an approximately 6% decrease observed in 6061-T6 aluminum also fatigued with a tensile mean stress to 75% of the fatigue life [63]. As will be discussed in Section 5.2.2 the strength decrease was related to an increase of voids observed in SEM imaging. A connection between additional fatigue-induced voids and subsequent decreases in strength was also found in the α-iron material.

Elongation, shown in Fig. 5.3b, did not change significantly due to any prior fatigue loading with the exception of one specimen that failed well below the other experiments. Strain hardening was also consistent for all cases of prior fatigue loading, Fig. 5.2b. Initial hardening rates are typical of 7xxx series aluminum and are greater than pure aluminum due to precipitates formed during aging [167,168]. There
Table 5.5: Mechanical results of the post-fatigue quasi-static tension testing on 7075-T6 aluminum alloy. Experimental measurement uncertainty for the stress measurements averaged to approximately ±11 MPa and uncertainty in the elongation measurement was approximately ±0.1%.

<table>
<thead>
<tr>
<th>Fatigue mean stress (MPa)</th>
<th>Fatigue life ratio $N/N_f$</th>
<th>0.2%$\sigma_y$ (MPa)</th>
<th>UTS (MPa)</th>
<th>Elongation</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>527</td>
<td>583</td>
<td>16.8%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>528</td>
<td>583</td>
<td>17.6%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>526</td>
<td>586</td>
<td>18.2%</td>
</tr>
<tr>
<td>0</td>
<td>0.25</td>
<td>515</td>
<td>571</td>
<td>18.1%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>513</td>
<td>567</td>
<td>17.7%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>516</td>
<td>569</td>
<td>18.3%</td>
</tr>
<tr>
<td>0</td>
<td>0.75</td>
<td>508</td>
<td>567</td>
<td>17.7%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>510</td>
<td>565</td>
<td>16.7%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>510</td>
<td>565</td>
<td>17.1%</td>
</tr>
<tr>
<td>97</td>
<td>0.25</td>
<td>495</td>
<td>553</td>
<td>14.8%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>503</td>
<td>559</td>
<td>17.3%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>504</td>
<td>560</td>
<td>15.6%</td>
</tr>
<tr>
<td>97</td>
<td>0.75</td>
<td>513</td>
<td>569</td>
<td>15.9%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>515</td>
<td>569</td>
<td>17.7%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>513</td>
<td>567</td>
<td>17.6%</td>
</tr>
<tr>
<td>194</td>
<td>0.25</td>
<td>509</td>
<td>561</td>
<td>16.6%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>505</td>
<td>562</td>
<td>17.4%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>507</td>
<td>561</td>
<td>15.1%</td>
</tr>
<tr>
<td>194</td>
<td>0.75</td>
<td>487</td>
<td>550</td>
<td>12.7%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>485</td>
<td>547</td>
<td>15.6%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>487</td>
<td>549</td>
<td>16.2%</td>
</tr>
</tbody>
</table>

is a constant linear slope until a distinct change is observed after 40 MPa. A change in slope for precipitation hardened aluminum alloys is associated with the transition between shearing and bypassing for dislocation/precipitate interaction and generally occurs near the peak strength [169]. Ultimately the transition between dislocation shearing and dislocation bypassing near the UTS is not associated with pre-fatigue loading since it was consistent across all specimens. Lack of change in the strain
Figure 5.3: Quasi-static mechanical properties of 7075-T6 aluminum alloy after fatigue loading at different mean stress values to different fatigue life ratios. (a) Strength results as a function of fatigue life; the bottom markers show yield stress and the top markers show UTS. (b) Elongation results as a function of fatigue life. (c) Best fit surface for yield stress. (d) Best fit surface for UTS.

Hardening is evidence, along with the microhardness measurements in Section 5.2.1, that there were minimal changes to the dislocation arrangements throughout the fatigue life and that fatigue at different mean stresses does not create unique dislocation structures. Nonetheless, if different dislocation arrangements and structures are formed during fatigue loading they are not significant enough to affect subsequent properties on either the micro or macroscale.
Fracture surfaces for sub-tensile specimens failed under subsequent quasi-static loading were imaged in the SEM to get a qualitative understanding how different pre-fatigue loading changed the characteristics of failure. Specimens at three fatigue conditions were imaged: 1) as-received; 2) $N/N_f = 0.75$ at $\sigma_m = 0$ MPa; 3) $N/N_f = 0.75$ at $\sigma_m = 194$ MPa). The overall fracture surface and a zoomed in portion at each fatigue condition is shown in Fig. 5.4. Observations from the fracture surfaces revealed two differences in failure due to the pre-fatigue loading. First, were the relatively large areas of flat cleavage surfaces on both the as-received material and the $N/N_f = 0.75$ at $\sigma_m = 0$ MPa material. Second, was the extensive amount of observed microcracks in the $N/N_f = 0.75$ at $\sigma_m = 194$ MPa material, which were absent in the other two samples. This specific fatigue condition makes the buildup of strain energy more likely since there are no reverse loading cycles. Easier crack initiation during subsequent loading could be expected in areas of the material with higher amounts of initial strain energy due to higher dislocation irreversibility, helping explain why prevalent secondary microcracking was only present at this fatigue condition.

5.1.3 Dynamic tensile testing and subsequent mechanical property evolution

On the other hand, dynamic response of the 7075-T6 aluminum alloy did not display any change in strength due to prior fatigue loading. The stress-strain curves in Fig. 5.5a are representative curves of all three tension tests at that specific condition. Stress-strain curves in Fig. 5.5a have been smoothed for clarity using a quadratic regression filter with a window size of 0.3. Flow stress in the dynamic tests, listed
(a) As-received.

(b) \( N/N_f = 0.75 \) at \( \sigma_m = 0 \text{ MPa} \).

(c) \( N/N_f = 0.75 \) at \( \sigma_m = 194 \text{ MPa} \).

Figure 5.4: Scanning electron microscope images of quasi-static fracture surfaces for different pre-fatigue conditions in the 7075-T6 aluminum alloy. Images on the left show the entire fracture surface; images on the right show the zoomed in areas designated by the boxed region. Arrows in the images mark some of the cleavage surfaces which were more dominant in the as-received and fully reversed fatigued material, in addition to denoting a few of the visible microcracks which were only prevalent in the material that had prior fatigue loading with no reverse loading cycles.

and shown in Table 5.6 and Fig. 5.6a, respectively, represents the maximum stress achieved during the constant strain rate portion of loading. Similar to the quasi-static
Table 5.6: Mechanical results of the post-fatigue dynamic tension testing on 7075-T6 aluminum alloy. Stress uncertainties were approximately ±21 MPa and elongation uncertainties were approximately ±0.1%.

<table>
<thead>
<tr>
<th>Fatigue mean stress (MPa)</th>
<th>Fatigue life ratio ($N/N_f$)</th>
<th>Max flow stress (MPa)</th>
<th>Elongation</th>
</tr>
</thead>
<tbody>
<tr>
<td>588</td>
<td>0</td>
<td>574</td>
<td>563</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>21.3%</td>
</tr>
<tr>
<td>574</td>
<td>0.25</td>
<td>572</td>
<td>577</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>18.2%</td>
</tr>
<tr>
<td>563</td>
<td>0.75</td>
<td>579</td>
<td>569</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>20.7%</td>
</tr>
<tr>
<td>558</td>
<td>0.25</td>
<td>571</td>
<td>578</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>22.9%</td>
</tr>
<tr>
<td>574</td>
<td>0.75</td>
<td>574</td>
<td>574</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>18.9%</td>
</tr>
<tr>
<td>574</td>
<td>0.25</td>
<td>574</td>
<td>558</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>19.2%</td>
</tr>
<tr>
<td>561</td>
<td>0.75</td>
<td>557</td>
<td>579</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>19.3%</td>
</tr>
<tr>
<td>561</td>
<td></td>
<td></td>
<td>21.4%</td>
</tr>
</tbody>
</table>

results, elongation refers to the average gauge section strain at fracture. While the average amount of elongation remained consistent, there appeared to be more scatter and variation after the material was fatigued to 75% of its fatigue life.

Strain hardening during dynamic tension was similar regardless of the prior fatigue loading and most conditions experienced a slight plateau or increase in hardening after the linear negative slope. This non-linearity in the strain hardening rate is representative of overaged precipitation hardened aluminum alloys [169, 170]. It
Figure 5.5: Dynamic stress-strain behavior of 7075-T6 aluminum alloy after cyclic loading at different mean stress values to different fatigue life ratios. (a) Average stress-strain curves after fatigue at each condition. (b) Corresponding strain hardening behavior after fatigue, where $\sigma_0$ represents the stress once a constant strain rate was obtained.

Figure 5.6: Dynamic mechanical properties of 7075-T6 aluminum alloy after fatigue loading at different mean stress values to different fatigue life ratios. (a) Maximum flow stress as a function of fatigue life. (b) Elongation as a function of fatigue life.

is possible that the adiabatic heating due to dynamic deformation led to changes in precipitate size similar to what occurs during overaging. Hardening values dur-
ing dynamic loading are approximately an order of magnitude greater compared to quasi-static strain rates due to a larger dislocation velocity. The fundamental Orowan equation, shown in Eq. (5.2), relates the loading strain rate to dislocation mechanics; where \( b \) is the Burgers vector magnitude, \( \rho \) is the dislocation density in line length per unit volume, and \( v \) is the average dislocation velocity [171,172]:

\[
\dot{\varepsilon} = \frac{d\varepsilon}{dt} = b\rho v
\]  

(5.2)

Assuming a constant dislocation density, an increase in the strain rate requires an increase in dislocation velocity, which causes dislocations to pile-up at obstacles more rapidly resulting in higher measured strain hardening rates.

5.2 Microstructure characterization and microscopy analysis

5.2.1 Microhardness measurements

Microhardness measurements of individual grains, near grain boundaries, and adjacent to voids and inclusions were taken at three fatigue conditions to test for possible differences in dislocation accumulation due to the varying degree of dislocation reversibility: 1) as-received; 2) \( N/N_f = 0.75 \) at \( \sigma_m = 0 \) MPa; 3) \( N/N_f = 0.75 \) at \( \sigma_m = 194 \) MPa). Local microhardness measurements were taken using a Struers DuraScan Vickers indenter with a load of 5, 10, or 25 gf and a dwell time of 10 s. Results of the Vickers microhardness (HV) measurements are summarized in Table 5.7. Figure 5.7 shows a representative image for the measurement locations. An average of fifty mea-
Measurements were taken at each fatigue condition. Diagonals of the indentations ranged from 7µm to 15µm resulting in an approximate indentation depth of 1µm to 2µm.

**Table 5.7**: Microhardness (HV) values for the 7075-T6 aluminum alloy at specified locations and the three different fatigue conditions.

<table>
<thead>
<tr>
<th>Fatigue condition</th>
<th>Grain center</th>
<th>Grain boundary</th>
<th>Void/inclusion</th>
<th>Overall</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-received</td>
<td>200 ± 6</td>
<td>200 ± 3</td>
<td>210 ± 12</td>
<td>206 ± 11</td>
</tr>
<tr>
<td>$\sigma_m = 0$ MPa $N/N_f = 0.75$</td>
<td>192 ± 3</td>
<td>194 ± 5</td>
<td>200 ± 10</td>
<td>197 ± 9</td>
</tr>
<tr>
<td>$\sigma_m = 194$ MPa $N/N_f = 0.75$</td>
<td>201 ± 8</td>
<td>203 ± 6</td>
<td>207 ± 7</td>
<td>204 ± 8</td>
</tr>
</tbody>
</table>

**Figure 5.7**: Microhardness measurement locations in the 7075-T6 aluminum alloy material for (a) the interior of an individual grain, (b) near the grain boundary, and (c) adjacent to a void or inclusion.

Transitioning from a fully reversible fatigue cycle to a stress ratio of $R = 0$ results in dislocation motion that is not reversed during the fatigue cycle. Elimination of reverse loading potentially leads to greater dislocation accumulation at obstacles such as grain boundaries and inclusion particles. Therefore, an increase in hardness might be expected due to greater dislocation densities at these locations and fatigue condition, relative to the material in the middle of a grain undergoing fully reversed
loading. However, as the results in Table 5.7 summarize no significant change in the microhardness between measurement locations was observed and values were within the statistical uncertainty. Additionally, there was no significant change between the different fatigue conditions as both fully reversed fatigue and fatigue with a tensile mean stress matched the as-received material hardness.

A constant microhardness, regardless of the measurement location and fatigue condition, indicates that throughout the bulk material there are minimal changes to the dislocation arrangements during fatigue loading with different mean stresses. It is possible that the microhardness measurements did not have the precision to capture if there were changes in the dislocation arrangements because the size of potential dislocation features formed during fatigue, see Fig. 2.12, are on the order of the indentation diagonals. However, it is important to note that as discussed in Section 5.1.2 the quasi-static strain hardening also remained consistent regardless of the type and amount of prior fatigue loading. A consistent response in the strain hardening, coupled with the constant microhardness measurements, supports the notion there are not significant changes to the dislocation arrangements as a result of the fatigue loading.

5.2.2 Scanning electron microscopy image analysis

Subsurface, post-fatigued material was imaged on the SEM to quantify characteristics of iron inclusions and obtain the amount of void space. Three material states were investigated: 1) the as-received, pristine material; 2) material fatigued to $N/N_r = 0.75$ at $\sigma_m = 0$ MPa; 3) material fatigued to $N/N_r = 0.75$ at $\sigma_m = 194$ MPa.
Material for was taken approximately 3 mm below the surface as a representation of the bulk material and imaged parallel to the drawing/loading direction. Energy dispersive spectroscopy was performed on the inclusion particles that appear white in the images. A representative SEM image and the EDS spectra associated with the marked areas is shown in Fig. 5.8 and Table 5.8. Void space, seen as black in the SEM images, is also visible in addition to the inclusions.

Image processing of the SEM images was performed in MATLAB® R2019a to segment and obtain quantitative data from the SEM images. Binarization of the images was based on the location of the maximum histogram peak for each image which corresponds to the grayscale value of the aluminum matrix—the majority of the pixels. One histogram peak above the maximum was chosen as the thresholding value to obtain only the inclusion pixels, whereas one histogram peak below the maximum was chosen to obtain only the void space pixels. The image processing code to segment

<table>
<thead>
<tr>
<th>Site #1</th>
<th>Site #2</th>
<th>Site #3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al: 70.2%</td>
<td>Al: 76.4%</td>
<td>Al: 64.0%</td>
</tr>
<tr>
<td>Cu: 15.4%</td>
<td>Fe: 12.3%</td>
<td>Cu: 18.7%</td>
</tr>
<tr>
<td>Fe: 14.0%</td>
<td>Cu: 3.8%</td>
<td>Fe: 11.7%</td>
</tr>
<tr>
<td>Zn: 0.5%</td>
<td>Cr: 3.5%</td>
<td>C: 3.7%</td>
</tr>
<tr>
<td>Si: 1.5%</td>
<td>O: 1.8%</td>
<td></td>
</tr>
<tr>
<td>Zn: 1.0%</td>
<td>Ni: 0.9%</td>
<td></td>
</tr>
</tbody>
</table>

**Figure 5.8**: Representative SEM image of the as-received 7075-T6 aluminum alloy parallel to the drawing direction. Corresponding EDS spectra for each circled inclusion particle is shown in the Table 5.8.
out the inclusion particles and void spaces is given in Appendix B. Multiple images taken at pixel resolutions between 0.15\( \mu \)m to 0.50\( \mu \)m from different positions and depths from each sample were analyzed. Features less than 15 pixels were discarded and the resulting segmented properties analyzed.

Results of the microscopy image analysis from material recovered after two different fatigue conditions are shown in Table 5.9. Total void space was calculated by summing the void area of each analyzed image and dividing by the total scan area. The number of inclusion crack sites represents the number of inclusions that had void space intersecting it. Inclusion cracking was identified during image processing by checking for overlap between the bounding box of all identified inclusions and voids. A bounding box is the smallest box that contains all of the pixels for the segmented feature. Inclusion cracking was assumed to be responsible for the segmented void/inclusion bounding box intersections. The two fatigue loading conditions were chosen because that material was at the same fatigue life ratio \( (N/N_f = 0.75) \), different mean stresses \( (\sigma_m = 0 \text{ MPa vs. } \sigma_m = 194 \text{ MPa}) \), and had a post-fatigue strength difference (see Fig. 5.3). Therefore, these samples would help inform what effects a tensile mean stress had on the material microstructure and what contributed to the strength decrease.

**Table 5.9:** Image processing results of 7075-T6 aluminum alloy SEM images from different stages of fatigue loading.

<table>
<thead>
<tr>
<th></th>
<th>As-received</th>
<th>( \sigma_m = 0 \text{ MPa} ) ( N/N_f = 0.75 )</th>
<th>( \sigma_m = 194 \text{ MPa} ) ( N/N_f = 0.75 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total scan area</td>
<td>2.61 mm(^2)</td>
<td>1.58 mm(^2)</td>
<td>2.88 mm(^2)</td>
</tr>
<tr>
<td>Inclusion crack sites</td>
<td>7</td>
<td>26</td>
<td>39</td>
</tr>
<tr>
<td>Total void space</td>
<td>0.161 %</td>
<td>0.141 %</td>
<td>0.258 %</td>
</tr>
</tbody>
</table>
Distribution of the as-received inclusion and void area is shown in Fig. 5.9. A kernel density estimate (KDE), using Gaussian kernels and a bandwidth defined by [173], is also shown to better represent the distribution instead of a discrete histogram. Both inclusion and void sizes in the as-received material roughly followed log-normal distributions. Two additional comparisons are shown in Fig. 5.10 which highlight microstructural differences between the fatigued states. First, the area of all inclusions next to the area of only the cracked inclusions is shown in Fig. 5.10a. Second, changes in the distribution of void area due to fatigue loading is shown in Fig. 5.10b.

![Figure 5.9: Inclusion and void area distribution from the analysis of multiple SEM images of the as-received 7075-T6 aluminum alloy. Data is shown with a logarithmic x-axis.](image)

Fatigued material had a much higher occurrence of inclusion cracking meaning that many of the detected inclusion cracks were caused by the fatigue loading. An example image from material in the as-received condition and the pre-fatigued condition is shown in Fig. 5.11. All of the inclusion particles are colored green and
Figure 5.10: Distributions of the cracked inclusion area and void evolution in 7075-T6 aluminum alloy due to a tensile mean stress during fatigue loading. (a) The area of all iron-rich inclusions (left side y-axis) compared to cracked inclusions (right side y-axis). (b) Change in void areas at each fatigue condition. Both figures are shown with a logarithmic x-axis.

All of the void spaces are colored orange. Detected instances of overlap between an inclusion particle and void space are boxed. Each detected instance was checked in the raw SEM image to verify an inclusion crack. The example images in Fig. 5.11 show how the pre-fatigued material contained a higher number of detected inclusions that were cracked.

It was also observed that cracking occurred mainly in larger sized inclusions. Figure 5.10a shows the distribution of area for all of the inclusions compared to the area of only the cracked inclusions. There was an order of magnitude difference between the distribution peaks of the cracked inclusions versus the all of the inclusions. Furthermore, the frequency of cracking in inclusions larger than 100μm² was much greater than smaller inclusions. Approximately 30% of inclusions above
Figure 5.11: Representative SEM images depicting the larger frequency of inclusion cracking between (a) the as-received material and (b) the pre-fatigued material. Inclusions are colored green and voids are colored orange. The boxes show each detected instance of inclusion cracking.

100 $\mu$m$^2$ cracked during the fully reversibly fatigue loading and approximately 35% of inclusions above 100 $\mu$m$^2$ cracked when the fatigue loading had a tensile mean stress. Compared to only 2% of inclusions below 100 $\mu$m$^2$, cracking in larger inclusions was much more prevalent. The overall amount of slightly more than 2% of total cracked inclusions is in decent agreement with other experimental values from Bozek et al. of 1.2% and 7.3% for low cycle fatigue [174]. Figure 5.10b shows the total void space increased from the as-received material only when there was a tensile mean stress. The increase in void space occurred due to larger number of voids with an area of 10 $\mu$m$^2$. There was no evidence showing a significant increase of voids larger than the initial mean area suggesting that the increase in void space arose from additional fatigue-induced voids and not growth of existing ones.
Signals recorded by acoustic emission during tension testing of 7075-T6 aluminum alloy were shown to be due to cracking of larger inclusion particles 20µm to 60µm wide [175]. Therefore, it could be possible to delay fatigue failure by decreasing the size of inclusions throughout the matrix. A similar result was obtained when predicting the fatigue life of 7075-T651 aluminum alloy using a microstructure-based multistage fatigue model [176]. Crack initiation in a 20µm inclusion particle resulted in a shorter fatigue life compared to initiation in a smaller 4µm particle. High magnification images of cracked inclusions, shown in Fig. 5.12, give an example of some detected inclusion cracking due to the manufacturing process versus fatigue. Cracked particles due to fatigue loading, Figs. 5.12b and 5.12c, compared to one in the as-received stock shown in Fig. 5.12a, have additional matrix material visible in the crack indicating that these inclusions cracked and separated during the cyclic loading.

5.2.2.1 Inclusion cracking analysis using PCA

A more rigorous analysis of the inclusion cracking was performed utilizing one of the machine learning approaches mentioned in Section 3.4 to identify other inclusion properties, besides area, that lead to cracking. Several different shape metrics regarding the inclusions were obtained from the segmented images. This image processed data was used to determine what properties of the inclusion were important in deciding whether or not an inclusion would crack during fatigue loading. Principle component analysis (PCA) was utilized with all of the inclusion information to
Figure 5.12: Occurrences of inclusion cracking in as-received and previously fatigued 7075-T6 aluminum alloy. (a) As-received material. (b) Fully reversible fatigue loading interrupted at 75% of the fatigue life. (c) Fatigue loading with 194 MPa mean stress interrupted at 75% of the fatigue life.

detect the most important metrics in determining whether or not it will crack during loading. Inclusion metrics from the image processing were:

- **Area**: the number of pixels of the inclusion in the segmented image.

- **Circularity**: defined as $\frac{4\pi A}{P^2}$, where $A$ is the area and $P$ is the perimeter. Circularity is a measure of roundness and a value of one denotes a circle.

- **Eccentricity**: the ratio of distance between the foci and major axis length of a best-fit ellipse. Eccentricity is another measure of roundness where a value of zero denotes a circle and a value of one denotes a line.

- **Extent**: the area of the feature divided by the area of the bounding box, $\frac{A_{\text{feature}}}{A_{\text{box}}}$.
Table 5.10: Loadings of each variable for the first three principle components of the 7075-T6 aluminum alloy inclusion dataset.

<table>
<thead>
<tr>
<th>Variable</th>
<th>PC #1</th>
<th>PC #2</th>
<th>PC #3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area</td>
<td>-0.3930</td>
<td>0.3014</td>
<td>0.8120</td>
</tr>
<tr>
<td>Circularity</td>
<td>0.5768</td>
<td>-0.0691</td>
<td>0.1315</td>
</tr>
<tr>
<td>Eccentricity</td>
<td>-0.4828</td>
<td>-0.3241</td>
<td>-0.3857</td>
</tr>
<tr>
<td>Extent</td>
<td>0.4454</td>
<td>-0.4790</td>
<td>0.3085</td>
</tr>
<tr>
<td>Orientation</td>
<td>0.2851</td>
<td>0.7549</td>
<td>-0.2819</td>
</tr>
</tbody>
</table>

- Orientation: the angle between a best-fit ellipse’s major axis and the x-axis of the image. The x-axis of the SEM images is aligned with the fatigue loading direction in this case.

Data was normalized using the z-score prior to performing the PCA because of the different variable units, e.g. area (µm²) and orientation (degrees). A scree plot of the variance contained in each principle component (PC) is shown in Fig. 5.13 and indicates that the first three principle components contain 90% of the overall variance. Therefore, only the first three principle components are presented and discussed while the last two are not reported. Loadings for the first three principle components are shown in Table 5.10 where the most influential variables for each principle component are signified. The loadings give an indication what the principle component is primarily a measure of. For example, the loadings in the first principle component indicate circularity, eccentricity, and extent have the most contribution to that principle component. Therefore, principle component one can be thought of as largely a measure of the inclusions shape. Whereas principle component two and three are primarily a measure of orientation and area, respectively.
Figure 5.13: Scree plot showing what amount of variation in the original data each principle component contains. In higher dimensional datasets the inflection point of the scree plot is frequently used to determine how many principle components to analyze.

Visualization and interpretation of the inclusion results is presented using bi-plots of the first three principle components shown in Fig. 5.14 and Fig. 5.15. Data in each principle component is normalized to one (i.e. divided by the maximum value) so that the loading vectors and data are on the same scale for the plot. In Fig. 5.14 most of the cracked inclusions are located by moving right to left on the plot indicating that inclusion cracking is primarily a function of the inclusions shape (recall the loadings for PC #1 which is the x-axis). Moreover, the y-axis, which is dominated by the inclusion orientation, does not show a significant correlation to inclusion cracking. Therefore, the inclusion orientation relative to the loading direction is not a deciding factor in whether or not it will crack.
Figure 5.14: Biplot of principal component #1 and principal component #2 for the 7075-T6 aluminum alloy inclusion data. The vectors (drawn in blue) for each variable are the loadings given in Table 5.10. It was observed that orientation does not appear to be a strong indicator of inclusion cracking.

Figure 5.15a replaces principal component two, on y-axis, with principal component three—predominantly a measure of area. It is observed that a higher percentage of the data in quadrant II, where the area increases, results in more cracked inclusions. This is the same conclusion reached by using the area distributions in Fig. 5.10a. However, the biplot makes clear that area is not the only determining factor for inclusion cracking, the other influential factor being circularity. Examining
a zoomed in portion of the biplot, shown in Fig. 5.15b, quantitative information regarding specific test points were obtained. Ignoring the one outlying cracked inclusion point off to the right, coordinates for a cracked inclusion with the greatest x-value in PC #1 vs. PC #3 space are (0.0648, 0.0763). These principle component values can be transformed back into the original variable space using Eq. (3.7) yielding the values in Table 5.11. Since PC #1 is most correlated to circularity, this coordinate represents the largest circularity value for a cracked inclusion.

Table 5.11: Original variable values of the specific cracked inclusion denoted in Fig. 5.15b.

<table>
<thead>
<tr>
<th>Variable</th>
<th>Point (0.648, 0.0763)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area</td>
<td>71.9 µm²</td>
</tr>
<tr>
<td>Circularity</td>
<td>0.741</td>
</tr>
<tr>
<td>Eccentricity</td>
<td>0.604</td>
</tr>
<tr>
<td>Extent</td>
<td>0.645</td>
</tr>
<tr>
<td>Orientation</td>
<td>51°</td>
</tr>
</tbody>
</table>

It was observed from the biplots that area and circularity were the most important factors in determining whether or not an inclusion will crack during fatigue loading. In general, inclusions must have a circularity value lower than 0.741 in order to crack; round, circular inclusions tend not to crack. In addition, orientation of the inclusion relative to the loading direction does not appear to affect inclusion cracking. Understanding that only larger, less circular inclusions will crack can help in possibly altering the microstructure to extend the fatigue life since fatigue failure begins at these cracked inclusions. This observation and knowledge about inclusion cracking would not have been made without the use of principle component analysis. Consider Fig. 5.16 where the data in its raw form is plotted. It is difficult to make sense and
discern any trends when the data is presented in this way, underscoring the importance and contribution PCA can have in data analysis. In summary it was observed from the SEM image processing and principle component analysis that iron inclusion cracking occurs throughout the entire material in larger sized particles (>72µm²) with a circularity lower than 0.74 due to either of the fatigue loading conditions. Additionally, the development of approximately 10µm² voids also took place, but only during fatigue with no reverse loading.
Figure 5.16: The same 7075-T6 aluminum alloy inclusion dataset as Figs. 5.14 and 5.15, but plotted as a function of the original variable values. Similar quantitative observations regarding inclusion cracking are not as easily discernible highlighting the usefulness of PCA.

5.2.3 Electron backscatter diffraction and dislocation slip transmission analysis

This section explains how a combination of machine learning, informed by crystal plasticity modeling, and grain boundary analysis on EBSD scans were used to discover a microstructural mechanism responsible for additional void nucleation and subsequent strength decreases as a result of non-reversible fatigue. A shift from reversible to non-reversible fatigue changes not only the back-and-forth motion of dislocations, but also the interaction of dislocations with grain boundaries. Therefore, different measurements and analyses were performed which looked at different aspects of possible dislocation behavior. The following subsections contain a discussion of: calculating slip transmission parameters between two grains, Section 5.2.3.1; using machine learning with crystal plasticity modeling results to determine active
slip systems for any arbitrary grain orientation, Section 5.2.3.2; and finally how slip transmission analysis from EBSD scans on grains with a void were used to identify a mechanism behind fatigue-induced void nucleation, Section 5.2.3.5.

5.2.3.1 Dislocation slip transmission criterion

It was presumed that microstructural features of certain grains contributed to the increase in voids during non-reversible fatigue. Therefore, grains containing a void were analyzed with EBSD in order to better understand grain characteristics that might contribute to the additional voids observed in the SEM image analysis. Figure 5.17 shows a representative EBSD scan marked with individual regions containing voids that were analyzed. Dislocation slip transmission across a grain boundary was one property between two grains that was analyzed. The propensity of slip transmission across a grain boundary can be evaluated using the parameters of Lee, Robertson, and Birnbaum (LRB) [177,178]. Parameters in the LRB criterion consider both the driving force (represented by the Schmid factor) and geometric alignment (residual Burgers vector and geometric condition) of slip systems in two neighboring grains. The residual Burgers vector, $b_r$, was calculated using Eq. (5.3) where $b_1$ and $b_2$ are the slip directions in grains one and two, respectively.

$$|b_r| = |b_1 - b_2|$$ (5.3)

The geometric condition considers the angle, $\theta$, between the lines of intersection of each slip plane with the grain boundary. Figure 5.18 shows two grains from
an EBSD scan to demonstrate parameters in the LRB criterion. Each grain’s mean crystal orientation is denoted by the inverse pole figure (IPF) map and rotation differences between the grains are illustrated by the cubic crystal representation. The slip plane with the maximum Schmid factor for each grain is drawn and the dashed lines show the intersection with the grain boundary plane. The angle $\theta$ between the two lines of intersection is one of the parameters to assess slip transmission and was calculated using Eq. (5.4) where $v_1$ and $v_2$ represent the vectors for the lines of intersection.

$$
\cos \theta = \frac{v_1 \cdot v_2}{|v_1| \cdot |v_2|} \quad (5.4)
$$

Determining the actual grain boundary plane requires more than a simple two-dimensional EBSD scan. Serial-sectioning and reconstruction of multiple EBSD scans of a specimen is one possibility, but as pointed out by Lieberman et al. accurate grain boundary normals are still difficult to obtain even with three-dimensional EBSD data [179]. Therefore, grain boundaries in this dissertation research were assumed to be perfectly in-plane with the EBSD scan as shown in Fig. 5.18. The vectors $v_1$ and $v_2$ were calculated using Eq. (5.5) where $n_i$ and $n_{gb}$ are the normal vectors to the slip plane and grain boundary plane, respectively.

$$
v_i = n_i \times n_{gb} \quad (5.5)
$$

By definition, the Miller indices represent the direction of the plane’s normal vector $n_i$. Vector directions must be rotated using the grain’s Euler angles returned
from the EBSD scan to put all vectors in the same global coordinate system. Finally, the slip direction for the maximum Schmid factor is also drawn for each slip plane in Fig. 5.18. It can be seen that as the slip directions become perfectly aligned the residual Burgers vector calculation in Eq. (5.3) will become zero indicating a higher probability of slip transmission between those two grains and slip systems.

Figure 5.17: Grain orientation from an EBSD scan of the 7075-T6 aluminum alloy material fatigued to 75% of the fatigue life under fully reversible loading. Regions containing individual voids were analyzed to investigate the grain boundary and assess the relative likelihood of slip transmission. An inset of region A is shown on the right with the grain boundary highlighted.

Slip transmission across a grain boundary is more likely to occur when:

1. The residual Burgers vector ($b_r$) is minimized.

2. The angle ($\theta$) between the lines of intersection of the grain boundary and each slip plane is minimized.

3. The resolved shear stress (i.e. the Schmid factor) is maximized.
Figure 5.18: Schematic showing the parameters used to assess likelihood of slip transmission across a grain boundary. The slip plane in each grain with the maximum Schmid factor is shown and colored according to the scale bar. The grain boundary plane is also shown and is assumed to be perfectly in-plane with the EBSD scan. The vectors $b_1$ and $b_2$ represent the slip direction in each grain whereas the vectors $v_1$ and $v_2$ represent the line of intersection between the slip plane and grain boundary. The residual Burgers vector, $b_r$, and the angle $\theta$ are used to determine the possibility of slip transmission across the grain boundary for a set of slip systems.

5.2.3.2 Crystal plasticity modeling

Active slip systems in each of the neighboring grains needs to be identified in order to use the LRB slip transmission criteria. In total there are 144 combinations of slip transmission pathways between two face-centered cubic (FCC) grains to analyze, but this number was reduced using crystal plasticity modeling to quantify the amount of slip accumulation on each slip system in the $\{111\}$$\langle110\rangle$ family depending on the grain orientation. Specific grain orientations for modeling were intentionally selected.
to cover one entire stereographic triangle. Discretization of the Euler angles resulted in a total of 593 different orientations. An inverse pole figure (IPF) key showing each individual orientation that was modeled is shown in Fig. 5.19.

![Figure 5.19](image)

**Figure 5.19**: Grain orientations that were used in the crystal plasticity modeling to quantify slip accumulation on each slip system. Each dot on the IPF map represents a unique grain orientation that was modeled. In total 593 orientations were modeled.

Each grain orientation was simulated under uniaxial loading to 0.01 strain (compared to a strain level of 0.005 for the fatigue loading) and the amount of slip accumulation for each slip system was computed. The simulations use the crystal plasticity constitutive model described in [180], which accounts for elastic anisotropy and employs a thermally-activated slip kinetic equation with a dislocation density based hardening law. The model parameters were taken from published literature values for pure aluminum, but with an appropriately scaled athermal slip resistance.
to account for the increased yield strength of 7075-T6 aluminum alloy relative to the pure material [181]. This adjustment was reasonable given the quasi-static loading conditions and the small applied strains.

The Schmid factor (M) could also be calculated for each slip system based on the grain’s specific orientation. Therefore, the Schmid factor and amount of accumulated slip on each slip system, for each orientation was determined. The Schmid factor was also normalized to the maximum value of its particular grain. The ratio of a slip systems Schmid factor to its grain’s maximum Schmid factor is denoted as \(M/M_{\text{max}}\). These variables were then used with an unsupervised machine learning technique to cluster the data appropriately between active and non-active slip systems.

5.2.3.3 Slip activity determination with DBSCAN

One advantage to density-based spatial clustering of applications with noise (DBSCAN) is the ability to group data points with no prior knowledge about how many groups there should be. A user only needs to input values for \(\text{MinPts}\) and \(\epsilon\) and the algorithm automatically distinguishes points that are naturally clustered. The following process was followed to avoid subjective values of the input parameters:

- Normalize the data being used to a mean of zero and a standard deviation of one. This type of normalization is called the Z-score.

- Choose an appropriate distance metric to calculate the distances between each data point and all the others. Euclidean distance was chosen for this example.
• Select a value for MinPts and find the distance from every point to that specific neighbor. For example, if MinPts=5, find the distance to the 5th furthest neighbor from every point.

• Plot the ordered distance as a function of the number of data points. The plot will display how many data points have similar distances to their neighbors and the density of clustering. Points belonging to the same group will have comparable cluster densities and appear as a relatively horizontal line.

• Find when the plot begins to increase exponentially upward (i.e. find the “knee” in the curve). The point of upward inflection represents a measure to when data points become representative of outliers. If a point has an exceptionally large distance, relative to the other points, to the same number of neighbors than it is reasonable to assume that point as an outlier.

• The y-axis value at the “knee” of the plot represents the ϵ value to use.

Following these steps with the crystal plasticity slip accumulation data, produces the following plots shown in Fig. 5.20. The first plot on the left, Fig. 5.20a, shows the distance to a specified neighbor when plotting the accumulated slip value as a function of the normalized Schmid factor. There is a very defined clustering trend in the plot as seen by many of the data points having a similar distance value. In addition, there is a well defined increase which represents a distance when points begin to not be associated with a cluster. On the other hand, the plot on the right, Fig. 5.20b, shows the same distance information, but when plotted using the Schmid factor as a function of the normalized Schmid factor. In this situation there is only
a small set of data points that can reliably clustered together followed by a linear increase and finally an upward inflection point. Using this data representation of maximum Schmid factor vs. normalized Schmid factor leads to constantly changing density distribution in the data making clusters less defined. Therefore, the data was clustered using accumulated slip vs. normalized Schmid factor.

\[(a)\]  

\[(b)\]  

**Figure 5.20:** Distance from each data point to a specified neighbor for use in the DBSCAN algorithm. (a) Distance when the data is plotted as accumulated slip vs. normalized Schmid factor. (b) Distance when the data is plotted as Schmid factor vs. normalized Schmid factor. Representing data in the accumulated slip vs. normalized Schmid factor plane produces a more consistent cluster density.

The next part in the cluster analysis is how to choose the upward inflection point in a consistent fashion. For this dataset, the point where the total deviation of the sum of squared differences between the data points and a least-squares linear fit changed the most, was used. Values of $\epsilon$ determined through this method are marked on each plot in Fig. 5.20. Finally, the minimum number of points to use, $MinPts$, had to be determined. It was decided to use $MinPts = \sqrt{n}$ neighbors, where $n$ is the total...
number of data points in the dataset. However, the choice of $MinPts$ did not make a significant effect on the final clustering result. Figure 5.21 shows how the clustering between slip activity and no slip activity groups changed as a function of the selection for $MinPts$. It was found that regardless of the selection for $MinPts$ the total number of points being clustered into each group did not significantly change. The DBSCAN algorithm always grouped the same points together because there was a well defined cluster of no slip activity points where the “knee” of the curve adequately represented points outside that original cluster.

![Figure 5.21: Change in the DBSCAN cluster result as a function of the input parameter $MinPts$. It is shown that the effect of $MinPts$ is minimal as long as $\epsilon$ from the curve in Fig. 5.20a was selected in a consistent fashion. Robustness across a wide range of input parameters produced confidence in the final clustering result.](image)

The final grouping determination displayed with respect to different variables (accumulated slip vs. normalized Schmid factor and Schmid factor vs. normalized...
Schmid factor) is shown in Fig. 5.22. In Fig. 5.22a the large collection of points with zero slip (from approximately $M/M_{\text{max}} = 0$ to 0.6) can be seen as the horizontal portion in Fig. 5.20a with a similar cluster density. Points outside the no slip activity cluster will then be assigned by the algorithm to a different cluster or as noise points. However, in this specific case, prior knowledge about the domain (mainly that there are only two groups) lets all of the other clusters and noise points be assigned together in the slip activity group and allows the DBSCAN algorithm to work effectively. In cases where different groups have dramatically different clustering densities, DBSCAN might have difficulty clustering the lower density groups and may not be the best algorithm choice.

**Figure 5.22**: Graph showing the location of all the active and non-active slip systems as a function of two different parameter sets. (a) Accumulated slip vs. normalized Schmid factor was used for the DBSCAN clustering algorithm. (b) Schmid factor vs. normalized Schmid factor was used for the KNN classification discussed in Section 5.2.3.5.
5.2.3.4 Cross-validation of crystal plasticity modeling for slip activity classification

This section contains the results and scoring from the K-nearest neighbor (KNN) cross-validation. In addition, a special type of repeated k-fold cross-validation was used to determine how many crystal plasticity simulations would be required to obtain a consistently high success rate when using the nearest neighbor classification to predict if a slip system is active.

Crystal plasticity and molecular dynamic simulations provide valuable information that might be impossible to obtain experimentally. These results supplement experimental research leading to new insights and deeper understanding of materials behavior. However, the simulations can be computationally expensive to perform and require a lot of time to obtain results. Therefore, it is impractical to always require a large number of simulations covering all possible instances. Machine learning offers the ability to dramatically reduce the number of required simulations, yet still produce enough meaningful results to inform analysis decisions. An important goal is to therefore show that large datasets are not always needed to utilize some of these machine learning algorithms. This section outlines an analysis process and shows results that a limited number of simulations informing the KNN algorithm was still capable of producing accurate predictions. It might not be realized that even small datasets— with only a few observations—can still be effective in creating a predictive machine learning model.
A processes being referred to as inverse k-fold cross-validation was used to show the model’s performance when only a small portion of data was used to create the model, instead of the typical repeated k-fold cross-validation detailed in Section 3.4.3.1. Figure 5.23 shows how data is divided between the training and test datasets for inverse k-fold cross-validation. Only one fold is used to create the model for inverse k-fold cross-validation and then $k - 1$ folds are tested to compute the $F_1$ score. Compare this to k-fold cross-validation where $k - 1$ folds are used to create the model and only one fold is tested. Using the inverse k-fold cross-validation data allocation allowed an assessment of how well the algorithm could classify slip and no slip activity as a function of how many crystal plasticity simulations were used to inform the training dataset.

A flowchart of the inverse k-fold cross-validation process is sketched in Fig. 5.24. First, the crystal plasticity modeling results (shown in Fig. 5.22b) are randomly divided into k-folds. Next, a different classifier model is created with each single fold being used as the training dataset (á la Fig. 5.23). Then, the $F_1$ metric is calculated using K-neighbors with the remaining data folds being used as query points. These steps, beginning with the random data assignment, are repeated for a number of iterations. Finally, the $F_1$ score is averaged resulting in a performance score for a number of k-folds as a function of K-neighbors. In this analysis, ten iterations were run for $K = 1$ to 30 nearest neighbors, using $k = 2$ to 50 folds. Setting the k-fold range from 2 to 50 meant that the classifier model was created using only 50% to 2% of the data which is equivalent to running 297 to 12 crystal plasticity simulations, respectively.
Inverse k-fold cross-validation

![Diagram of data allocation for inverse k-fold cross-validation]

Figure 5.23: Depiction of data allocation for inverse k-fold cross-validation. Similar to Fig. 3.6, blue colored folds represent the new training dataset used to create the model and orange colored folds represent the withheld test points. Only a small portion of the original dataset is used to train the model with inverse k-fold cross-validation.

The overall $F_1$ score is shown in Fig. 5.25 and reveals the model was able to predict, with an $F_1$ score greater than 0.80, whether or not a slip system would be considered active for a wide range of the k-fold and K-neighbor parameters. Only when the number of k-folds were greater than 35 or the number of K-neighbors greater than 16 did the $F_1$ score drop below 0.80. Accuracy results were not calculated since there were more than six times the number of non-active data points than active data points. If a model was created that classified every single data point as “no slip
activity”, the accuracy would still be 86% based on the amount of data imbalance. Clearly a useless model, but with deceivingly good accuracy results.

Scoring results from creating a model based on a specific number of crystal plasticity simulations is shown in the bottom graph of Fig. 5.25, specifically for 12, 25, and 297 crystal plasticity simulations. For both 25 and 297 simulations the $F1$ score remains fairly constant and adequately predicts an active slip system regardless of the value for K-neighbors. It was also found that the predictive capability begins

Figure 5.24: Flowchart of the inverse k-fold cross-validation process. The output is an $F1$ calculation for each number of k-folds, as a function of K-neighbors, averaged over a number of iterations.
Figure 5.25: Performance of the KNN classification model based on the inverse k-fold cross-validation. (top) $F_1$ scores over the range of input parameters for k-folds and K-neighbors with a plane located at 0.80. (bottom) Comparison of $F_1$ scores using data from different number of crystal plasticity simulations.

to decrease rather significantly at approximately $K = 15$ neighbors in the extreme case of using only 12 simulations. There could be as few as only 12 data points for slip activity in this situation, one for each simulation. Therefore, the model will start
to have a higher bias and poor predicting capability as the number of K-neighbors nears the total number of data points in one of the classification groups. These results are what would be expected—namely that creating a model on too little data returns more incorrect classifications and does not have a reliable predictive capability.

It was determined that any value of K-neighbors between 5 to 15 results in the same consistently high $F_1$ score. Ultimately a value of $K = 11$ was chosen giving $F_1 = 0.87$ when using 25 crystal plasticity simulations. Compare this $F_1$ score to one from a model created by flipping a coin to get a sense of the score’s quality. In this scenario, it is assumed that a coin flip will correctly classify the data points exactly 50% of the time, leading to a baseline score of $F_{1\text{base}} = 0.21$. Then the actual $F_1$ score can be normalized with the baseline score using Eq. (5.6) to obtain how effective the model is compared to a random coin flip. The $F_{1\text{norm}}$ score is 0 for the baseline coin flip model and 1 for a perfect model.

$$F_{1\text{norm}} = \frac{F_1 - F_{1\text{base}}}{1 - F_{1\text{base}}}$$  (5.6)

A calculated score of $F_{1\text{norm}} = 0.84$ for the selected parameters of $K = 11$ and 25 simulations is gotten. This high $F_{1\text{norm}}$ score shows that a model created using a limited number of crystal plasticity simulations obtains classification results nearing that of an optimum model compared to a randomized coin flip model. These results led to a selection of $K = 11$ in the KNN classification analysis of the 7075-T6 aluminum alloys slip transmission study detailed in Section 5.2.3.5.
5.2.3.5 Slip transmission analysis with KNN

Data from the crystal plasticity modeling and clustering analysis described was subsequently used in the calculation of parameters to estimate the possibility of dislocation slip transmission between two grains. K-nearest neighbors (KNN) was used with the dataset in Fig. 5.22b to determine which slip systems in any arbitrary grain orientation determined through EBSD should be considered active.

Slip systems selected for the slip transmission analysis were determined by whether or not the specific system was deemed active based on the crystal plasticity modeling, DBSCAN clustering, and KNN parameters from cross-validation. Orientations of grains with voids and their neighboring grains were obtained through EBSD. Schmid factors for each of the grain’s slip systems could then be calculated from the orientation and loading direction information. Then, each slip system was plotted as a function of its Schmid factor versus the normalized Schmid factor (normalizing to the grain’s maximum Schmid factor). Finally, each slip system was classified as having slip activity or no slip activity through KNN and the training dataset shown in Fig. 5.22b. Classification predictions with KNN were done in the Schmid factor vs. normalized Schmid factor plane because only Schmid factors, and not accumulated slip values, could be calculated from the EBSD grain orientation. A value of $K = 11$ was chosen as the input parameter for the KNN algorithm based on $F1$ scoring during the cross-validation (see Section 5.2.3.4).

Grains with voids in as-received material state (which can be considered process-induced voids) were compared to grains with voids (which can be considered as a com-
bination of both process-induced and fatigue-induced) from material fatigued with a tensile mean stress of 0 MPa and 194 MPa to 75% of its fatigue life. Four voids were investigated for the as-received material, however, additional material from a different interrupted fatigue sample was also investigated to check for repeatability in the observation. Using two samples brought the total number of voids investigated to 7 and 9 for the reversible and non-reversible fatigued material, respectively. Residual Burgers vectors and $\theta$ were calculated for each combination of slip systems, that were classified as being active, between grains with a void and all their neighbors.

The resulting values of $b_r$ and $\theta$ were plotted against each other and the distribution density of the resulting slip transmission parameters for the three material conditions is shown in Fig. 5.26. Each contour color represents a higher density of data points in that specific contour region. The distributions for the as-received material and material fatigued to 75% of the fatigue life under fully reversible loading (Figs. 5.26a and 5.26b) display the greatest peak in the same region occurring at low $b_r$ and $\theta$ values. Having a large number of slip system combinations centered in a region with low slip transmission parameters means that slip is more likely to occur across the grain boundary. Furthermore, the similarity in location of the highest density peak between the as-received and fully reversible samples reveals that voids present in both material states are likely process induced and not a result of fatigue loading. This observation agrees with the SEM image analysis that found little difference in the total amount of void space between these two material states. However, an increase of approximately 40° in the angle $\theta$ when the material was fatigued with a tensile mean stress was observed from the distribution density plot.
in Fig. 5.26c. Grouping of the slip transmission parameters at an angle 40° larger would inhibit dislocation motion across the grain boundaries compared to the as-received and fully reversible material. The lack of relative dislocation transmission across grain boundaries is an explanation for why additional voids were observed and formed during the non-reversible fatigue loading. A noticeable, slightly lower peak in Fig. 5.26c, which matches the peaks from the other two material states, would be expected since SEM imaging concluded approximately half of the voids would be from initial processing and half from fatigue loading.

A shift in the distribution of slip transmission parameters helps clarify the reason behind the post-fatigue property degradation of 7075-T6 aluminum alloy. Namely, that the subsequent strength decrease was a result of additional void formation inside grains that were not conducive to slip transmission. Interpretation of the results show that the fatigue stress amplitude has more of an influence on crack propagation from cracks originating in inclusions on the surface than the bulk material, thus leading to shorter fatigue lives and no subsequent mechanical property changes. On the other hand, tensile mean stresses have a greater influence on accumulating fatigue defects throughout the bulk material, which manifests as additional voids and consequently a strength degradation in subsequent post-fatigue mechanical testing.

Overall, the microscopy analysis on the 7075-T6 aluminum alloy material shows internal iron inclusion cracking—of larger, less circular inclusions—due to macroscopically elastic fatigue loading at 70% of the yield stress. Cracking of the same iron-rich inclusion particles was also detected throughout the bulk of a 2024-T3 aluminum alloy [182] and µXCT results support the notion that surface observations of
Figure 5.26: Distribution density of the slip transmission parameters $b_r$ and $\theta$ for the 7075-T6 aluminum alloy material. (a) The as-received material, (b) material fatigued to 75% of the fatigue life under fully reversible loading, and (c) material fatigued to 75% of the fatigue life under a 194 MPa tensile mean stress. A shift of approximately 40° in the angle $\theta$ can be seen during the non-reversible fatigue loading.

Inclusion behavior during fatigue also occur in subsurface particles throughout the material [183]. Additionally, there is evidence that inclusion particle cracking takes place before yield during a single loading cycle (i.e. what would be experienced in a monotonic test) [184,185]. These observations also support the conclusion of inclusion cracking not contribute to any change in post-fatigue strength. Instead, an increase
of approximately 10µm² voids in grains with a higher likelihood to prevent slip transmission being responsible for a measured 7% decrease in quasi-static strength.
CHAPTER 6

CONCLUSION

Several broad conclusions were reached over the course of this dissertation research. Most important, is the fact that fatigue loading—even in the elastic regime—produces irreversible dislocation motion, microscopic defect accumulation, and subsequent mechanical property evolution. From an engineering design point of view it is necessary to consider the effect of potential property degradation as a result of in-service fatigue loading. Moreover, it was shown that microscopic defect accumulation and subsequent mechanical property evolution are dependent on a number of variables including the material, the amount of fatigue loading, the type of fatigue loading, and the subsequent loading strain rate. In both materials researched, the observed mechanism producing subsequent strength changes was primarily fatigue-induced void formation.

In the α-iron material, a relationship between the volumetric void evolution during elastic fatigue loading and subsequent mechanical properties at varying strain rates was established. It was shown that the decreases in strength were related to microstructural changes, first the elimination of a fine grained substructure and second the nucleation of voids which evolved and accumulated throughout the fatigue
loading. Each of the observed microstructural phenomena was linked to a macroscopic measurement to help explain the strain evolution or subsequent changes in the material’s strength.

In the 7075-T6 aluminum alloy material, microscopic defects generated during fatigue loading were quantified by analyzing microscopy images. These defects included development of fatigue-induced voids and cracking of inclusion particles throughout the bulk material. The accumulation of these defects and their effect on subsequent mechanical properties was dependent on the amount of reversibility in the fatigue loading. Mechanisms for both strength degradation and fatigue-induced void formation were determined. The rest of the chapter will highlight some of what was learned during the dissertation research.

6.1 Microscopic defect accumulation during fatigue

Starting from a microscopic viewpoint, the following conclusions were made regarding the defect accumulation during fatigue loading:

1. \(\alpha\)-iron:

   - Stages of observed microstructural defect evolution during fatigue included:
     1) the reduction of voids and elimination of a fine grained substructure induced during manufacturing; 2) the growth of existing voids; 3) the formation of voids along lines parallel to the fatigue loading direction; 4) and development of the void lines to longer lengths.
• Both the total void volume and average void size decreased upon initial fatigue loading, but increased during additional fatigue loading. There was a tendency of void growth over void formation in the bulk material. This conclusion was based on the results obtained between 31% and 73% of the fatigue life showing the average void size increased, to more than double the initial volume, despite the overall void ratio remaining relatively stable.

2. 7075-T6 aluminum alloy:

• Inclusion cracking of iron-rich particles throughout the entire material occurred due to any fatigue loading and primarily took place in larger, less circular inclusions. All instances of inclusion cracking were found to be perpendicular to the direction of fatigue loading.

• Approximately 30% of iron-rich inclusions above 100µm² cracked during fatigue loading compared to only 2% of inclusions with a smaller area. Moreover, a principle component analysis of the physical shape metrics revealed that inclusions with a circuitry value greater than 0.741 will not crack. Therefore, the alloy’s fatigue life might be extended by reducing the size and circular shape of iron inclusions by decreasing the overall number of inclusion cracks—notably those which occur on the surface.

• An increase in fatigue-induced voids throughout the bulk material developed during non-reversible fatigue loading. The fatigue-induced voids are thought to arise primarily in grains that prohibit dislocation slip transmission, along active slip systems, into neighboring grains.
• A machine learning classification technique, informed by crystal plasticity modeling, was effective in predicting slip activity for any arbitrary grain orientation from an EBSD scan. An effective classification of slip activity allowed the calculation of slip transmission parameters, for only active slip system combinations, creating an accurate distribution density of the parameters $b_r$ and $\theta$. Obtaining correct distribution densities were vital in identifying differences between process-induced and fatigue-induced voids.

• Comparison of the microhardness near grain boundaries versus the middle of grains showed that additional dislocation pile-ups due to non-reversed dislocation motion was unlikely. In addition, comparable microhardness values, regardless of the fatigue condition, implied the fatigue loading conditions studied had a minimal effect on dislocation arrangements.

6.2 Post-fatigue mechanical property evolution

Moving to a macroscopic viewpoint, the following conclusions can be made regarding the subsequent mechanical property evolution:

1. $\alpha$-iron:

• Residual macroscopic tensile strain during fatigue was linked to the large amount of void groupings, parallel to the fatigue loading direction. Increases in the amount of accumulated tensile strain appeared to be accommodated by the extension of void groupings to longer lengths.
• Initial decrease of the quasi-static yield stress and ultimate tensile stress between 0% and 31% of the fatigue life was due to the elimination of a substructure within grains which consisted of fine grains approximately 1\(\mu\)m\(^2\) to 5\(\mu\)m\(^2\) in area.

• Decreases in the quasi-static yield stress and ultimate tensile stress between 31% and 94% of the fatigue life were due to increases in the average void size, total percentage of voids, and grouping of voids along the loading axis. Steady decreases in the yield stress correspond to the continually changing void sizes, whereas a sharp decline in the ultimate tensile stress corresponds to the increase of void volume and grouping of voids along the same axis.

• The effect of fatigue-induced voids in the material’s subsequent mechanical response was found to be strain rate dependent. There was a decrease of strength during quasi-static loading, but an increase of strength during dynamic loading potentially caused by a higher amount of twinning.

• The Hugoniot elastic limit (HEL) (i.e. a measure of yield stress under uniaxial compressive strain) also decreased as a function of fatigue life. Conversely, the spall strength remained constant as a function of the fatigue life, perhaps due to the elimination of the relatively small volume fraction of fatigue-induced voids during the initial shock compression or alignment of the fatigue-induced voids parallel to the shock wave.

2. 7075-T6 aluminum alloy:
• Mechanical response after fatigue loading was dependent on the fatigue mean stress and subsequent loading strain rate. Fully reversible fatigue loading did not change the subsequent strength of the alloy, however when there was a tensile mean stress the strength decreased 7%. Although, this strength decrease was not present during high strain rate deformation again demonstrating the strain rate sensitivity of subsequent mechanical behavior. For design and engineering application purposes, a 7% reduction of quasi-static strength due to fatigue loading can be mitigated with appropriate safety factors.

• The mechanism responsible for strength degradation in the aluminum alloy material was fatigue-induced void formation, as the drop in quasi-static strength correlated to a higher percentage of observed void space from the microscopy image analysis. Fatigue-induced voids formed due to barriers restricting dislocation slip transmission across the grain boundary.

• There was no evidence that widespread iron-rich inclusion cracking changed the subsequent mechanical properties of the material, despite the prominent role of inclusion cracking in fatigue failure of 7075 aluminum alloys. Furthermore, there was no evidence that the fatigue-induced voids resulted in any other mechanical property changes besides a decline in quasi-static strength.
CHAPTER 7

FUTURE WORK

This chapter will briefly discuss several potential research directions based on the knowledge and experience gained during completion of the dissertation research.

7.1 Pure metals with different crystalline structures

The purpose of this follow up research would be to investigate the effects of the material’s crystalline structure on its propensity to accumulate fatigue defects. Inherent tension-compression asymmetry for body-centered cubic (BCC) materials was displayed in the strain evolution of $\alpha$-iron during fatigue loading (Fig. 4.15). Initial experimental results showed that pure aluminum, a face-centered cubic material (FCC), exhibited a symmetrical tension-compression response during fatigue loading. Pure aluminum also exhibited a strength increase of more than 30% after only several thousand loading cycles. The stark contrast in fatigue response, and subsequent strength evolution between the pure aluminum and iron, justifies more research into explaining the microscopic phenomenon (e.g. dislocation behavior) behind the discrepancies and how crystalline structure plays a role. Finally, additional experimental studies on hexagonal close-packed (HCP) material would allow compar-
ison to material systems that experience a greater degree of deformation twinning. Activating deformation twinning instead of dislocation slip during the fatigue loading could help explain how different deformation mechanisms contribute to defect accumulation. These sets of experiments would help the fundamental understanding of how different deformation mechanisms influence defect accumulation during fatigue.

7.2 4340 steel alloy experimental investigation

The purpose of this research study would be to perform a set of similar experiments as the α-iron, but with a steel alloy containing impurities and constituent particles that might be prone to damage, such as the iron-rich inclusions in 7075 aluminum alloy. The presented results of α-iron show that the additional fatigue-induced voids do not change the spall strength of the material, Section 4.2.4. A constant spall strength as a function of fatigue life is an indication that the pre-existing void defects were eliminated during the initial shock compression. It is plausible to presume the longitudinal stresses during the shock compression (> 5 GPa) effectively eliminated the microscopic voids that accumulated during the prior fatigue. On the other hand, defect accumulation during fatigue of an alloy material containing impurities is not limited to fatigue-induced void nucleation. Therefore, it is of interest to experimentally measure what effect other kinds of pre-existing defects have on a material’s spall strength.
7.3 Alternative fatigue loading profiles

The purpose of researching additional fatigue loading conditions would be to change the fatigue failure mechanisms, thereby altering the microscopic defect accumulation mechanisms and subsequent mechanical property evolution. All of the fatigue loading in this dissertation was performed at 70% of the yield stress to study the effects of macroscopic elastic fatigue loading while maintaining a reasonable number of cycles to failure for each material. However, failure mechanisms during fatigue loading, in a qualitative sense, transition from surface crack initiation during low cycle fatigue (LCF) to subsurface crack initiation in very high cycle fatigue (VHCF) [186,187]. Changing the origin of fatigue failure should also change both the types of microscopic defects and the amount of defects that have time to accumulate during the large number of cycles ($>1 \times 10^9$ cycles) of a VHCF experiment. One might assume that fatigue failure originating in subsurface material may contain a greater extent of microscopic defect accumulation than material that fails in fewer loading cycles due to surface crack initiation. In other words, if the fatigue failure mechanism changes, the microscopic defect accumulation and subsequent mechanical properties might also change. Research on 7075 aluminum alloy under combined high cycle and VHCF loading discusses failure from a crack not originating at an iron-rich inclusion [188], explicitly showing a different fatigue failure mechanism. A future area of research would then be to study how different fatigue conditions affect defect accumulation and subsequent mechanical property evolution.
APPENDICES
APPENDIX A

STATISTICAL ANALYSIS METHODOLOGY TO DETERMINE FEATURE RESOLUTION

This appendix contains the details regarding a statistical analysis approach developed as a supplementary part of this dissertation research.

A.1 Introduction

Standardization of data analysis methods of various image processing techniques is lacking despite the abundant use of these microscopy tools to characterize the microstructure of various materials. Two parameters that are typically reported for digital imaging include the scan resolution (e.g., pixel size, voxel size) and the spatial resolution (i.e., size of the smallest detectable object) [189]. However, neither scan nor spatial resolution give the experimenter an estimation of the minimum feature size for the experimental conditions. Furthermore, the experimenter does not get any indication of the features that could not be resolved. Therefore, the term feature resolution is used to define the effective minimum detection limit of any physical metric such as pore diameter, crack length, crack perimeter, and crack thickness. Deciding when the measurement of an object feature becomes unreliable is critical in
determining the resolution capability of an instrument under the given operating parameters. Resolution of detectable features is not only dependent on the instrument and hardware, but also the contrast between the features and surrounding regions of the imaged material and the segmentation processing algorithm. As detailed by Rose [190], less contrast will require larger objects to achieve the same image quality and resolution. Furthermore, Cohen et al. [191] showed the different resolution capabilities inherent for various image analysis algorithms. However, this work only considers data that has already been segmented and processed.

Methods that evaluate the feature resolution of processed image data can be grouped into three central categories: (1) assessment of the theoretical limitations of the instrumentation, (2) utilization of a calibration standard, and (3) the utilization of “phantom data” to calculate the feature resolution. For example, the simplest estimation of feature resolution is through the use of the Nyquist sampling criterion which requires a minimum of two pixels in the radius of the Airy disk [192]. The Airy disk is the spot size of the focused beam from the optic lenses of the instrumentation. This suggested requirement indicates that features must be resolved with at least four pixels for 2D data and eight voxels for 3D data, as used in Pavan et al. [193]. However, a more appropriate spatial resolution can be determined through more advanced physical considerations. For example, the modulation transfer function (MTF) is experimentally determined to calculate the spatial resolution by taking into consideration imaging hardware specifications such as detector width, focal spot size, and field of view in addition to analysis methods such as handling of the line-spread function (LSF) and reconstruction kernel for tomographic instru-
ments [194,195]. However, a major drawback is that the MTF is dependent on the instrumentation imaging parameters [196], such that a new MTF may be required for each chosen parameter set.

Additionally, a calibration standard with known features can be used to determine the effective spatial resolution capability of an imaging technique. Various calibration processes and materials have been successfully demonstrated using glass microspheres [197] and predrilled micro-holes [198]. However, the effective spatial resolution is still dependent upon the imaging parameters and material type, and may require re-calibration for each set of imaging parameters. Furthermore, an individual calibration standard does not inform the actual resolving limit of the imaging technique. A set of progressively smaller calibration standards are required to be evaluated until features can no longer be accurately resolved to determine the effective resolution limit.

Finally, the use of “phantom data” can be utilized to estimate the feature resolution by simulating a grid to represent the imaging pixel/voxel structure to characterize an object of interest. Simulated objects of prescribed geometry and dimension, or “phantom data”, are characterized by the grid to examine the accuracy of the measured features corresponding to a specific scan resolution. For example, Patterson et al. [199] utilized a set of simulated cylindrical objects of various dimensions that were modeled in cubic grids corresponding to 25–900 voxels per side. Various phantom data have also been used to estimate the feature resolution of different grain characteristics representative of material microstructure (e.g., grain volume, surface area, etc.) [200,201]. However, a disadvantage with the utilization of “phantom data”
is the difficulty in representing real microstructural features such as complex crack networks and shape features with simulated structures. Additionally, this method disregards the effect of various interactions concerned with the imaging process such as scatter noise, streaking artifacts, and large local attenuation differences. Instead, it relies on a consistent and well-defined spatial resolution throughout the imaged material of interest.

The objective of this statistical analysis approach was to establish a method for determining the minimum observable resolution of any arbitrary physical feature from a previously acquired dataset by utilizing an assumed distribution. Prior knowledge of the distribution was required to simplify the process of fitting a distribution curve. Using large, well-defined features in the dataset will result in an accurate match to the assumed distribution curve. Deviation from the assumed distribution curve will occur when smaller, poorly-defined features approaching and below the feature resolution are also included. Therefore, the percent error between the assumed distribution curve and the dataset can be calculated as a function of how much data is included. The feature resolution is then determined once an allowable amount of deviation from the assumed distribution curve is reached. This process can be applied for any metric of interest, with any type of assumed distribution. Analysts can use the method, and resulting feature resolution, as a guide to help inform instrument setup among typical constraints. Several physically relevant metrics obtained from scanning electron microscope images of pressed 1,3,5,7-tetranitro-1,3,5,7-tetrazoctane (HMX) powder and μXCT images of a polymer bonded explosive material were examined using the developed methodology.
A.2 Methodology

Two datasets following a log-normal data distribution were generated, using random variates, to represent arbitrary physically relevant metrics of interest. In the capture of real data, features will contain a whole number of pixels or voxels. A single pixel or voxel represents the scan resolution. Therefore, all generated samples were rounded to integer values to simulate the effect of scan resolution present in real data.

A simulated scan resolution, of $r = 1$, was chosen such that the datasets correspond to a mean near the theoretical scan resolution $\mu \approx r$ for the lower mean valued dataset, and a mean much greater than the theoretical scan resolution ($\mu \gg r$) for the greater mean valued dataset. Specifically, the datasets were comprised of 100,000 samples with mean values of 16 and 3600. All samples that exist below the chosen scan resolution were removed to yield two truncated datasets. These datasets represented as histograms are presented in Fig. A.1. As shown in Fig. A.1, the effect of integer rounding is more prevalent for data near the scan resolution, as is present in the dataset with the lower mean valued data ($\mu \approx r$). Even though the original dataset is log-normal distributed, there are significant peaks with gaps on the lower side of the data.

The probability density function (PDF) describing a log-normal distribution is defined by two shape parameters: the mean ($\mu$) and variance ($\sigma^2$). These shape parameters can be estimated using only a subset of the relevant domain of the distribution, i.e., a truncated distribution. For example, the shape parameters describing
Figure A.1: Two log-normal distributed datasets that were rounded to the nearest integer and truncated below a selected scan resolution value of $r = 1$. The effect of scan resolution limit and pixelation rounding effect on the distribution of data is clearly demonstrated in the data corresponding to a mean value near the scan resolution limit ($\mu \approx r$). The $\mu \gg r$ and $\mu \approx r$ data have mean and standard deviation values of $\mu = 16$ and $\mu = 3600$, respectively.

The distribution of a normal dataset truncated to remove the lower portion can be calculated using Eqs. (A.1) and (A.2), [202, 203].

\begin{align*}
\mu &= \mu_0 + \frac{\sigma_0 \phi(\alpha)}{Z} \quad \text{(A.1)} \\
\sigma^2 &= \sigma_0^2 \left[ 1 + \frac{\alpha \phi(\alpha)}{Z} - \left( \frac{\phi(\alpha)}{Z} \right)^2 \right] \quad \text{(A.2)}
\end{align*}

where

\begin{align*}
Z &= 1 - \phi(\alpha) \quad \text{(A.3)} \\
\alpha &= \frac{(a - \mu_0)}{\sigma} \quad \text{(A.4)}
\end{align*}
$$\phi(x) = \frac{1}{\sqrt{2\pi}} \left( \exp \left( -\frac{1}{2} x^2 \right) \right) \quad (A.5)$$

$$\Phi(x) = \frac{1}{2} \left( 1 + \text{erf} \left( \frac{x}{\sqrt{2}} \right) \right) \quad (A.6)$$

$\mu_0$ is the calculated mean, $\sigma_0$ is the calculated standard deviation, and $a$ is the lower limit of the truncated data.

To investigate the effect of scan resolution on the accuracy of a distribution of measured data, the data was treated as a lower truncated dataset with the scan resolution value, $r$, equal to the lower truncation limit, $a$. The value of the lower truncation limit, or cutoff value, was varied from the minimum value of the data to a value that excludes 99% of the data. Due to the implicit form of the system of Eqs. (A.1) and (A.2), a minimization routine was used to determine the shape parameters of the truncated distribution that best describes the observed truncated data. The routine was used to minimize the differences between the left and right hand sides of Eqs. (A.1) and (A.2) by iterating upon the shape parameters $\mu$ and $\sigma$. In this work, the Nelder-Mead simplex method for function minimization [204] was used via the SciPy Optimize package [205] with the initial guess for each parameter being the calculated mean, $\mu_0$, and the calculated standard deviation, $\sigma_0$, of the dataset. Examples of two selected cutoff values and the resulting best-fit PDF are presented in Fig. A.2. As illustrated in Fig. A.2b, including only a small portion of the upper data will result in a poor fit due to the inability to represent the distribution using a few percent of the total data. Therefore, enough of the data must be included to accurately reproduce the data distribution. However, data at the lower
end of the distribution near the scan resolution can be inaccurate due to the integer rounding discussed above. This scenario presents a case of two competing effects for determining the appropriate cutoff value to represent the original data: enough data must be included to effectively represent the distribution of the data while excluding inaccurate data at the lower end of the distribution.

Figure A.2: Best-fit PDF distributions using the calculated shape parameters from the truncated dataset (blue) and the original data (gray) represented via histogram. (a) Data below the cutoff value is still well represented by only using data above the cutoff value to estimate the PDF. (b) Truncating too much data results in a poor estimated fit to the overall data.

Kernel density estimates (KDE) [206] were used to represent the distribution of the full dataset in a smooth continuously defined function. In this methodology, Gaussian basis functions were used as the kernel and the bandwidth, \( h \), was calculated by:

\[
h = \left( \frac{4\sigma^5}{3n} \right)^{1/5}
\]  

(A.7)

following [207], where \( \sigma \) is the standard deviation of the dataset and \( n \) is the total number of data points. A comparison of the histogram and KDE representation of the two idealized datasets are given in Fig. A.3a.
The PDF and KDE were determined for the full range of cutoff values using the shape parameters calculated from the truncated dataset corresponding to the cutoff value. The percent error difference between the PDF and KDE was evaluated at the value of the specified cutoff value as demonstrated in Fig. A.3b. The percent difference between the PDF and KDE was then calculated, using Eq. (A.8), at each selected cutoff value for the entire dataset. The resulting percent error differences between the PDF and KDE as a function of the cutoff value are shown in Fig. A.3c. The percent error plots for the datasets are constructed by calculating the percent error between the PDF and KDE from point-to-point at each threshold value. The observed trend in the percent error demonstrates that when data near the scan resolution is included in the analysis, there is an increase in error between the PDF and the KDE. This result illustrates that data near the scan resolution contains a greater degree of uncertainty relative to data much greater than the scan resolution. This error is due to the integer rounding of the values that represents pixelation error. As the error increases with the decreasing cutoff value, it is evident that the represented data no longer follows the log-distribution and can be assumed to be below the effective feature resolution limit. The feature resolution limit is therefore defined where the percent error difference between the PDF and KDE reached a specified error, e.g., 10%. This error limit is a user-defined value dependent upon the tolerable amount of error for the specific metric of interest. Caution must also be used at the extreme upper end of the dataset. Using only a small portion of the upper data will result in a poor distribution fit as detailed in Fig. A.2b, and an increase in percent error as shown on the right sides of Fig. A.3c. Additionally, the KDE value could reach zero
resulting in an error of unity from Eq. (A.8). Therefore, a minimum amount of data, in this case data above the mean plus one standard deviation, must be included to alleviate these issues.

\[
\% \text{ error} = \left| \frac{(KDE - PDF)}{PDF} \right| \times 100 \tag{A.8}
\]

**Figure A.3**: Resolution determination of the \( \mu \approx r \) (top row) and \( \mu \gg r \) (bottom row) log-normal datasets. (a) Representation of log-normal distributed data using kernel density estimates (smooth continuous function) and a histogram (discrete bin points). (b) Percent error between the best-fit PDF and KDE at a specific cutoff value. Data above the cutoff value is used to fit the assumed data distribution. (c) Percent error between the best-fit PDF and KDE over all cutoff values. As the percent error plots are constructed from the point-to-point difference between the PDF and KDE at each cutoff value, there exists only one error plot for each dataset.

**Figure A.4** summarizes the analysis methodology and the individual steps used to determine the feature resolution of a dataset for a specified error value. This method allows any data obtained from image processing to be analyzed. However, if an incorrect distribution type is assumed or the distribution of the features cannot
be well described by the assumed distribution type, the method would no longer be valid. Specific scenarios with significant deviation from distributions at the tails may be accounted for through the use of multi-modal or other non-Gaussian distributions. Furthermore, if there are not a sufficient number of points (either through too small a dataset or by truncating too much data) to represent the assumed distribution accurately, the method would also no longer be valid. Regardless of the type of distribution, divergence between the best-fit PDF from the data represented as a KDE indicates that data with higher amounts of uncertainty is present for that value. Therefore, this method is useful in selecting the practical feature resolution of an analyzed dataset. In addition, this method allows for the inference of information below the feature resolution.

The feature resolution for different specified error values of 1%, 5%, and 20% are illustrated in Figs. 5a, 5b, and 5c, respectively. Only data above the feature resolution limit is determined to be accurately resolved, within the specified amount of error. By tolerating a higher error value, more of the data can be included from the lower end of the distribution. Once again the effect of pixelation error due to the scan resolution limit can be observed in Fig. A.5b. As expected, the amount of usable data in the $\mu \approx r$ dataset (samples near the scan resolution) is much less than the $\mu \gg r$ dataset (samples much greater than the scan resolution). Furthermore, by utilizing the assumption that the observed data follows a known distribution, information of the data below the resolution limit can be inferred. This allows for an estimate of the number of samples that exist below the feature resolution limit and the scan resolution, $r$.
**Figure A.4**: Flowchart of the process to determine the feature resolution limit of a dataset. The best-fit parameters are determined of an assumed PDF fit to the dataset, determined for each truncation cutoff value. Percent error between the best-fit PDF and KDE representation of the data distribution is then determined as a function of the truncation cutoff value. The feature resolution is then obtained at the cutoff value when a user defined percent error threshold is exceeded.

Additional datasets were analyzed to investigate the effects of the distance between the scan resolution and the mean of the distribution of the data. For these investigations, a sufficient number of points (100,000) were used in each dataset to avoid under sampling. It was found that in datasets with a mean less than two times the scan resolution, i.e. datasets with a significant number of samples affected by large pixelation error, there was an average error of 4% and 5% of the mean and standard deviation estimation, respectively. If the mean of the dataset was above
Figure A.5: Feature resolution determined for $\mu \approx r$ (top row) and $\mu \gg r$ (bottom row) log-normal datasets at different error thresholds of: (a) 1\%, (b) 5\%, and (c) 20\%. As the amount of allowable error increases, from 1\% to 20\%, the amount of usable data from the measurement technique also increases. Less than half of the total density can be characterized within 1\% uncertainty, whereas nearly all of the data can be characterized within 20\% uncertainty. The increases in the percent error at the lower ends of the datasets imply that there is higher uncertainty of those samples due to the pixelation error.

this value, the error of both the mean and standard deviation estimates decreased to less than 1\%.

A.3 Results and Discussion

The developed method of identifying the feature resolution limit was applied to data obtained from scanning electron microscope (SEM) and micro X-ray computed tomography ($\mu$XCT) imaging methods. For each of these imaging methods, the acquired images were segmented based on the grayscale value and segmented into binary images constituting of the bulk material and the material of interest, either
voids or particles, within the samples. Specifically, a bi-modal Gaussian curve was
fit to the grayscale values of the image histogram and a fixed number of standard
deviations below or above the mean value of the upper mode was used to determine
the threshold value. The binary images were then filtered using a cross-shaped 3x3
kernel as the structuring element for a sequential dilation and erosion filter operation.
Individual objects were identified from the filtered images as four-node connected re-
gions. Figure A.6 shows examples of the acquired and binarized images for both
materials and characterization techniques.

Figure A.6: Representative images of each sample obtained via SEM and μXCT
imaging techniques. (a) SEM Image of FEM HMX powder pressed to a nominal
TMD of 75%: (top) unprocessed image and (bottom) segmented image showing
the internal void space. (b) SEM image of a pressed Class V HMX pellet: (top)
unprocessed image and (bottom) segmented image showing the internal void space.
(c) A 2D image slice from the 3D reconstructed μXCT data of a polymer bonded
explosive material: (top) unprocessed image and (bottom) segmented image showing
the constituent of interest.
A.3.1 Scanning electron microscope data

Two samples were used to investigate the internal void structure of pressed pellets via SEM. Sample A consists of fluid energy milled (FEM) HMX powder pressed to a nominal TMD of 75\% and sample B consists of a Class V HMX powder pressed to a nominal theoretical maximum density (TMD) of 90\%. Micrographs were obtained from sectioned and polished surfaces of each sample via a Hitachi NX9000 focused ion beam-scanning electron microscope. A total of 55 images were collected for each sample at a resolution of 10 nm per pixel and individual internal void structures were identified and characterized. Metrics of the identified voids were calculated to include perimeter, area, circularity, solidity, and the minimum area bounding box. Circularty was defined as the ratio of the perimeter of the object to the perimeter of an area equivalent circle. The minimum area bounding box was defined as the minimum area rectangle that fully encompasses the object and was allowed to rotate. Solidity was defined as the ratio of the measured object area to the area of the minimum area bounding box. Crack networks or “cracks” were selected from the group of void objects which exhibit a greater spatial complexity that follow a branching structure. In this work, cracks were defined as void objects where the circularity is less than 0.65, solidity was less than or equal to 0.30, or the aspect ratio of the minimum area bounding box was greater than or equal to 7.

A total of 84,244 cracks were identified in the collection of SEM data acquired from sample A and the distribution of the calculated areas of the cracks is shown in Fig. A.7a. The distribution of the areas of the cracks corresponds well to a log-
normal distribution, and this distribution type was assumed for these data in order to enable the estimate of the feature resolution limit. Following the proposed method, an acceptable error limit of 10% was chosen such that the feature resolution limit was estimated to be 30 pixels. For cracks that are defined with at least 30 pixels, the total number of cracks, corresponding to that area, can be estimated with an error less than 10%. Additionally, the number of cracks with an area smaller than the feature resolution limit can be inferred by the best-fit PDF. In this example, less than 5% of the total estimated number of cracks had an area below the feature resolution limit. Furthermore, the corresponding data of objects below the scan resolution can also be inferred, giving an indication of the data unable to be captured due to the resolution capability of the instrument.

A total of 47,881 cracks were identified in the collection of SEM data acquired from sample B and the distribution of a different metric of interest, the measure of the mean crack thickness of the individual crack networks, is given in Fig. A.7b. These data were also found to follow a log-normal distribution. The absence of data below the mean crack thickness of one pixel signifies the limit of the scan resolution and no data was measured below this value. However, information about the features below the scan resolution can be inferred by the best-fit distribution curve. The overall feature resolution limit of the mean crack thickness was estimated to be 1.3 pixels with an acceptable error limit of 15%. These estimated feature resolution limits provide a quantitative description of the limitation of the imaging technique in determining specific physically relevant features. If smaller features are desired to
be accurately resolved, then a different image technique with greater resolution or different settings on the same instrument is required.

![Figure A.7](image)

**Figure A.7**: (a) Distribution of the area of cracks identified in sample A (FEM HMX pressed to a nominal TMD of 75%) measured in number of pixels. The number of cracks with an area of 30 pixels and above can be resolved within a 10% error. (b) Distribution of the mean crack thickness of cracks identified in sample B (Class V HMX pressed to a nominal TMD of 90%) measured in pixel length. Cracks with a thickness of 1.3 pixels and above can be resolved within a 15% error. The number of cracks with an area below the feature resolution limit and the number of cracks with a thickness below the scan resolution (i.e., less than one pixel) can be inferred from the best-fit distribution curves.

### A.3.2 Micro X-ray computed tomography data

Sample C consisted of a polymer bonded explosive material and was imaged with a Zeiss Xradia 520 Versa 3D X-ray microscope to inspect the inclusion of particles within the bulk sample, with a corresponding scan resolution of 19.5\( \mu \text{m} \) per voxel. These particles were identified following the same imaging processing techniques as demonstrated for the SEM micrographs by sampling 63 individual 2D image slices from the reconstructed 3D \( \mu \text{XCT} \) data. A total of 400788 individual particles were identified from the collected data and the distribution of the measured perimeters...
of the particles is shown in Fig. A.8. Again, these data were assumed to follow log-normal distributions and the feature resolution limit was found to be equivalent to a length of 33 pixels with an acceptable error limit of 15%. It is evident that in this case there exists a significant amount of information below the resolution limit and this result suggests that a higher resolution imaging technique is required to adequately capture this measured metric of interest.

![Particle Perimeter](image)

**Figure A.8**: Distribution of the measured perimeter of identified particles within Sample C (polymer bonded explosive material) measured from 2D image slices of reconstructed 3D µXCT data. The feature resolution limit of the void perimeter was found to be 33 pixels corresponding to an acceptable error limit of 15%. There exists a significant amount of data below the feature limit suggesting that a higher resolution imaging technique is needed for accurate representation of the distribution of the measured particle perimeter.

Percent error as a function of value of the investigated metrics of interest illustrates that the error increases as smaller features are included in the analysis, due to the pixelation error associated with the imaging techniques. These analyzed datasets highlight that different physical features have different resolution limits. The estima-
tion of the feature resolution limit is useful in determining the effective capability of the imaging technique and imaging parameters for specific features of interest.

A.4 Summary

A method to determine the feature resolution limit of physically relevant metrics of data obtained from multiple imaging acquisition techniques was developed. Specifically, the method fundamentally assumes a distribution of the physically relevant metric and calculates a best-fit PDF using only the larger, more accurately measured features while truncating the smaller, more uncertain features. The distribution of the data was represented using a KDE and the percent error between the best-fit PDF and KDE was calculated as a function of the span of the feature metric. The feature resolution limit was then selected according to the amount of user-specified acceptable error.

An advantage of this method over other techniques to determine feature resolution is its ability to be used on any measured data that follows an assumed distribution type. The determination of the feature resolution fundamentally accounts for the influence of the instrumentation and imaging parameters. Additionally, the developed process provides an estimation of the data that falls below the imaging resolution capability. However, it is critical that the dataset contains a sufficient number of samples to represent correctly the distribution of data, and that the data follows a known or assumed distribution. Violation of either of these requirements will invalidate the method’s ability to determine feature resolution.
This method was shown to provide useful information in determining the capability of an imaging technique to evaluate specific physically relevant features. This feature specific resolution and associated error information is much greater than the information provided from the scanning resolution alone and illustrates that each physical feature has its own independent resolution for accurate representation.
APPENDIX B

SOFTWARE CODE FOR SEM IMAGE PROCESSING

This appendix contains the MATLAB® R2019a code used to threshold and segment inclusion particles and void spaces in the 7075-T6 aluminum alloy SEM images.

```matlab
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% User inputs
save_im = 'no'; % Flag to save images: "yes" or "no"

% Flag to manually identify inclusion cracking: ...
% "yes" or "no"

% Flat to set what type of diameter ...
% representation to use when calculating pore/inclusion ...
dist_type = 'Feret';
% distance: "Feret" or "Eq"

% Eliminate everything smaller than this ...
% number of pixels in bwareaopen function
connected_area = 15;

% Number of pixels in each void must be ...
larger than this to be flagged
pixels_thresh = 15;

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% Load the images
```

[file, path] = uigetfile({'*.*;*.tif;*.tiff', 'TIFF Images';...
    '*.jpg;*.jpeg', 'JPEG Images';...
    '*.png', 'PNG Images'; '*.*', 'All Files'}), 'Select all the ...
    images to crop',...
'MultiSelect', 'on');
%
% Read selected images
if ischar(file) == 1
    filename = char(file(1, :)); % Convert cells to characters
    pic = imread(fullfile(path, filename)); % Load image into matlab
    singlefile = 1; % There is only 1 image loaded
else
    filename = char(file(1, 1)); % Convert cells to characters
    pic = imread(fullfile(path, filename)); % Load image into matlab
    singlefile = 0; % There are multiple images loaded
end
%
% Determine if there is more than one image to analyze
if singlefile == 1
    totalfiles = 1;
else
    totalfiles = size(file, 2);
end
%
% Load the image resolutions
data_par = xlsread('ImageData', 'Parallel Scan Resolution');
%
% Create empty matrix to store area information in
area_inclusions_tot = [];
area_pores_tot = [];
area_scan = [];
area_cracked_inclusions_tot = [];
Centroid_inclusions = [];
Centroid_pores = [];
EqDia_inclusions = [];
FeretDia_inclusions = [];
EqDia_pores = [];
FeretDia_pores = [];
PoreInclusion_distance = [];
Circularity_inclusions = [];
Eccentricity_inclusions = [];
Extent_inclusions = [];
Orientation_inclusions = [];

% Cycle through all the files
for zz=1:totalfiles
    % Get the image name
    if singlefile == 1
        filename = char(file(1, :)); % Convert cells to characters
    else
        filename = char(file(1, zz)); % Convert cells to characters
    end
    % Define directory and path for saving images
    SavePath = fullfile(path, 'Segmented Images\');
SavePathImage = fullfile(path, 'Segmented Images', ...)
    strtok(filename, '.');
% Load image into matlab
pic = imread(fullfile(path, filename));
% Set image resolution
im_name = strsplit(filename, '_');
im_name = im_name{1};
im_num = strsplit(im_name);
im_num = im_num{end};
im_res = data_par(str2double(im_num));

% Send image to the Inclusion Segmentation function
[area_inclusions, pixel_list_inclusions, inclusion_box, ...
    Centroid, EqDia, FeretDia, Circularity, Eccentricity, ...
    Extent, Orientation] = ...
    InclusionSegmentation(pic, connected_area, im_res);
% Append all the area information to the overall matrix
area_inclusions_scaled = area_inclusions*im_res^2;
area_inclusions_tot = vertcat(area_inclusions_tot, ...
    area_inclusions_scaled);
% Append centroid information of segmented inclusions to the ...
    overall matrix
Centroid_inclusions = vertcat(Centroid_inclusions, Centroid);
% Append diameter information of segmented inclusions to the ...
    overall matrix
EqDia_inclusions = vertcat(EqDia_inclusions, EqDia);
FeretDia_inclusions = vertcat(FeretDia_inclusions, FeretDia);
% Append shape metric data of segmented inclusions to the overall matrix

Circularity_inclusions = vertcat(Circularity_inclusions, Circularity);

Eccentricity_inclusions = vertcat(Eccentricity_inclusions, Eccentricity);

Extent_inclusions = vertcat(Extent_inclusions, Extent);

Orientation_inclusions = vertcat(Orientation_inclusions, Orientation);

% Send image to the Porosity Segmentation function

[area_pores, pixel_list_pores, pore_box, Centroid, EqDia, FeretDia] = PorositySegmentation(pic, connected_area, im_res);

% Append all the area information to the overall matrix

area_pores_scaled = area_pores*im_res^2;

area_pores_tot = vertcat(area_pores_tot, area_pores_scaled);

% Append centroid information of pore inclusions to the overall matrix

Centroid_pores = vertcat(Centroid_pores, Centroid);

% Append diameter information of segmented pores to the overall matrix

EqDia_pores = vertcat(EqDia_pores, EqDia);

FeretDia_pores = vertcat(FeretDia_pores, FeretDia);

% Store scan area of each image

pic_dim = size(pic); % Dimensions of image
pic_size = pic_dim(1)*pic_dim(2); % Total size (number of pixels) in image

pic_area = pic_size*im_res^2; % Area of image in um^2
pic_area = pic_area/1e6; % Convert area to mm^2
area_scan = vertcat(area_scan, pic_area); % Store image area to array

if strcmp(crack_chk, 'yes') == 1
% Check for inclusion cracking

threshold = 5; % Number of pixels the inclusions/pores must be next to eachother to trigger a positive

[inclusion_outlines, pore_outlines, ...
area_cracked_inclusions] = ...
InclusionCracking(inclusion_box, pore_box, ...
area_inclusions_scaled, threshold);

% Display image with flagged crack sites
pic_filt = imnlmfilt(pic);

pic_rgb = cat(3, pic_filt, pic_filt, pic_filt);
% Highlight the inclusions and pores
for i = 1:length(pixel_list_inclusions)
    pic_rgb(pixel_list_inclusions(i, 2), ...
    pixel_list_inclusions(i, 1), :) = 65535*[0.466 0.674 0.188];
end

for i = 1:length(pixel_list_pores)
    pic_rgb(pixel_list_pores(i, 2), pixel_list_pores(i, ... 1), :) = 65535*[0.85 0.325 0.098];
end

figure('Position', [420 89 1265 934])

imshow(pic_rgb)

% Draw the bounding boxes and have manual verification of the pores

del_idx = [];

for i = 1:size(inclusion_outlines, 1)
    BB = inclusion_outlines(i, :);
    box = rectangle('Position', [BB(1, 1)-BB(1, 3)*0.325, BB(1, 2)-BB(1, 4)*0.325, BB(1, 3)+BB(1, 3)*0.325, BB(1, 4)+BB(1, 4)*0.325], 'EdgeColor', 'b', 'LineWidth', 2);
    answer = questdlg('Is this representative of a crack?', 'Inclusion Cracking Selection', 'Yes', 'No', 'Yes');
    if strcmp(answer, 'No') == 1
        del_idx = vertcat(del_idx, i);
    end
    delete(box);
end

inclusion_outlines(del_idx, :) = [];
area_cracked_inclusions(del_idx) = [];

for i = 1:size(inclusion_outlines, 1)
    BB = inclusion_outlines(i, :);
box = rectangle('Position', [BB(1, 1), BB(1, 2), ... 
    BB(1, 3), BB(1, 4)], 'EdgeColor', 'b', ... 
    'LineWidth', 2);
end

set(gcf, 'Color', 'w')
sv_nm = fullfile(path, 'Inclusion Cracking\', ... 
    strtok(filename, '.'));
saveas(gcf, strcat(sv_nm, '_InclusionCracking.png'))
close(gcf)
area_cracked_inclusions_tot = ...
    vertcat(area_cracked_inclusions_tot, ... 
        area_cracked_inclusions);

% Save cracked inclusion information
saveName = strcat(filename, '_CrackedInclusionArea.mat');
save(saveName, 'area_cracked_inclusions');
end

% Save images with the inclusions and pores colored separately
if strcmp(save_im, 'yes') == 1
    pic_filt = imnlmfilt(pic);
    % Convert to RGB image
    pic_rgb = cat(3, pic_filt, pic_filt, pic_filt);
    % Highlight the inclusions and pores
    for i = 1:length(pixel_list_inclusions)
for i = 1:length(pixel_list_pores)
    pic_rgb(pixel_list_pores(i, 2), pixel_list_pores(i, 1), :) = 65535*[0.85 0.325 0.098];
end

end

% Save image with the inclusions and pores highlighted
figure('Position', [1989 252 915 699], 'visible', 'off')
imshow(pic_rgb)
set(gcf, 'Color', 'w')
% Save figure
saveas(gcf, strcat(SavePathImage, '_SegmentationOverlay.png'))
close(gcf)
end

% Sum the total scanned area
area_scan_tot = sum(area_scan) % Total analyzed scan area in mm^2

% Percentage of inclusions
pct_inclusions = (sum(area_inclusions_tot)/1e6)/area_scan_tot*100

% Percentage of porosity
pct_pores = (sum(area_pores_tot)/1e6)/area_scan_tot*100

% Total inclusions larger than the mean of the cracked inclusions
idxs = area_inclusions_tot >= 10^1.9927;
area_inclusions_cracklarge = area_inclusions_tot(idxs);
% Save the raw data
save('AreaData.mat', 'area_inclusions_tot', 'area_pores_tot', ...
     'area_cracked_inclusions_tot')
save('DistanceData.mat', 'PoreInclusion_distance')
save('InclusionProperties.mat', 'area_inclusions_tot', ...
     'Circularity_inclusions', ...
     'Eccentricity_inclusions', 'Extent_inclusions', ...
     'Orientation_inclusions')

% Finish the script
fprintf('\nend')

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
function [area, pixel_list, box, Centroid, EqDia, FeretDia, ...
     Circularity, Eccentricity, Extent, Orientation] = ...
     InclusionSegmentation(pic, connected_area, im_res, SavePath)

% Calculate the number of input arguments
if nargin == 4
    % Set flag to save the individual images
    save_flag = 1;
else
    save_flag = 0;
end

% Change image to grayscale
[\text{\neg, \neg, colors}] = \text{size(pic)};

\textbf{if} colors > 1

\hspace{1em} \text{pic\_gray} = \text{rgb2gray(pic)}; \hspace{1em} % Use weighted sum to convert all RGB channels to grayscale

\textbf{else}

\hspace{1em} \text{pic\_gray} = \text{pic}; \hspace{1em} % Do nothing

\textbf{end}

\hspace{1em} \% Filter the image to remove noise

\hspace{2em} \text{pic\_filt} = \text{imnlmfilt(pic\_gray)};

\hspace{1em} \% Threshold the image

\hspace{2em} \text{pic\_hist} = \text{imhist(pic\_filt)}; \hspace{1em} % Take histogram of image

\hspace{2em} [\text{hist\_peaks}, \text{hist\_location}] = \text{findpeaks(pic\_hist)}; \hspace{1em} % Calculate the peaks and locations of histogram

\hspace{2em} \% findpeaks(pic\_hist); \hspace{1em} % Plot the histogram peak figure

\hspace{2em} \text{gry\_location} = \text{find(hist\_peaks == max(hist\_peaks))}+1; \hspace{1em} % Find index of peak immediately after maximum

\hspace{2em} \% If there is no peak in the histogram after the maximum, reverse contrast assignment so image comes out all black (pores are white)

\hspace{2em} \textbf{if} \text{gry\_location} == 0

\hspace{3em} \text{gry\_value} = \text{hist\_location(gry\_location)}+1; \hspace{2em} % Use the maximum histogram value

\hspace{3em} \text{pic\_contrast} = \text{imadjust(pic\_filt, [0 gry\_value/255], [1 0])}; \hspace{1em} % Change contrast of black and white picture

\hspace{2em} \% If there is no histogram

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elseif isempty(gry_location) == 1
    gry_value = 254;
    pic_contrast = imadjust(pic_filt, [0 gry_value/255], [1 0]);

    % For "regular" histogram
else
    gry_value = hist_location(gry_location); % Find value of peak ...
        immediately before maximum

    % If there are two locations with the same peak
    if gry_value > 1
        gry_value = gry_value(1);
    end

    pic_contrast = imadjust(pic_filt, [0 gry_value/255], [0 1]); ...
        % Change contrast of black and white picture
end

% Image adjustments to isolate inclusions
pic_adjusted = imadjust(pic_contrast, [250/255 1], [0 1]); % ...
        Rethreshold all non-white values to black
pic_adjusted = bwareaopen(pic_adjusted, connected_area, 8); % ...
        Eliminate all white pixels smaller than the specified size
pic_adjusted = imclose(pic_adjusted, strel('disk', 5)); % Merge ...
        all pixels within 15 pixels disk radius
pic_adjusted = imdilate(pic_adjusted, strel('disk', 2)); % Dilate ...
        the grayscale image with a structured disk 2 pixels in radius
pic_adjusted1 = imerode(pic_adjusted, strel('disk', 1)); % Erode ...
        the grayscale image with a structured disk 1 pixels in radius
pic_adjusted2 = imerode(pic_adjusted, strel('disk', 2)); % Erode ... the grayscale image with a structured disk 2 pixels in radius

% Image segmentation:
% Area: Actual number of pixels in the region, returned as a scalar.
% BoundingBox: Smallest rectangle containing the region.
% Centroid: Center of mass of the region. The first element is ... the horizontal coordinate (or x-coordinate), the second ... element is the vertical coordinate (or y-coordinate).
% Circularity: Circularity that specifies the roundness of ... objects. For a perfect circle, the circularity value is 1.
% Eccentricity: The eccentricity is the ratio of the distance ... between the foci of the ellipse and its major axis length (0 = ... circle, 1 = line segment).
% EquivDiameter: Diameter of a circle with the same area as the ... region: sqrt(4*Area/pi).
% Extent: Ratio of pixels in the region to pixels in the total ... bounding box. Computed as the Area divided by the area of the ... bounding box.
% MaxFeretDiameter: Maximum feret diameter of a convex hull that ... encloses the segmented object
% PixelList: Locations of pixels in the region
region = regionprops(pic_adjusted1, 'Area', 'BoundingBox', ... 'Centroid', 'Circularity', 'Eccentricity', 'EquivDiameter', ... 'Extent', 'MaxFeretProperties', 'Orientation', 'PixelList'); % ... Find properties of each remaining white area
% Area properties

area = cat(1, region.Area); % Number of pixels in each remaining white area

area_scaled = area*im_res^2; % Area in um^2 of each white area

area_avg = mean(area_scaled);

pixel_list = cat(1, region.PixelList);

box = cat(1, region.BoundingBox);

Centroid = cat(1, region.Centroid);

EqDia = cat(1, region.EquivDiameter);

FeretDia = cat(1, region.MaxFeretDiameter);

Circularity = cat(1, region.Circularity);

Eccentricity = cat(1, region.Eccentricity);

Extent = cat(1, region.Extent);

Orientation = cat(1, region.Orientation);

if save_flag == 1
    % Create area histogram/KDE figure
    figure('visible', 'off')
    h = histogram(area_scaled, 50);
    % Calculate the total histogram area
    scale = sum(h.BinCounts*h.BinWidth);
    hold on
    KDE = calcDensity(area_scaled, 'range', ...
        [min(area_scaled)-std(area_scaled); ...
        max(area_scaled)+std(area_scaled)]);
    % Multiply KDE values by the scale factor to get KDE in terms ...

    of count

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% (default KDE is normalized with a total area of 1)
plot(KDE.GridVectors{1,1}, KDE.Values*scale, 'linewidth', 2)
xax = xlim; yax = ylim; xlim([0 max(h.BinEdges)]);

text(max(h.BinEdges)*0.70, yax(2)*0.75, strcat({'Mean ... Inclusion Area = ', num2str(round(area_avg)), {' ... \mu^2'}}, 'FontSize', 24, 'HorizontalAlignment', 'center'))

hold off

xlabel('Area (\mu^2)', 'FontSize', 24)
ylabel('Count', 'FontSize', 24)
title('Inclusion Area', 'FontSize', 24)

ax1 = gca;
ax1.XMinorTick = 'on'; ax1.YMinorTick = 'on';
x_axis = get(gca, 'XAxis'); set(x_axis, 'FontSize', 24);
y_axis = get(gca, 'YAxis'); set(y_axis, 'FontSize', 24);

set(gcf, 'Position', [2038 134 1044 911], 'Name', 'Inclusion ... Area', 'Color', 'w')

% Save figure
saveas(gcf, strcat(SavePath, '_inclusionArea.png'))
close(gcf)

% If there is only 1 inclusion, skip the KDE because it will ...
crash
if length(area) > 1
% Create log-normal area histogram/KDE figure

figure('visible', 'off')

h = histogram(log10(area_scaled), 50);

% Calculate the total histogram area

scale = sum(h.BinCounts*h.BinWidth);

hold on

KDE = calcDensity(log10(area_scaled), 'range', ...
    [min(log10(area_scaled))-std(log10(area_scaled)); ...
    max(log10(area_scaled))+std(log10(area_scaled))]);

% Multiply KDE values by the scale factor to get KDE in ...
% terms of count
% (default KDE is normalized with a total area of 1)

plot(KDE.GridVectors{1,1}, KDE.Values*scale, 'linewidth', 2)

xax = xlim; yax = ylim; xlim([0 max(h.BinEdges)]);

text(max(h.BinEdges)*0.70, yax(2)*0.75, strcat({'Mean ... 
    Inclusion Area = '}, num2str(round(area_avg)), {' ... 
    \mu m^2'}), 'FontSize', 24, 'HorizontalAlignment', 'center')

text(max(h.BinEdges)*0.70, yax(2)*0.675, ...
    strcat(num2str(length(find(area_scaled > 50))), {' ... 
    inclusions > 50 \mu m^2'}), 'FontSize', 24, ...
    'HorizontalAlignment', 'center')

hold off

xlabel('log_{10}Area (\mu m^2)', 'FontSize', 24)

ylabel('Count', 'FontSize', 24)

title('Inclusion Area', 'FontSize', 24)

ax1 = gca;
ax1.XMinorTick = 'on'; ax1.YMinorTick = 'on';

x_axis = get(gca, 'XAxis'); set(x_axis, 'FontSize', 24);
y_axis = get(gca, 'YAxis'); set(y_axis, 'FontSize', 24);
set(gcf, 'Position', [2038 134 1044 911], 'Name', ...
   'Inclusion Area', 'Color', 'w')

% Save figure
saveas(gcf, strcat(SavePath, '_inclusionArea_LogNormal.png'))
close(gcf)
end

% Create side-by-side image of original and segmented
figure('Position', [1788 379 1256 549], 'visible', 'off')
imshowpair(pic_filt, pic_adjusted1, 'montage')
% Save figure
saveas(gcf, strcat(SavePath, '_inclusionComparison.png'))
close(gcf)

% Create segmented image
figure('Position', [1989 252 915 699], 'visible', 'off')
imshow(pic_adjusted1)
set(gcf, 'Color', 'w')
% Save figure
saveas(gcf, strcat(SavePath, '_inclusionSegmentation.png'))
close(gcf)

% If there is only 1 inclusion, skip the KDE because it will ...
   crash
if length(area) > 1

    % Create centroid location "heat map"
    % Extract centroid data
    centroid_pts = cat(1, region.Centroid)/1000;
    x_pts = centroid_pts(:, 1);
    y_pts = centroid_pts(:, 2);
    % Calculate 2D KDE using function from MTEX toolbox
    kde2D = calcDensity([x_pts, y_pts], 'range', ...
        [min(x_pts)-std(x_pts), min(y_pts)-std(y_pts); ...
         max(x_pts)+std(x_pts), max(y_pts)+std(y_pts)]);

    % Create a contour grid to create contour map for KDE values
    [x, y] = ndgrid(linspace(min(centroid pts, [], 'all'), ...
        max(centroid pts, [], 'all')));

    figure('Position', [1989 252 915 699], 'visible', 'off')
    hold on
    contourf(x, y, kde2D(x, y))
    colormap jet
    shading interp
    plot(x_pts, y_pts, '.k', 'MarkerSize', 20)
    xlabel('Position (mm)', 'FontSize', 28)
    ylabel('Position (mm)', 'FontSize', 28)
    set(gcf, 'Color', 'w')
    saveas(gcf, strcat(SavePath, '_inclusionCentroid.png'))
    close(gcf)

end
end
function [area, pixel_list, box, centroid, EqDia, FeretDia] = ...  
PorositySegmentation(pic, connected_area, im_res, SavePath)

% Calculate the number of input arguments
if nargin == 4
  % Set flag to save the individual images
  save_flag = 1;
else
  save_flag = 0;
end

% Change image to grayscale
[~, ~, colors] = size(pic);
if colors > 1
  pic_gray = rgb2gray(pic); % Use weighted sum to convert all RGB channels to grayscale
else
  pic_gray = pic; % Do nothing
end

% Filter the image to remove noise
pic_filt = imnlmfilt(pic_gray);

% Threshold the image
pic_hist = imhist(pic_filt); % Take histogram of image

[hist_peaks, hist_location] = findpeaks(pic_hist); % Calculate ... the peaks and locations of histogram

% findpeaks(pic_hist); % Plot the histogram peak figure

gry_location = find(hist_peaks == max(hist_peaks))-1; % Find ... index of peak immediately after maximum

% If there is no peak in the histogram after the maximum, reverse % contrast assignment so image comes out all black (pores are white)
if gry_location == 0
    gry_value = hist_location(gry_location+1); % Use the maximum ... histogram value
    pic_contrast = imadjust(pic_filt, [0 gry_value/255], [1 0]); ... % Change contrast of black and white picture

% If there is no histogram
elseif isempty(gry_location) == 1
    gry_value = 254;
    pic_contrast = imadjust(pic_filt, [0 gry_value/255], [1 0]);

% For "regular" histogram
else
    gry_value = hist_location(gry_location); % Find value of peak ... immediately before maximum
    % If there are two locations with the same peak
    if gry_value > 1
        gry_value = gry_value(1);
    end
pic_contrast = imadjust(pic_filt, [0 gry.value/255], [0 1]); ...  
% Change contrast of black and white picture
end

% Image adjustments to isolate inclusions
pic_adjusted = imadjust(pic_contrast, [5/255 1], [0 1]); % ...  
Rethreshold all non-white values to black
pic_adjusted = imcomplement(pic_adjusted); % Inverse the colors ...  
so pores show as white
pic_adjusted = bwareaopen(pic_adjusted, connected_area, 8); % ...  
Eliminate all white pixels smaller than the specified size
pic_adjusted = imclose(pic_adjusted, strel('disk', 5)); % Merge ...  
all pixels within 15 pixels disk radius
pic_adjusted = imdilate(pic_adjusted, strel('disk', 2)); % Dilate ...  
the grayscale image with a structured disk 2 pixels in radius
pic_adjusted1 = imerode(pic_adjusted, strel('disk', 1)); % Erode ...  
the grayscale image with a structured disk 1 pixels in radius
pic_adjusted2 = imerode(pic_adjusted, strel('disk', 2)); % Erode ...  
the grayscale image with a structured disk 2 pixels in radius

% Image segmentation:
% Area: Actual number of pixels in the region, returned as a scalar.
% BoundingBox: Smallest rectangle containing the region.
% Centroid: Center of mass of the region. The first element is ...  
the horizontal coordinate (or x-coordinate), the second ...  
  element is the vertical coordinate (or y-coordinate).
% Circularity: Circularity that specifies the roundness of objects. For a perfect circle, the circularity value is 1.
% Eccentricity: The eccentricity is the ratio of the distance between the foci of the ellipse and its major axis length (0 = circle, 1 = line segment).
% EquivDiameter: Diameter of a circle with the same area as the region: sqrt(4*Area/pi).
% Extent: Ratio of pixels in the region to pixels in the total bounding box. Computed as the Area divided by the area of the bounding box.
% MaxFeretDiameter: Maximum feret diameter of a convex hull that encloses the segmented object
% PixelList: Locations of pixels in the region

region = regionprops(pic_adjusted1, 'Area', 'BoundingBox', 'Centroid', 'Circularity', 'Eccentricity', 'EquivDiameter', 'Extent', 'MaxFeretProperties', 'PixelList'); % Find properties of each remaining white area

% Area properties
area = cat(1, region.Area); % Number of pixels in each remaining white area
area_scaled = area*imres^2; % Area in um^2 of each white area
area_avg = mean(area_scaled);
pixel_list = cat(1, region.PixelList);
box = cat(1, region.BoundingBox);
centroid = cat(1, region.Centroid);
EqDia = cat(1, region.EquivDiameter);
FeretDia = cat(1, region.MaxFeretDiameter);

if save_flag == 1
    % If there is only 1 pore, skip the KDE because it will crash
    if length(area) > 1
        % Create area histogram/KDE figure
        figure('visible', 'off')
        h = histogram(area_scaled, 50);
        % Calculate the total histogram area
        scale = sum(h.BinCounts*h.BinWidth);
        hold on
        KDE = calcDensity(area_scaled, 'range', ...
                          [min(area_scaled)-std(area_scaled); ...
                          max(area_scaled)+std(area_scaled)]);
        % Multiply KDE values by the scale factor to get KDE in ...
        % terms of count
        % (default KDE is normalized with a total area of 1)
        plot(KDE.GridVectors{1,1}, KDE.Values*scale, 'linewidth', 2)
        xax = xlim; yax = ylim; xlim([0 max(h.BinEdges)]);
        text(max(h.BinEdges)*0.70, yax(2)*0.75, strcat(
            {'Mean ... Pore Area = '}, num2str(round(area_avg)), {' ... \(\mu m^2\)'),'FontSize', 24, 'HorizontalAlignment', ...
            'center'})
        text(max(h.BinEdges)*0.70, yax(2)*0.675, ...
            strcat(num2str(length(find(area_scaled > 50))), {' ... pores > 50 \(\mu m^2\)'),'FontSize', 24, ...
            'HorizontalAlignment', 'center'})
hold off

xlabel('Area (\text{\textmu m}^2)', 'FontSize', 24)
ylabel('Count', 'FontSize', 24)
title('Porosity Area', 'FontSize', 24)

ax1 = gca;

ax1.XMinorTick = 'on'; ax1.YMinorTick = 'on';

x_axis = get(gca, 'XAxis'); set(x_axis, 'FontSize', 24);
y_axis = get(gca, 'YAxis'); set(y_axis, 'FontSize', 24);

set(gcf, 'Position', [2038 134 1044 911], 'Name', ...
    'Porosity Area', 'Color', 'w')

% Save figure

saveas(gcf, strcat(SavePath, '_porosityArea.png'))
close(gcf)

end

% Create side-by-side image of original and segmented

figure('Position', [1788 379 1256 549], 'visible', 'off')
imshowpair(pic_filt, pic_adjusted1, 'montage')

% Save figure

saveas(gcf, strcat(SavePath, '_porosityComparison.png'))
close(gcf)

% Create segmented image

figure('Position', [1989 252 915 699], 'visible', 'off')
imshow(pic_adjusted1)

set(gcf, 'Color', 'w')

% Save figure
saveas(gcf, strcat(SavePath, '_porositySegmentation.png'))

close(gcf)

% If there is only 1 pore, skip the KDE because it will crash
if length(area) > 1
    % Create centroid location "heat map"
    % Extract centroid data
    centroid_pts = cat(1, region.Centroid)/1000;
    x_pts = centroid_pts(:, 1);
    y_pts = centroid_pts(:, 2);
    % Calculate 2D KDE using function from MTEX toolbox
    kde2D = calcDensity([x_pts, y_pts], 'range', ...
        [min(x_pts)-std(x_pts), min(y_pts)-std(y_pts); ...
        max(x_pts)+std(x_pts), max(y_pts)+std(y_pts)]);
    % Create a contour grid to create contour map for KDE values
    [x, y] = ndgrid(linspace(min(centroid_pts, [], 'all'), ...
        max(centroid_pts, [], 'all')));
    figure('Position', [1989 252 915 699], 'visible', 'off')
    hold on
    contourf(x, y, kde2D(x, y))
    colormap jet
    shading interp
    plot(x_pts, y_pts, '.k', 'MarkerSize', 20)
    xlabel('Position (mm)', 'FontSize', 28)
    ylabel('Position (mm)', 'FontSize', 28)
    set(gcf, 'Color', 'w')
    saveas(gcf, strcat(SavePath, '_porosityCentroid.png'))
close(gcf)
  end
  end
end
REFERENCES


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[195] Saul N. Friedman, George S. K. Fung, Jeffrey H. Siewerdsen, and Benjamin M. W. Tsui. A simple approach to measure computed tomography (CT) modulation transfer function (MTF) and noise-power spectrum (NPS) using the American College of Radiology (ACR) accreditation phantom. *Medical Physics*, 40(5):051907, apr 2013.


