THE MICROSTRUCTURAL EVOLUTION OF FATIGUE CRACKS IN FCC METALS

BY

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DISSERTATION
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The microstructural evolution during fatigue crack propagation was investigated in a variety of planar and wavy slip FCC metals. The planar materials included Haynes 230, Nitronic 40, and 316 stainless steel, and the wavy materials included pure nickel and pure copper. Three different sets of experiments were performed to fully characterize the microstructural evolution. The first, performed on Haynes 230, mapped the strain field ahead a crack tip using digital image correlation and electron backscatter diffraction techniques. Focused ion beam (FIB) lift-out techniques were then utilized to extract transmission electron microscopy (TEM) samples at specific distances from the crack tip. TEM investigations compared the measured strain to the microstructure. Overall, the strain measured via DIC and EBSD was only weakly correlated to the density of planar slip bands in the microstructure.

The second set of experiments concerned the dislocation structure around crack tips. This set of experiments was performed on all the materials. The microstructure at arrested fatigue cracks on the free surface was compared to the microstructure found beneath striations on the fracture surfaces by utilizing FIB micromachining to create site-specific TEM samples. The evolved microstructure depended on the slip type. Strong agreement was found between the crack tip microstructure at the free surface and the fracture surface. In the planar materials, the microstructure in the plastic zone consisted of bands of dislocations or deformation twins, before transitioning to a refined sub-grain microstructure near the crack flank. The sub-grain structure extended 300-500 nm away from the crack flank in all the planar slip materials studied. In contrast, the bulk structure in the wavy slip material consisted of dislocation cells and did not transition to a different microstructure as the crack tip was approached. The strain in wavy slip was highest near the crack tip, as the misorientations between the dislocation cells increased and the cell size decreased as the crack flank was approached.

The final set of experiments involved reloading the arrested crack tips in monotonic tension. This was performed on both the Haynes 230 and 316 stainless steel. This technique exposed the fracture surface and location of the arrested crack tip away from the free surface, allowing for a sample to be extracted via FIB micromachining and TEM evaluation of the microstructure. This permitted the crack tip microstructure to be investigated without exposing the microstructure to crack closure.
or free surface effects. These experiments confirmed what was inferred from the earlier experiments, namely that the banded structure was a product of the crack tip plastic zone and the refined structure was a product of the strain associated with crack advance.

Overall the microstructural complexity presented in this work was much higher than would be predicted by current models of fatigue crack propagation. It is recommended that future models attempt to simulate interactions between the dislocations emitted during fatigue crack growth and the pre-existing microstructure to more accurately simulate the processes occurring at the crack tip during crack growth.
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CHAPTER 1
INTRODUCTION

Fatigue of materials, the subcritical failure of materials when exposed to cyclic stresses, is thought to account for 90% of mechanical service failures [1]. The particulars of the process, from damage induced by fatigue [2], initiation of fatigue cracks [3] and the propagation of fatigue cracks [4] have been extensively studied and reviewed. The total fatigue lifetime is determined by the lifetime of each of the phases combined, however each of the phases does not contribute equally. During high-cycle fatigue, the crack initiation phase is dominant and accounts for up to 80% of the lifetime. In contrast, during low-cycle fatigue 80% of the lifetime consist of the crack propagation phase. This difference is largely attributable to the difference in the number of cycles necessary for crack initiation; the total number of cycles for fatigue crack propagation remains relatively unchanged.

Investigations into fatigue crack growth have relied on laboratory testing of standardized samples, which are then extrapolated to cracks in other configurations by the principle of similitude, which claims that cracks of the same material at the same stress intensity factor will have the same crack growth rate. This principle arises from the idea that the stress intensity factor completely describes the stress conditions at the crack tip, including the stress and strain fields [5]. However, recent research has called into question this principle, suggesting that specimen geometry [6-9] and microstructural features [10, 11] play a significant role in determining the plastic zone, and hence the stress and strain fields, of the crack tip. This has led to proposals to replace the current empirical models of crack growth with physically-based design principles, formed by combining continuum mechanics with mesoscale and atomistic simulations to more accurately model fatigue crack growth [12].

Accurate portraits of the microstructure are needed to link the strain predicted by continuum mechanics with the dislocation structure predicted by the models. The microstructural evolution is effected by three separate factors: the cyclic loading, the plastic zone, and the strain associated with crack advance. While the microstructural evolution under cyclic loading is well understood [13-15], how this structure is modified first by the crack tip plastic zone and then by the strain associated with crack advance has not been characterized in detail. Previous attempts at
characterized the evolved microstructure have used two different techniques. The first technique involves directly interrogating the microstructure via transmission electron microscopy (TEM) [16-28]. This technique has several drawbacks, mainly on the small sampled area and the difficulty of sample preparation. The dislocation microstructures next to crack tips have been partially characterized, but the various strain influenced evolution of the microstructure is unknown. Additionally, these techniques have heretofore been limited to interior cracks, and were unable to investigate cracks on the free surface.

The second technique involves mapping the strains contained within the plastic zone [10, 29-31]. These studies employed indirect strain mapping techniques that track the movement on the free surface to deduce the strain field. However, there are limitations of these techniques and the information they can access. Current strain mapping techniques:

1. measure strains at the surface, and the stress state at the surface is not the same as the interior. Due to the free surface, there are three possible directions of strain, while the interior of the grains are limited to two directions.
2. are limited in resolution. While the capabilities of very high spatial resolution, on the order of 140nm x 140 nm, have been demonstrated [32], these rely on precise speckle pattern generation. In experiments mapping the strain around a fatigue crack tip, a more practical resolution of 870 nm x 870 nm has been achieved [10]. This prevents any accurate strain measurements within this distance from the crack tip where strains are at their highest.
3. are an indirect measurement technique, and there is no correlation with the evolved microstructural state.

Thus, for FCC metal alloy the following questions remain:

1. Can a correlation be drawn between the measured plastic strain at the surface and the underlying dislocation microstructure?
2. Can samples be produced to examine the dislocation microstructure and the strain state at the crack tips on the free surface?
3. Can a comparison be drawn between the dislocation microstructure at the crack tip at the surface and in the interior of the sample?
4. Are the dislocation structures produced by a fatigue crack in a ductile FCC metal universal across along ductile FCC metals or do significantly different microstructures exists?

5. Can the contribution of the different influences of the plastic strain be identified?

6. How do the dislocation microstructures around crack tips compare to those predicted by physically-based models?

To answer these questions, single edge notch tension specimens will undergo fatigue loading to propagate cracks. The crack will be arrested and the plastic zone surrounding it will be examined via digital image correlation and electron backscatter diffraction. Then a Focused Ion Beam (FIB) will be utilized to produce site specific Transmission Electron Microscope (TEM) samples. TEM samples will be produced at both the free surface from an arrested crack, and the fracture surface from cracks allowed to propagate to failure or loaded monotonically after arrest.

A review of the current understanding of fatigue crack propagation is presented in Chapter 2. The experimental methods are discussed in Chapter 3, while the results are presented in Chapter 4 and discussed in Chapter 5. The conclusion about the dislocation structures created by propagating fatigue cracks are presented in Chapter 6.

1.1 References

CHAPTER 2
BACKGROUND

The total lifetime of materials under cyclic loading is dependent on the two interrelated phenomena of crack initiation and crack propagation. The lifetime of each stage varies based on the applied stress, the microstructure, and the material. The relevant literature on fatigue damage and crack initiation is reviewed first to provide context for the subsequent review of fatigue crack growth, and provide a detailed description of the dislocation microstructures associated with cyclic loading. This work will focus on metals with a face centered cubic (FCC) crystal structure and works related to this crystal structure will be discussed whenever applicable. Occasionally relevant works utilizing body centered cubic (BCC) or hexagonal close packed (HCP) crystal-structured metals will be discussed in relation to the current topic. Any change in the crystal structure will be noted whenever it occurs. Additionally, there are two different classes of FCC metals: wavy and planar slip. Much of the existing literature on the fatigue describes wavy slip materials and these materials will generally discussed first in relation to the current topic, with planar slip materials described later.

This chapter is organized as follows: discussion of total fatigue lifetime driven by the deformation under cyclic loading; the dislocation structure formed under pure cyclic loading; fatigue crack initiation; and finally fatigue crack propagation. Models relevant to the current topic will be discussed following experimental evidence on the topic.

2.1 Fatigue Lifetime and the S/N Curve
The most basic method of studying fatigue failure and fatigue crack growth is through the relationship between applied cyclic stress and the number of cycles to failure, otherwise known as the S/N curve [1]. Whöler, credited as the first to study fatigue, generated S/N curves in his study of railway axles [2]. The dependence of the S/N curve on material is demonstrated in Figure 2.1[3], which compares the S/N curves for 1045 steel, a BCC mild carbon steel, and 2014-T6 aluminum, an FCC aluminum-copper alloy [1]. These materials represent the two primary types of material response to fatigue loading. The first, represented by the aluminum alloy, shows that the number
of cycles to failure increases with decreasing applied cyclic stress. However, the alloy will eventually fail for any level of applied stress. The second response, here typified by the 1045 steel, has a similar increase in the number of cycles to failure upon decreasing cyclic stress, until $10^6$ cycles. After $10^6$ cycles, there is no further decrease in the cyclic stress necessary to cause failure, indicating that the sample will not fail as long as the cyclic stress is at or below this stress. This stress is known as an endurance or fatigue limit.

![Figure 2.1: A comparison between the applied stress amplitude and the number of cycles to failure for 1045 steel and 2014-T6 aluminum. The fatigue limit, or the endurance limit is the applied stress amplitude below which 1045 will never fail.][3]

The S/N curve is typically divided into three stages [4]. The first stage is composed of two processes: crack initiation and microstructurally small crack (MSC) growth. Upon loading, the sample will have multiple crack initiation sites and microstructurally small fatigue cracks. The first stage ends when one of the small fatigue cracks grows large enough to initiate long crack growth, transitioning into the second stage. At high applied loads the number of cycles within the first stage is essentially zero, while at low applied loads the first stage accounts for 90% of the total number of cycles to failure [1]. The first stage is dependent on both the applied load and the local microstructure. The microstructural dependence gives rise to scatter at low applied loads. At these loads, both crack initiation and MSC growth will be dependent on stress concentrators, which
can include precipitates, grain boundaries and surface features. Since the size and distribution of stress concentrators will vary from sample to sample, the total fatigue life will be dependent on the largest stress concentrator in the sample. Such variance makes the accurate prediction of the total fatigue life challenging.

The second stage involves crack growth. The number of cycles within this stage remains relatively constant across a large range of applied stresses. At high stresses, most of the fatigue life is spent in the crack growth stage, as the crack initiation stage is nearly nonexistent. At low applied stresses, the number of cycles in stage two increases, but the large increase in the number of cycles necessary for crack initiation makes crack growth a comparatively small component of the total number of cycles to failure. This second portion of the S/N curve is mostly occupied by steady state crack growth, with the stress intensity factor related to the amount of crack growth per cycle via a power law relationship [5].

The final stage is the failure process. This stage occurs when the remaining uncracked ligament of the material can no longer support the maximum applied load. The sample then fails upon a final loading cycle by processes that are similar to that of monotonically-failed samples.

2.2 Simulation of Fatigue Lifetimes

Various methods have been used to attempt to simulate material fatigue lifetimes. These have been extensively reviewed by Fatemi and Yang [6] and, while a complete review would be beyond the scope of this section, the methods broadly can be categorized as those utilizing either damage-based, energy-based, or crack-growth-based criteria for failure.

Damage-based models were first proposed by Palmgren [7] and represented mathematically by Miner [8]. Such models assume the material has a finite amount of damage that can be absorbed before failure, with the endurance limit serving as an indicator of the critical damage needed for failure [9]. The estimate of accumulated damage was based on the amount of work absorbed per cycle. The original linear damage rule did not include any load-level interaction, chiefly the lower crack growth rate following an overload (i.e. when a higher than typical stress is applied to the sample). Later iterations on the model introduced load-level dependence by introducing a power relationship [10] or damage curves [11]. Additional accuracy was gained by dividing the
summation into two parts: the amount of damage needed to initiate a crack; and the amount of
damage needed to propagate the crack to failure [12]. However, this model could no longer account
for load-level interactions.

Crack-growth-based models attempt to apply the growth modes described by linear elastic and
elastic-plastic fracture mechanics to predict fatigue lifetimes. Simple models rely on summation
of the crack growth from each cycle based on power laws, while simultaneously accounting for
the effect of crack closure [13, 14]. More complex models attempt to model retardation that occurs
from load-level interactions by including the effect of overloads on the residual plastic zone [15].
To more accurately model crack growth across a range of crack lengths, the model can be divided
into the small crack growth and long crack growth regimes [16-18]. Unfortunately, the small crack
growth regime has proven difficult to accurately model as it is microstructurally sensitive and the
mechanisms are poorly understood.

The final type of lifetime model is based on the strain energy [19, 20]. Strain energy models are
popular as they can interpret loading types other than cyclic fatigue loading. The strain energy can
be computed via the cyclic stress-strain curve (CSSC) for Masing-type behavior typical of most
metallic materials [21]. To incorporate the effect of mean stress, the summation of the plastic and
elastic components are preferred, though early models used only the plastic component [22]. The
failure criterion is determined by constructing a power law relation analogous to an S/N curve [23,
24]. Total energy can then be summed in a linear [22] or non-linear fashion [25]. The advantage
of energy-based lifetime models is that non-linear damage models can be constructed to account
for both load interactions and the changes in the strain hardening in response to the stress.

In summary, a variety of models have been proposed to simulate the total fatigue lifetime without
using preexisting S/N curve data. The models have a tendency to separate total lifetime into
multiple parts, one of which is crack propagation. Understanding crack propagation is essential to
producing accurate models of fatigue lifetime.

2.3 Cyclic Strain Behavior
Examining the damage done to a material by cyclic strain is important for understanding the
resistance of a material to fatigue crack initiation. The response of the material to cyclic loading
can be measured by the cyclic stress-strain curve (CSSC). An example of a CSSC for a 4340 steel is shown in Figure 2.2 [26]. The CSSC takes the form of a hysteresis loop that expands each cycle due to cyclic hardening, which occurs because of a microstructural accumulation of dislocations [27]. The material will cyclically harden until a saturation stress is reached, wherein the yield stress ceases to increase despite a constant plastic strain amplitude. The saturation stress is dependent on the plastic strain amplitude. Additionally, the material yields at a lower stress during the reverse cycle than during the initial loading due to the accumulation of dislocations in long range pile-ups [28]. When the stress is reversed, the high internal strain energy associated with the development of dislocation pile-ups ensures that dislocation motion begins at a lower stress than the initial yield stress. This phenomenon is termed the Bauschinger effect.

Figure 2.2: Representative monotonic (dashed line) and cyclic (solid line) stress-strain curves for 4340 steel [26]
In addition to cyclic hardening, there is a second type of material response typical of high strength and work-hardened materials wherein the material yields at a lower stress during each cycle until a saturation stress is reached. This response is known as cyclic softening. In high strength materials, this occurs because mechanisms for dislocations to bypass microstructural obstacles become energetically more favorable after several cycles of loading [5, 26]. In work-hardened materials, cyclic softening occurs as dislocations accumulate in areas of greater density, resulting in the mutual annihilation of existing dislocations [29]. The saturation stress reached by cyclic softening in work-hardened materials is the same as the saturation stress reached by cyclic hardening in well-annealed versions of the same materials at a given plastic strain amplitude.

In single crystals, there is a plateau in the saturation stress across a range of plastic strain amplitudes. This is clearly seen in Figure 2.3, a semi-logarithmic graph of average peak saturation shear stress versus the resolved plastic shear strain for a copper single crystal loaded along an orientation to produce single slip with a Schmid factor of 0.5. The plateau occurs at the region in the center where increases in the plastic strain amplitude cause no increase in the saturation stress. The plateau is attributed to the actions of Persistent Slip Bands (PSBs), a dislocation structure that will be further described in Section 2.4 [27, 30]. The plateau at the saturation stress does not exist for polycrystalline materials as the formation of the plateau region is reliant on both the formation

Figure 2.3: Peak shear stress vs applied plastic shear strain amplitude for a single crystal of copper loaded along an orientation to produce single slip with a Schmid factor of 0.5, exhibiting a plateau region [30]
of PSBs and unconstrained deformation of the sample [31]. In a polycrystalline material, only the surface grains are unconstrained, preventing the plateau from forming.

Understanding the evolution of the dislocation structure under cyclic loading is essential to understanding cyclic hardening and the microstructure surrounding crack tips. The evolution of the dislocation distribution in FCC crystals under cyclic loading varies depending on the type of slip observed. Two types of slip, wavy and planar, are observed in FCC metals, which reflect the ease with which dislocations can cross-slip in the metal. The stacking-fault energy can serve as an indicator of slip planarity, with low stacking-fault energy generally associated with planar slip in a material. However, stacking-fault energy is not the only determining factor, especially under cyclic loading. The size and shape of precipitates [32, 33] and the presence of short-range order [34, 35] can act to suppress cross-slip and induce planar slip. Another factor that influences the deformation type is the amount of deformation. For instance, austenitic stainless steels will act as planar slip materials at low plastic strain amplitudes and as wavy slip materials at high plastic strain amplitudes [36-38]. At medium plastic strain amplitudes, a mixture of the planar and wavy structures are seen with no specific transition structure [39]. The causes of this transition are unclear.

Planar slip materials have a different response to cyclic loading than wavy slip materials. While the mechanisms of cyclic hardening and softening are similar in planar slip materials, planar slip materials do not show a plateau in the saturation stress [40]. The missing plateau region is a result of planar slip materials not forming PSBs, the cause of the plateau in wavy slip materials.

In summary, the response of a material to cyclic loading can be measured using the CSSC. The possible responses include cyclic hardening and softening. The response is plainly influenced by the microstructure, with both hardening and softening driven by changes to the dislocation density. In addition, the potential plateau in the saturation stress shows that dislocation structures and a slip character play a key role in determining the response of the material to cyclic stress.

2.4 Wavy Slip Microstructural Response to Cyclic Loading
The microstructural response to cyclic hardening depends on the type of slip and must be understood for proper evaluation of the dislocation structures surrounding fatigue cracks. The low
energy dislocation structures that form in response to cyclic loading and generate the cyclic stress-strain response have been extensively studied [41-43]. This evaluation was initially developed in an attempt to describe the response of single crystals, but is also applicable to large-scale polycrystals. Copper was primarily studied as a model material for wavy slip single and polycrystalline material response with the focus on the dislocation evolution through cyclic hardening. Thus, the following description is focused on the microstructural evolution of wavy slip materials; the response of planar slip materials is detailed in Section 2.7.

For wavy slip FCC materials the initial material response is dominated by dipole-dipole interactions [42]. A dislocation dipole is a structure formed by two dislocations with opposite Burgers vectors on parallel slip planes, lowering the total stress field compared to that of individual dislocations. As the dislocation density increases, interaction between the stress fields of different dislocation dipoles will occur. These dipole-dipole interactions raise the stress required to create new dislocations, slowing the dislocation generation process. As cyclic stress is continually applied to the sample, the dislocation dipoles will be forced closer together. These dipoles begin to form into larger, ordered structures, initiating the loop patch structure [42, 44].

A loop patch is composed of a dense set of dislocations arranged in a Taylor lattice [42]. While initially formed from the dislocation dipoles, the loop patch then grows via the trapping of random glissile dislocations that were moving through the lattice in response to the applied stress. The loop patches represent strong barriers to dislocation glide, especially at the density in which they develop in fatigue loading, which leads to the simultaneous development of the vein structure. The vein structure consists of relatively dislocation-free channels between loop patches and allows dislocation glide. Both the glide dislocations and the loop patches will carry the plastic strain associated with the fatigue loading, with loop patches carrying slightly less strain due to the higher strength of the loop patches [41]. The glide dislocations in the vein structure have the same sign and orientation, though the sign will flip every half cycle as the strain and direction of dislocation motion reverses.

At increasing plastic strain the loop patches begin to break down and PSBs form. This process is not instantaneous and both PSBs and the loop patch structures can exist in the material at the same time, through the plateau region, until the entire microstructure is converted to PSBs. PSBs are bands of dislocations that form and carry the plastic microstrain of the material [27, 42, 45]. The
exact mechanism by which PSBs form has been debated, with possible mechanisms including: breakdown of loop patches due to secondary slip [43], hardening of the veins between loop patches [46], or a lack of plastic deformation within the vein structure [47, 48]. Regardless of the origin of PSBs, dislocation dipoles agglomerate to form the PSB walls [49, 50]. Since the walls of the PSB act as strong barriers to dislocation slip, the material hardens. During cyclic loading, both the dislocation density and the hardness of the PSB wall will increase [51]. PSBs localize large parts of the strain within themselves and not in the harder loop patch structure and allow for continued deformation within the walls. This combination produces a 3D dislocation arrangement of PSB walls, which all contain dislocations on the primary slip plane [52]. The walls consist entirely of edge dislocation dipoles, as screw dislocations are likely to cross-slip and annihilate [45, 53, 54]. The walls have a near-zero net Burgers vector, though there are some residual internal stresses [27, 55, 56]. PSBs appear as a ladder-like structure of short, irregular cylinders contained within the dense walls when viewed normal to the primary slip plane, and as a series of elongated dislocation cells when viewed normal to the primary Burgers vector. These dislocation cells appear similar to those formed under monotonic deformation, however they are fundamentally different as they only consist of dislocations with the same Burgers vector. The structure begins to resemble dislocation cells even more strongly upon the activation of secondary slip dislocations, which will intersect with the PSB walls and further divide the structure into dislocation cells [57].

PSBs are found in both single crystal and polycrystalline materials, though the dislocation wall structure only occurs in favorably-oriented grains for the latter [58-60]. For example, PSBs do not form in grains with a <111> or <100> orientation parallel to the loading direction [61]. PSBs also form more easily in larger grains than smaller ones [61-63], with computer simulation suggesting a minimum grain size of 1 µm for dislocation wall formation typical of PSBs [64]. PSBs have been demonstrated to affect the local stresses on grain boundaries [65] and twin boundaries [66], which can result in microcrack formation [67].

PSB-grain boundary interactions are complex phenomena that vary based on the grain boundary type and misorientation. It is important to note that there is a distinction between the transmission of a PSB across a grain boundary and the transmission of dislocations within the PSB across a grain boundary. Transmission of PSBs involves the continuation of a PSB on either side of a grain boundary. The transmission of a PSB across a grain boundary requires that the operative slip
system is coplanar across the grain boundary [68]. This means that generally PSBs are capable of transmission across low angle grain boundaries (those with a misorientation angle <15°) [69-71] and not across random high angle grain boundaries [70, 72]. The dislocations within the PSB are capable of transmission across random high angle grain boundaries with the transmission governed by the magnitude of the residual Burgers vector left in the grain boundary [73-75]. As a consequence, high angle grain boundary-PSB interactions are likely spots for fatigue crack initiation due to the build-up of damage and residual dislocations in the boundary.

In summary, the evolution of the dislocation microstructure under cyclic loading is well understood for copper and is thought to be broadly applicable to other wavy slip materials. A similar dislocation evolution has been found in nickel [76], which would be considered as another wavy slip material. The initial response is the formation of dislocation dipoles that grow into loop patches with dislocation-free veins between them. The final structure initiates with the formation of PSBs and has both ladder-like components and dislocation cell components. The formation and structure of these dislocation features must be determined if the process of fatigue failure is to be understood.

2.5 Modeling of Low Energy Dislocation Structures and Persistent Slip Bands

The evolution of wavy slip fatigue dislocation structures has been investigated via a number of different models. Neumann utilized an early two-dimensional dislocation dynamics model that balanced the forces acting on an edge dislocation in a Taylor lattice in order to demonstrate that loop patch and vein structures were stable, low energy structures [77, 78]. The two-dimensional model has been extended to a three-dimensional dislocation dynamics model to show how the slip band arrangements at the surface would result in surface roughness in the form of intrusions and extrusions on the surface [79-81].

Other work has focused on the stress field created by the dislocation structures. Brown investigated dislocation dipoles using the Airy’s stress functions to solve the stress field to understand long-range stresses in the PSBs [82] and dipole cross-slip events [83] in fatigue. He attempted to find the minimum spacing that can be developed between screw dislocations before annihilation by cross-slip occurs. Brown related the minimum spacing to the saturation stress in cyclic loading and claims that this spacing is the sole determinant of cyclic deformation properties [82].
Essman and Mughrabi created a model of a PSB in single crystals focused around randomly distributed, irreversible slip process caused by the equilibrium of dislocation multiplication and annihilation in the saturated wall of the PSB [53]. The model demonstrated that the dislocation density within the PSB was relatively constant. The annihilation produced a net excess of vacancies, which diffused to the free surface along the PSB walls.

2.6 Modeling of Crack Initiation from Persistent Slip Bands

Modeling efforts of PSBs have also focused on the strain accumulation and crack initiation. Lin and Ito [84] developed a model of the plastic strain field of the dislocation. This model was revisited and expanded by Mura and Tanaka [85-87] to create a micro-mechanical model of PSBs in relation to crack initiation. The model treated the PSB as two series of dislocations on opposite layers accounting for deformation from the cyclic loading. The model exhibits slip irreversibility arising from irreversible layer deformation due to the differing levels of back stresses on the planes. Failure occurred when the energy of the PSB reached the energy necessary to crack the material. This model demonstrated that the shear stress within the PSB had an inverse square relationship with the grain size, similar to a Hall-Petch model. This stress distribution was confirmed by EBSD analysis performed by Britton and Wilkinson [88].

Later models of PSB formation emphasized the Gibbs free-energy evolution as a function of loading cycles [89, 90]. During loading, the change in Gibbs free-energy increases, reaches a maximum value, and then sharply declines. The decline of the Gibbs free-energy is attributed to the lower energy of the dislocations in the wall of the PSB. This type of model correctly predicts the change in the ease of crack initiation due to atmospheric pressure, temperature, and proximity to a free surface. When slip irreversibility is accounted for, the Gibbs energy model agrees with experimental data [91]. This model does not take into account the normal stress or dislocation structures, but instead simply consists of an energy balance. Another model developed by Sauzay and Vor [92] examined PSBs utilizing Crystal Plasticity Finite Element Analysis (CP-FEA) to study the energy distribution of a PSB. This model provides energy figures not given by the earlier dislocation pile-up based theories.

In summary, both physically-based and energetically-based models have been used to examine the dislocation evolution due to cyclic loading. These models have been used both to describe the
dislocation evolution and predict the materials response to such loadings, with a particular focus on predicting how the microstructural evolution can lead to crack initiation.

2.7 Alloy and Planar Slip Material Responses to Cyclic Loading

Earlier descriptions of dislocation structures are primarily applicable to pure wavy-slip FCC metals. The microstructural evolution within wavy-slip alloys is broadly similar. In particular, the existence of PSBs has been observed in wavy-slip FCC alloys such as austenitic steels [39] and γ’ strengthened Ni-based superalloys [93].

Planar slip materials, the most studied of which are Cu-16%Al and Cu-30%Zn, have different hardening behavior and a different microstructural evolution in response to cyclic loading [94]. During the early stages of cyclic deformation, planar slip materials do not form the dipole-dipole structure nor do they form the loop patch and vein structure. Instead, dislocations form planar arrays upon initial hardening. These planar arrays increase in both numerical density as well as dislocation density as cyclic hardening occurs. Additionally, in planar slip materials, secondary slip initiates at lower plastic strain amplitudes than in wavy slip materials [40]. As discussed above, planar slip materials do not form PSBs, but instead form Planer Lüder’s Bands (PLB) [95-97]. A PLB is a localized slip phenomenon of an intense planar array of dislocations [94]. Unlike PSBs, PLBs are mobile, initiating sequentially across the sample. The lack of a distinct dislocation microstructure and the transient nature means that PLBs are difficult to image ex-situ.

2.8 Crack Initiation and the Role of Strain Localization

Crack initiation is a complex phenomenon with multiple possible sources: surface roughness, grains boundaries, and the interaction of PSBs with grain boundaries. Most of the recent work toward understanding crack initiation has revolved around understanding the role that localization of strain plays in crack initiation.
2.8.1 Crack Initiation from Surface Roughness

Localization of strain, the concentration of strain in certain areas, plays an important role in the initiation of surface fatigue cracks. Work by Seeger [98] highlighted that cyclic work hardening of materials would naturally result in slip band formation due to cross-slip around sessile locked dislocations. The intersection of the slip bands with the surface has been treated as an area of a strain localization. Slip band-surface interactions can result in the formation of an extrusion, pushed out from the surface by the effects of dislocations on the slip band, or an intrusion, where the surface has a divot at a slip band. A schematic of extrusions and intrusions at a free surface can be seen in the Figure 2.4 [99].

![Figure 2.4: A schematic showing the formation of extrusion and intrusion due to the actions of slip bands. [99]](image)

Two separate interactions have been proposed to account for how fatigue cracks initiate at extrusions and intrusions. The first focuses on the role that extrusions/intrusions play as stress concentrators. Déprés et al. [100] utilized a discrete dislocation dynamics model to examine the stresses surrounding a extrusion that formed due to the action of a PSB. They proposed that a microcrack initiated from an extrusion when a critical stress was reached. Two different events are required for this. First, the extrusion must have grown large enough for there to be a critical stress field surrounding the extrusion. Second, a large dislocation multipole (analogous to the ladder configuration in a PSB) must travel up the PSB and be absorbed by the free surface. The wake of the multipole will create a large, highly triaxial stress concentration at the extrusion, resulting in the cleavage of the PSB slip plane and generating a microcrack.
The second proposed mechanism for crack initiation at extrusions/intrusions relies around the build-up of irreversible plastic strain, i.e. the strain that causes changes in the microstructure during each cycle. The height of the extrusion has been examined by atomic force microscopy to provide insight into the amount of plastic slip irreversibility of the PSB beneath extrusions [101-103]. It has been found that there is a critical height necessary for crack initiation. This critical height is material-specific, but is independent of the grain size. The exact mechanism by which the crack initiates after the critical extrusion height is reached remains unclear. One potential mechanism was proposed by Repetto and Ortiz [104], wherein the dislocation generation and annihilation within the PSB produces vacancies during the annihilation process. This results in a net flux of vacancies along the walls of the PSB to the surface. The vacancies then interact with the surface causing it to recede, producing a sharp crack in the surface.

2.8.2 Crack Initiation from Grain Boundaries-PSB Interactions

Intersections of PSBs and grain boundaries have been observed to act as potential fatigue crack initiation sites. The size of grains has been shown to influence crack initiation as a smaller grain size suppresses PSB formation [62, 63], preventing crack initiation due to a lack of PSBs. However, multiple small grains separated by low angle grain boundaries can act as a single larger grain for PSB formation, negating some of the grain size effect [105]. The type of grain boundary involved also plays a role, as low angle grain boundaries [105] and grain boundaries with high coincident site lattice values (i.e. low Σ values) are not observed to crack [106], with the exception of twin boundaries which will be discussed in Section 2.8.3.

Three different mechanisms of fatigue crack initiation at grain boundaries have been proposed. The first is based on the strain and strain energy at a specific point on the grain boundary with a critical strain energy necessary for crack initiation. The second is based around stress and focuses on how stress concentrators near grain boundaries lead to strain localization and crack initiation. The third is focused on quantifying the energy associated with the entirety of the PSB-grain boundary interaction.

Recent techniques have been developed to enable high resolution strain mapping such that the value of the isolated strain can be measured, the development of which has led to the proposal that
localization of strain at the grain boundary plays a key role in crack initiation. Tschopp et al. [107] used EBSD in connection with in situ SEM Digital Image Correlation (DIC) to create a map of strain values, sorted by the Schmid factor, of the grains for fatigued polycrystalline René 88DT, a nickel-based superalloy. Tschopp et al. found that the shear strain increased with an increasing Schmid factor and that the range of maximum shear strain increased as grain boundaries were approached, indicative of strain localization to a few grain boundaries. Abuzaid et al. [108] built on the prior work by utilizing high resolution DIC imaging accompanied by EBSD. EBSD was used to create a high resolution orientation map of the underlying grain structure. While the overall strain was 2%, some grain boundaries showed strains of up to 5%, more than double the overall strain. Grain boundaries that had accumulated residual dislocations with large Burgers vectors were found to have higher local strains, suggesting grain boundaries with a higher energy of strain transfer are more prone to crack initiation.

Modeling the formation of cracks at grain boundaries, Dunne et al. [109, 110] used a CP-FEA model to study fatigue crack initiation in C263, a γ’-strengthened nickel superalloy, by incorporating a back-stress term within the model to account for the cyclic loading. Dunne et al.’s model was able to account for geometrically necessary dislocations (dislocations which result in a net change in crystal orientation due to the accumulation Burgers vectors; this definition excludes dislocation structures which have a net Burgers vector of zero, e.g. dislocation dipoles) and microstructural effects by taking the curl of the strain gradient tensor. This allows for the density of geometrically necessary dislocations to be tracked during cyclic loading. Tracking the geometrically necessary dislocation structures allows for changes in the plastic velocity gradient to be tracked. Thus, the loading conditions on individual slip systems within the crystal were tracked, and the slip systems were observed to activate upon reaching the critical resolved shear stress. In this model, the failure condition, i.e. crack nucleation, was achieved when the accumulated amount of slip reached a predetermined level. This has led to the proposed mechanism that interaction between PSBs and grain boundary triple points produces the highest localized strain and leads to crack initiation.

The second proposed mechanism described how stress concentrators lead to higher localized slip near grain boundaries. Dunne et al. [111] created a discrete dislocation dynamics-crystal plasticity model which was compared to EBSD results. When examining the interaction of a carbides stress
field with the dislocation generated under cyclic loading in a nickel sheet, they found that high stresses drove the strain localization around the carbide. Guilhem et al. [112] showed similar results in a CP-FEA model of 316L, finding that strain localization occurred due to the long-range stresses that grain boundaries exerted on each other. Miao et al. [113] showed that grains that were abnormally large, and thus subjected to higher stresses, were more likely to initiate fatigue cracks at Σ3 twin boundaries in the Ni-base superalloy Rene’ 88DT.

A CP-FEA model was constructed by McDowell et al. [114] to investigate crack initiation at intermetallic particles in a 7075 aluminum alloy. The model was used to determine the stress distribution around an intermetallic Fe-rich particle with respect to the local microstructure, including the orientation differences between the particle and the parent grain, the particle size and shape, slip localization, and the effects of cyclic loading. The crack nucleates when the stress within the particle exceeds the strength of the particle.

A thorough investigation in grain boundary crack nucleation was conducted by Sangid et al. [115, 116], with the goal of having a complete understanding of the relationship between the energy involved in grain boundary-PSB interactions and the nucleation of a crack in nickel-based superalloys. The model utilized an energy balance to track the evolution of a PSB-grain boundary interaction. The total energy of the interaction was divided into five components. The first is the PSB formation energy, which is composed of the energy needed to shear the γ and γ’ phases and that required to accommodate newly formed antiphase boundaries and stacking faults. The second is the energy expended due to work hardening. As multiple dislocations shear the same particle, the energetic barriers to shearing lower, encouraging the dislocations to share a slip plane. This causes dislocation accumulation and interaction on that slip plane. The third component relates the energy associated with applied stress on the PSB through a Schmid factor relationship.

The two other components were computed using atomistic molecular dynamics (MD) simulations to understand, at the atomic level, the energy necessary for dislocation emission and absorption during PSB-grain boundary interactions. The energies were identified by examining dislocation grain boundary interactions using a bicrystal MD straining experiment to get the energy necessary for dislocation absorption and emission. These energy barriers change during the interaction as the density of dislocations changes and ledges form on the grain boundary. Understanding the different components of the energy involved in the grain boundary-PSB interaction and how these
components change during loading allows the model to accurately capture the energetic evolution during cyclic loading. Crack initiation occurred when the PSB became energetically stable, i.e. the total energy of the PSB-grain boundary interaction reached a minimum. This criteria is consistent with both the Griffith fracture criterion and the ductile-to-brittle condition developed by Rice and Thompson [117]. Physically, this failure criterion manifests when the dislocations within the PSB are in a low energy configuration and crack nucleation is energetically preferred over dislocation generation.

In summary, models of the mechanism of crack initiation from grain boundaries fall into three categories: those with stress-based criteria; those with strain-based criteria; and those based on energetic considerations. Under the stress-based theory, stress concentrators create high localized stresses, which in turn drive the development of plastic strain. In the strain-based theories, crack initiation is correlated to the incidence of grain boundaries oriented for dislocation slip transfer interactions that leave dislocations in the grain boundary with a large Burgers vector. The energy-based theories examine how the energy changes over the course of the PSB-grain boundary interaction to find a point where cracking is energetically preferred to dislocation slip.

### 2.8.3 PSB – Twin Boundary Interactions

Generally, high-coincident-lattice site grain boundaries do not serve as sites for crack initiation. However, an exception comes in the form of twin boundaries, which have been shown to be preferential sites for crack nucleation [118]. This interaction is most often attributed to the orientation relationship between twin boundaries and PSBs. Since PSBs are often perpendicular to twin planes, the PSB-twin boundary interaction will introduce many out-of-plane artifacts on the twin boundary including non-coherent steps, ledges and extrusions on the twin boundary [119, 120]. These boundary defects act as stress concentrators and dislocation sources, eventually becoming preferential sites for crack initiation. Planar-slip materials are more susceptible to crack initiation as the dislocation-twin boundary in planar-slip materials creates further, secondary slip, which increases the amount of dislocation content within the twin boundary [120, 121].

The stress field surrounding twin boundaries has been investigated to determine its role in fatigue crack nucleation. Kim and Laird examined how surface slip traces transferred across twin boundaries in copper, finding that the plastic anisotropy inherent to the twin boundary would
localize the plastic strain to a small number of slip bands, leading to crack initiation [122]. Peralta et al. [123] examined the elastic stress concentrations on an idealized twin boundary in different orientations, finding that, regardless of the loading orientation, significant elastic stresses would develop on at least one side of the twin boundary. These stress concentrations led to a localized increase in primary and secondary slip near the twin boundary, which led to fatigue cracking. Finite element models of the PSB-twin boundary interaction show similar elastic stress concentrations as Peralta’s model [124, 125].

2.9 Microstructurally Small Crack Growth

The previous sections dealt how crack initiation occurs in metal alloys. Such initiation is followed by the MSC growth regime. Cracks are said to be microstructurally small when the length of the crack is comparable to microstructural features, i.e. the grain size. The MSC regime is distinct, as the crack growth rate is much smaller than extrapolations from the large scale crack regime, due to the difference in crack growth mechanism. Traditional models of MSC growth show the crack initiating at surface roughness and propagating on the planes of maximum shear, i.e., 45° from the loading direction. Recent studies of MSC have revealed it to be a complex phenomenon that dominates material lifetime during high cycle fatigue. This section will provide an overview of the current understanding of MSC, which has also been the subject of a recent review by Pineau et al. [126].

MSC growth has its roots in crack nucleation. If the crack nucleates from surface roughness, i.e. extrusions and intrusions on the surface, it will then grow along the PSB away from the surface [127]. Since PSBs with the highest dislocation activity will be along the slip plane with the highest Schmid factor, cracks tend to grow 45° from the loading orientation.

Crack growth along PSBs is highly susceptible to the influence of grain boundaries. Ferrite grain boundaries and grain boundary triple points in a low carbon BCC steel were found to decrease the crack growth rate and cause irregular crack growth as the grain boundary was approached [128]. Additionally, prior-austenite grain boundaries were shown to have a similar effect [129]. When the crack reached sufficient length, approximately three times the average grain diameter, the growth rate was no longer affected by grain boundaries as obstacles.

In other materials crack growth occurs not along PSBs but along certain crystallographic features. Miao et al. [113] found that MSCs in Rene’ 88DT initiated near, then propagated parallel to, Σ3
annealing twin boundaries. A consequence of this was that cracks could only cross other grain boundaries when there was a small misorientation (<20°) between the grains. Larger misorientations would result in crack arrest. In a duplex phase, i.e. one that has both HCP and BCC components, Ti-6Al-4V alloy crack growth occurs with the formation of α-phase crystalline facets, with arrest or propagation determined by the mode of deformation involved in facet formation [130].

To monitor the growth rate of MSCs, several different techniques have been used. High energy X-ray diffraction microscopy can be used to map the microstructure, including the size and orientation of grains. This technique is then used in conjunction with X-ray diffraction contrast tomography, also known as microCT, to monitor the three-dimensional evolution of crack tips. This approach allows for the crack growth rate, the crack morphology, and the shape and orientation of the grains to be monitored [131-133]. This technique was performed on a BCC β-titanium alloy to monitor the crystallographic orientation of the fracture surface during crack growth. The resolution was limited to 5 µm segments with smaller lengths interpolated. Crack growth preferentially occurred on \{101\} planes, even if they were not experiencing the highest Schmid factor. When other planes were aligned with the highest Schmid factor, crack growth would occur on alternating \{101\} planes to achieve growth in the preferential plane.

In summary, microstructurally small crack growth can be influence by various microstructural factors including grain size, orientation and the dislocation microstructure. Many cracks will initiate in the material and grow via MSC growth. This regime ends when one of the multiple cracks grows large enough to enter the long crack growth regime.

### 2.10 Long Crack Growth and the Paris Law

Long crack growth is different from MSC growth in several ways, including the mechanisms of crack growth, the direction of crack growth, and the crack growth rate. During long crack growth, the crack propagates normal to the applied loading direction and the crack growth rates are tied to the stress intensity factor. The specific mechanisms of crack growth are discussed in section 2.16.

The correlation between the stress intensity factor and the crack growth rate was first described by Paris in 1963 [134, 135]. The stress intensity factor is a variable that describes the stress around a
crack tip in linear-elastic fracture mechanics and takes the form of \( K = Y \sigma \sqrt{a} \), where \( K \) is the stress intensity factor, \( Y \) is a geometric adjustment, \( \sigma \) is the applied stress, and \( a \) is the length of the crack. If the crack is under linear-elastic fracture mechanics conditions and the small scale yielding constraint is applicable the stress intensity factor will describe similar crack behaviors despite difference in geometry, stress, and crack length.

Paris [134, 135] extended the concept of the stress intensity factor to account for the cyclic loading by examining the change in the stress intensity factor from maximum load to the minimum load, annotated as \( \Delta K \). This means that \( \Delta K \) is the important stress intensity for determining crack propagation rates in linear elastic fracture mechanics. Under elastic-plastic fracture mechanics the \( \Delta J \) integral, which is a measure of the of the energy release rate, provides a description of the crack tip strain. Under linear-elastic conditions the \( \Delta J \) integral is related to the \( \Delta K \) value by \( \Delta J = \frac{\Delta K^2}{E'} \), where \( E' \) is the effective Young’s modulus which relates to the Young’s modulus as:

\[
E' = E \quad \text{for plane stress} \\
E' = \frac{E}{1 - \nu^2} \quad \text{for plane strain}
\]

where \( E \) is Young’s modulus and \( \nu \) is Poisson’s ratio.

Paris examined how changing the \( \Delta K \) level would affect the crack growth rate. Among the findings was that below a certain level of \( \Delta K \), called the threshold \( \Delta K \) and notated as \( \Delta K_{TH} \), fatigue cracks will not propagate. The threshold stress intensity is a material constant for similar environmental conditions.

How the propagation of fatigue cracks varies with \( \Delta K \) is shown in Figure 2.5 [4]. Here the logarithm of \( \Delta K \) is compared to the logarithm of change in crack length \( (da) \) per cycle \( (dN) \). There are three important stages that occupy this schematic. The first stage begins at \( \Delta K_{TH} \) and is characterized by a decrease in \( da/dN \). This occurs gradually until the sample enters into the second or power law stage. Here the rate of fatigue crack growth is determined by the equation

\[
\frac{da}{dN} = C \Delta K^m \quad \text{where} \ C \ \text{and} \ m \ \text{are material constants. This stage occupies most of the lifetime of fatigue crack propagation. Stage III fatigue crack growth is the final stage occurring at high } \Delta K
\]
levels. Stage III crack growth is characterized by a rapidly increasing rate of fatigue crack propagation. As the ΔK level begins to approach the inherent fracture toughness of the material, the fatigue crack growth mechanism will become mixed with alternate crack growth mechanisms including possible microvoid coalesce or cleavage type fractures. This deviation becomes more severe the closer the maximum K is to fracture toughness of the material, as more mixed mechanism crack growth occurs.

![Figure 2.5: Representative curve of the stress intensity factor versus crack growth rate](image)

In addition to the stress intensity factor, the local microstructure can play a role in determining the rate of crack propagation. Grain size can play a strong role in determining fatigue crack propagation, especially near the threshold stress intensity factor. When the grain size is smaller than the plastic zone, the dislocations generated by the crack tip will encounter the grain boundaries as an obstacle. Upon encountering grain boundaries, strain transfer and secondary slip will be initiated. Such slip is irreversible creating a larger irreversible plastic zone surrounding a fatigue crack tip. The effect of this is to lower the ΔK_{th} as the grain size decreases [136].
Slip character is expected to play a significant role in determining the crack growth rate via the influence on the reversed plastic zone. Wavy slip materials have relatively homogenous deformation, causing dislocations to cross-slip and become locked, limiting the reversal of slip. Conversely, planar slip materials tend to confine deformation into slip bands. The slip bands do not feature extensive cross-slip, making slip reversal easier to achieve. Therefore, planar slip materials tend to show lower crack growth rates due to the smaller reversed plastic zone size [137].

2.11 The Plastic Zone and the Reversed Plastic Zone

The plastic zone surrounding a fatigue crack is a product of the stress concentration of the crack and influences the rate of crack growth as detailed in Section 2.12. Figure 2.6 [1] shows the evolution of the plastic zone at two different points in cyclic loading. Figure 2.6a shows the loading cycle, with the dots labeled 1 and 2 indicating the evolution of the stress intensity factor over time. Figure 2.6b shows the sample at position 1, the maximum stress. The graph shows the stress as the crack tip is approached. Theoretically an infinitely sharp crack tip acts as an infinitely strong stress concentrator. In actuality, the stress does not exceed the yield stress, labeled \( \sigma_y \). This creates an area of plastic yielding ahead of the crack tip. Theoretically the size of the plastic zone is given by the equation 2.1 and 2.2 for plane stress and plain strain respectively:

\[
\begin{align*}
    r_y &= \frac{1}{2\pi} \left( \frac{K}{\sigma_y} \right)^2 \quad \text{plane stress} \\
    r_y &= \frac{1}{6\pi} \left( \frac{K}{\sigma_y} \right)^2 \quad \text{plane strain}
\end{align*}
\]

where \( r_y \) is the radius of the circular plastic zone, \( K \) is the stress intensity factor, and \( \sigma_y \) is the yield strength. An assumption inherent to linear elastic fracture mechanics and theoretical plastic zone sizes, presented in the equations 2.1 and 2.2, is that the plastic zone is homogenous. This is not true for real polycrystalline materials. High strength materials and grain boundaries can lead to substantial amounts of strain localization, further complicating the idea of the plastic zone [138].

The plastic zone size changes when the stress intensity factor is lowered, as seen in Figure 2.6c. When the tensile load is removed or lowered, the elastic material surrounding the plastic zone will force the plastic zone to conform to its original shape. This introduces residual compressive
stresses throughout the original plastic zone, the dotted line in Figure 2.6c, and forms a compressive plastic zone at the crack tip, the dashed region in Figure 2.6c. Theoretically, the size of the reversed plastic zone is given by equations 2.3 and 2.4 for plane strain and plane stress respectively:

\[
    r_y = \frac{1}{8\pi} \left( \frac{K}{\sigma_y} \right)^2 \quad \text{plane stress} \tag{2.3}
\]

\[
    r_y = \frac{1}{24\pi} \left( \frac{K}{\sigma_y} \right)^2 \quad \text{plane strain} \tag{2.4}
\]

While theoretically the size of the reversed plastic zone is exactly 25% of the size of the monotonic plastic zone, more recent investigations have indicated that it is only 10-15% [139].

![Figure 2.6](image)

Figure 2.6: Illustration of the plastic zone and reversed plastic zone; a: details a representative loading curve with the point of highest K labeled ① and the lowest loading labeled ②; b: a representation of the stress as infinitely sharp crack is approached at the highest load. The area higher than the yield stress, a.k.a. the plastic zone, is shaded; and c: a representation of the yield stress at the lowest load. The plastic zone from b is highlighted by the dashed line, while the area compressively yielding is shaded. Image courtesy of Kelly Nygren utilizing data taken from [1].

### 2.12 Plasticity Induced Crack Closure

An important modification to the Paris Law arises from the crack closure mechanism. Crack closure refers to the phenomenon in fatigue crack growth wherein the crack will remain closed even under tension until a sufficient \( K_I \) is reached. Since the crack is unable to propagate when closed, the effective stress intensity factor is the applied \( K_I \) minus the \( K_I \) necessary for crack opening (\( K_{IO} \)). Since the stress intensity factor necessary for crack opening is a constant, the effects of crack closure are felt most strongly at near-threshold \( \Delta K \) levels, where the effects of crack closure can produce significant deviations from the measured crack growth rate. The mechanisms and theories have been extensively reviewed by Ritchie [140] and include roughness-induced
crack closure, oxide-induced crack closure, and plasticity-induced crack closure. Plasticity-induced crack closure deserves special attention in this review.

Plasticity-induced crack closure was first documented by Elber [141]. Plasticity-induced crack closure arises from the compressive stresses of the reversed plastic zone. Understanding the size and shape of the plastic zone in front of crack tip is critical to accurately measuring $\Delta K_{\text{IO}}$ and the crack growth and lifetime. Methods for measuring the plastic zone and the reverse plastic zone, as well as accurately simulating it, can be found in Section 2.13.

The effect of plasticity-induced crack closure can be seen in the differing growth rates between Physically Small Cracks (PSCs) and long cracks. PSCs are defined as having a plastic zone that is smaller than the microstructure features (i.e., grain size). PSC are different from MSCs, where the crack length is on a comparable scale to the microstructural features, PSC have a plastic zone size comparable to microstructural features. PSCs grow by the same processes as long cracks, but have higher crack growth rates at comparable stress intensity factors. Since the plastic zone is smaller for PSCs than long cracks, increased plasticity induced crack closure accounts for the difference in the two crack growth rates.

2.13 Measuring the Plastic Zone
Digital Image Correlation allows for the evolution of fatigue damage to be monitored in situ [108, 142-144]. Bartali et al. [145] monitored in situ crack growth to examine the accumulated plastic deformation in the different phases of a duplex stainless steel (one with both BCC and FCC components), finding that strain damage first accumulated in the austenite grain before transferring to the ferritic grains. Carroll et al. [142] monitored the plastic zone ahead of a growing fatigue crack in Hastelloy X, finding that the plastic strain was organized into two lobes oriented 40° from the direction of crack advance. The lobes were found to alternate in operation, i.e. the upper one would activate during a portion of the crack advance with little to no activity noted in the lower band. This activity pattern would then change to the other lobe; the change appeared to be random. Both a total change where no concurrent activity was noted, and a period where both lobes were active was observed. The damage within the was found to be inhomogeneous both between the upper and lower lobes and within the lobes themselves. Such inhomogenieties extended below the grain size and were attributed to microstructural features including grain size and orientation.
Additionally, no strain was noted directly ahead of the crack tip, at least to within one grain size (20 µm). Other studies have found similar strain lobes, with most showing simultaneous operation of multiple slip systems [144, 146]. While the inhomogeneities were primarily attributed to the microstructural features, how the dislocation structure changed in response to these features and the stress state was not noted.

### 2.14 Simulations of the Plastic Zone

Physically based models of crack closure, and the experimental evidence has been supplemented by Finite Element Analysis (FEA) models. Such models are limited to exploring the effects of plasticity-induced closure, as roughness-induced and oxide-induced crack closure are unable to be accounted for in FEA models. FEA models of crack closure have been created to solve both the two-dimensional problem as well as the three-dimensional problems.

Both two-dimensional and three-dimensional models follow the same general formulation. First, a mesh is created to simulate the sample of interest, typically some type of cracked specimen geometry. Next, an alternating load is applied to the mesh to simulate the cyclic loading. During the tensile portion of this load, the crack will advance by an increment \( \Delta a \) that is equal to the element size immediately ahead of the crack tip. This incremental advance is accomplished by decoupling the nodes of the element ahead of the crack. Finally, the strain on the elements surrounding the crack is analyzed.

The most common method of advancing the crack is simply to release the node at the crack tip regardless of any condition. Alternate crack advance schemes have attempted to tie the release of the crack tip node to physical features including a critical strain criterion [147], a critical stress criterion [148], or the use of cohesive elements that will fracture in a physics based manner [149].

Nodal release schemes are also varied in the manner of execution, affecting accurate measurements of crack closure. Different nodal release schemes see the release occurring at the minimum stress [150, 151], the maximum stress [151], following the maximum stress [152], or during the loading/unloading cycle [153], with release at the maximum or minimum being the most common. McClung et al. [150] investigated the difference between nodal release at the maximum and minimum stress conditions and concluded that there is little difference on which is used, whereas
Wu and Ellyin [151] suggested that convergence issues arise when using the maximum nodal release scheme. Additionally, Wu and Ellyin [151] found that crack tip opening displacement will vary based on the nodal release scheme used, with the displacement being significantly higher when the crack is advanced at the maximum load.

Since the advancement of the crack is explicitly linked to the size of the finite elements immediately ahead of the crack, proper discretization of the mesh must be used to ensure the crack growth is reasonable for the simulated applied load. If the mesh is too coarse, then the crack tip opening displacement will be too large, leading to larger forward and reverse plastic zones than would be expected [154]. This constraint has led researchers to create models with large applied loads so that coarser mesh sizes can be used, as coarser meshes are less computationally intensive. Smaller applied loads, with correspondingly finer meshes, have demonstrated that the crack tip opening displacement was primarily dependent on mesh size [155, 156].

Another constraint demonstrated by McClung et al. [157] is that accurate depiction of the forward and reverse plastic wakes does not occur immediately. As crack growth is a steady state process during this time, the plastic zone around the crack tip is an accumulation of the plastic slip from multiple cycles. Therefore, forward and reverse plastic wakes will only stabilize once the crack tip has grown completely out of the initial plastic zone. This technique is necessary, but is computationally intensive, as several cycles of crack growth need to be simulated for an accurate measurement of the plastic zone size.

Another consideration of these models is the compressive forces of the reverse plastic zone. During the compressive portion of the load cycle, the two crack flanks will meet and compressively yield if the closure stress is high enough. Without a constraint to simulate the resistance of the crack faces to movement between each other, the unbonded finite element nodes would pass through each other. To simulate the resistance of the crack flanks, Newman et al. [154] utilized a spring truss model. Such a model monitors the nodes along the crack path to determine whether they should be open, closed, or released. The model then applies a strong spring force, several orders of magnitude higher than the elastic constants, to the nodes to hold the nodes ahead of the crack closed and prevent the already released nodes from overlapping during contact. When the model determines that the nodes should be open, it reduces the spring force to zero. Crack advance is
achieved by changing the spring force to zero and releasing the node. This method is less computationally intensive than other methods and has been adopted by many [150-152, 155-159].

The choice of the two-dimensional stress state can have a significant effect on the FEA models. Both plane stress or plane strain have been used, with the chosen strain state having a significant effect on the FEA model. The plastic deformation of the material ahead of the crack requires the transfer of material from elsewhere in the material. In the plane stress case, the out-of-plane strain can allow for material transfer from the thickness to the height of the material. This mechanism is, by definition, impossible under plane strain loading, as no out-of-plane strain exists [150, 160, 161]. Some have claimed that this lack of an obvious material transfer mechanism implies that plasticity-induced crack closure is impossible under plane strain conditions [161]. This finding is refuted by the many studies that show that crack closure occurs under both plane stress and plane strain, with the plane strain sample having a smaller reverse plastic zone size and a lower K10 stress intensity factor [152, 159, 162]. To investigate the effect of the specimen geometry on crack closure in fatigue loading, Fleck and Newman [163] investigated both an M(T) specimen and three-point bend specimen in plane strain conditions, finding that crack closure occurred in the M(T) specimen, but not in the three-point bend specimen. The difference was attributed to the compressive T-stress with the M(T) specimen, while the three-point bend specimen had a tensile T-stress.

The R-ratio also plays a role in the amount of plasticity-induced crack closure. Solanki et al. [155] investigated the effect using an M(T) specimen geometry, finding that crack closure was lessened at a higher R-ratio as a smaller reversed plastic zone occurred around the crack tip.

Three-dimensional FEA models of fatigue crack growth and plasticity-induced crack closure [164, 165] have also been created. Three-dimensional models are more computationally intensive than two-dimensional models, but have similar issues, including mesh refinement and the crack advance scheme, that need to be resolved for accurate modeling of crack growth. The crack advance scheme is particularly important for three-dimensional FEA models as three-dimensional crack growth is tortuous due to various areas of the crack front growing at different rates and angles. Thus, the crack front will continuously evolve during the loading resulting in varying levels of crack closure along the crack front.
In summary, plasticity-induced crack closure and the plastic zone have been extensively studied via finite element models. Crack closure has been shown to occur in plane stress, plane strain and three-dimensional models. Most of these models are based on mesoscale continuum mechanics, and make no assumptions about the underlying dislocation structures. Understanding dislocation structures within the crack tip plastic zone could lead to more accurate model development.

2.15 Striation Properties
The most prominent feature on the fracture surfaces of fatigue cracks are striation marks. Striation marks appear as a series of ridges on the fracture surface oriented perpendicular to the crack growth direction and parallel to the crack front. Striations have been observed on the fracture surfaces of BCC, FCC, and HCP metals and alloys, as well as engineering plastics [1]. Striations can have a variety of heights and appearances: from nearly flat to large and ductile. While a complete understanding of the factors governing striation morphology is elusive, it has been noted that within oxidizing environments flatter striations are more common, while ductile striations are more common in inert environments [166]. Additionally oxidizing environments are known to eliminate striation markings over time following fatigue crack growth.

Striations markings are not the only feature seen on the fracture surface. While common during FCG at intermediate \( \Delta K \) levels, striations are less common at both low and high \( \Delta K \) levels. At high \( \Delta K \) levels microvoids begin to appear and dominate the fracture surface [167]. At lower levels of \( \Delta K \) faceted cleavage-like markings begin to dominate [168].

Multiple theories have been proposed to account for FCG and the appearance of striations, however there is disagreement on the exact nature of cyclic loading, striation formation, and crack advance. One common assumption is that each loading cycle produces a single crack advance event that leaves a single striation on the surface [169-171]. This relationship forms the basis for some of the theories of fatigue crack propagation. Forsyth and Ryder [170] investigated the relationship between striations and the loading cycles by applying periodic overloads to an aluminum alloy. Since the overloads were visible on the fracture surface, the number of striations between overload events could be counted and compared to the number of loading cycles between the overload events. Forsyth and Ryder found that there was a strong agreement between the number of loading cycles and the number of striations found on the surface. A more recent study
utilized in situ X-ray tomography to compare striation spacing to the measured rates of crack advance, finding strong agreement [172]. However, while this link may be true for some loading conditions, it does not hold in all circumstances [173].

Striations have been shown to have a minimum size, between 120 nm and 150 nm, and at low ΔK levels, the crack growth per cycle can fall under the minimum observed striation size [174, 175]. This has led to two separate proposals about crack growth under these conditions. The first, suggests that the crack growth and striation formation remain linked. Under this theory, a crack would no longer grow incrementally during each cycle, but instead through bursts of crack growth [174]. The second proposed interaction suggests that crack advance continues incrementally with each loading cycle. If this was the case, then the formation of the striation markings would be divorced from both the cyclic loading and the crack advance [176].

Another factor in striation formation are crystallographic conditions. Striations have been shown to preferentially form on \{111\} slip planes in aluminum when crack growth is parallel to the <110> direction [177]. This agrees with macroscale observations suggesting that fracture planes are parallel to the \{110\} or \{100\} planes [178]. This suggests that intense striations appear on preferentially oriented grains while striations will be weak or nonexistent on non-preferentially oriented grains. In addition to the intensity of the striations, the size of striations varies based upon the underlying grain orientation [179]. Even within the same grain striation size can vary, suggesting that the macroscopic crack growth rate is an average of various microscopic crack increments indicated by the striations spacing.

In summary, striation formation is a complex phenomenon influenced by the crystallography of the grains, the crack growth rate, the environment, and the ΔK. Theories as to the specific mechanisms governing the fatigue crack propagation must account for striation formation and the array of factors that can influence the spacing.

2.16 Fatigue Crack Propagation in the Paris Law Regime

The classical mechanism for long fatigue crack growth was proposed by Laird and Smith [169]. The crack growth is based on physical deformation processes that occur at the crack tip under cyclic loading. This proposed mechanism equates a loading cycle with the formation of a single
striation and a singular unit of crack advance. Figure 2.7 illustrates this process. When a crack is at zero load, as in Figure 2.7a, the crack is closed. As the load increases the crack begins to open and the tip begins to deform to accommodate the crack opening. This deformation is accompanied by the emission of dislocations along the planes of maximum shear oriented ±45° for the crack growth direction, as depicted in Figure 2.7b. At the maximum loading the crack tip has fully opened with dislocations at the crack tip creating additional surface to accommodate this blunting; Figure 2.7c depicts the fully opened crack tip. On changing to a compressive load, the direction of slip is reversed. This causes the crack tip to fold inward as depicted in Figure 2.7d. This creates a sharp crack for the next loading cycle, as seen in Figure 2.7e, which begins to blunt open again, as seen in Figure 2.7f. The striation markings are created during the unloading and compressive portion of crack growth where the crack tip folds in upon itself.

Figure 2.7: The Laird and Smith model for ductile blunting: a: a sharp crack at zero load; b: the crack opening in response to the applied load; c: the crack fully opened at the maximum load; d: the following the maximum load; the crack tip has folded in on itself creating striation marks; e: the crack tip at the maximum compressive load and f: the crack tip opening in response to the applied load in a second cycle. This is similar to b however, the crack has advance by a set distance. Image courtesy of Kelly Nygren, using data taken from [180]

Unfortunately, the assumption inherent to the Laird and Smith model, that one loading event results in a single crack advance event and the formation of a striation, is not valid across all ΔK levels. At low ΔK levels, it may take many loading cycles to advance the crack. In a 2024 aluminum alloy, Davidson and Lankford [173] found that it took up to 43 loading cycles to advance the crack one striation spacing. Thus, the fully geometric conditions predicted by the Laird and
Smith model of the blunting and resharpening at the crack tip may be accurate at high $\Delta K$ levels, but do not fully capture the complexity at lower $\Delta K$ levels.

An alternative way that the ductile blunting mechanism could be accomplished was proposed by Pelloux [166] and Neumann [181] and observed in a variety of single [181] and polycrystalline materials [182-184]. This so called unzipping mechanism is shown in Figure 2.8 [185]. The ductile blunting here is accomplished by an unzipping decohesion of the crack tip. Figure 2.8a shows a sharp crack tip in the material. A number of slip bands are present oriented 45° from the direction of crack growth. These slip systems are numbered 1-7. Upon loading, the crack tip moves along the slip band labeled 1 by a distance equal to the spacing between the slip bands, as seen in Figure 2.8b. Consequently, the crack tip will advance along the slip band labeled 2, oriented 90° from the first slip band, once again advancing by one slip band spacing, Figure 2.8c. This continues on alternating slip bands until the crack fully blunts, shown schematically in Figure 2.8d.

![Figure 2.8](image.png)

**Figure 2.8**: A diagram of the Neumann model for fatigue crack growth. The crack and slip bands oriented for crack growth are shown, with the slip bands labeled 1-8 in the order in which decohesion events occur: *a*: the sharp crack tip in the material; *b*: the crack opens via a shear decohesion along the slip band labeled 1; The total distance it opens is equal to the spacing between slip bands; *c*: the crack has a shear decohesion event on the slip band labeled 2; *d*: the crack tip after two more decohesion events occur on slip bands 3 and 4 [185].

In a polycrystalline material, if the slip bands were not precisely aligned with the direction needed for the sliding, such action could be accomplished through the actions of multiple slip bands operating to produce the same desired movement. This could partially explain the dependence of striations on the underlying grain orientation [185].

Support for the ductile blunting mechanism, whether based on crack tip deformation or the shear sliding mechanism, comes from various studies. McClintock [186] examined the Crack Tip
Opening Displacement (CTOD) during fatigue crack growth of copper at high $\Delta K$ levels and demonstrated that under the plastic blunting model the crack growth per cycle should be equal to half the CTOD. McClintock [186] found strong correlation between the CTOD and striation spacing in copper. Davidson and Lankford [173] investigated this connection in an aluminum alloy, finding that when tested in an air environment, the striation spacing correlated with the CTOD, but when tests were performed under vacuum, the striation spacing showed a correlation with the effective $\Delta K$. This change was attributed to the differing levels of plasticity surrounding the crack tips in air versus vacuum.

Other studies have argued that there is no correlation between the CTOD and striation spacing. Liu and Kobayashi [185] compared the CTOD with the striation spacing across a range of steels and aluminum alloys, typically finding that there was an order of magnitude difference between the CTOD and the striation spacing. An explanation for the divergent data on the correlation was provided by Tanaka et al. [171], who examined the crack tip blunting in copper at different $\Delta J$ levels. At high strains the opening displacement is confined almost entirely to the crack tip. This causes the CTOD to contribute significantly to the crack growth per cycle, although the amount of crack growth is not 100% of the CTOD. At lower strains, crack opening assumes a more parabolic arc, resulting in less displacement at the crack tip for a given amount of crack advance.

In summary, the assumption inherent to the Laird and Smith model, that one loading event results in a single crack advance event and the formation of a striation, does not hold in these studies. While Davidson and Lankford agreed with the assessment that the formation of striations was due to advancing of a crack, they noted that it could take many loading cycles, up to 43, to advance the crack one striation spacing [173]. Thus, the fully geometric conditions of blunting and resharping at the crack tip maybe accurate at high $\Delta K$ levels, but do not fully capture the complexity at lower $\Delta K$ levels.

2.17 Modeling of Fatigue Crack Propagation

Modeling efforts of fatigue crack propagation have attempted to model the mechanism of fatigue crack propagation, rate of fatigue crack propagation, or a combination of the two. As such this section will be split into two subsections. The first will discuss the efforts to mechanistically model crack growth, while the second will discuss the efforts to model the rate of crack propagation. Some models attempt both and will be cited in the relevant places.
Another type of model, that has become common in recent years, are atomistic MD simulations. A recent review of this class of simulations was prepared by Horstemeyer et al. [187]. While these are a promising tool for future qualification of crack growth current, MD simulations are limited to high strain rates and small areas of interest. As such they have primarily been utilized to study crack growth behavior of nanocrystalline materials [188, 189] and there current relevance to large scale polycrystalline crack growth is questionable. As such they have been largely been omitted from this literature review to focus on continuum and dislocation level models applicable to large scale fatigue crack growth.

2.17.1 Mechanistic Modeling of Fatigue Crack Propagation

FEM models have been composed to show how crack tip shape changes could lead to crack advance and striation formation [190-192] and how this would influence the crack tip plastic zone [193, 194]. Toribio et al. [190, 191] used this approach to study crack advance in a mild steel. As the steel was treated as a continuum material, no crystallographic information was considered. The FEM simulation showed considerable stretching of the elements along the crack growth direction during the blunting process, advancing the crack. This continued as the crack tip was cycled 5 times, with the crack tip elements progressively opening, and continuing to stretch in the crack growth direction. No remeshing techniques were utilized, so the elements at the crack tip gradually became more and more distorted. The low peak loads did not show any evidence of resharpening. The crack tip had a greater displacement than the crack flank further away, something not found experimentally. No evidence of striations was seen. This model does suggest that plastic deformation due to blunting could advance a crack.

To properly investigate striation formation, larger displacements at the crack tip are needed. Levokovitch et al. [192] did a large scale simulation of a single-edge notch specimen for 5 cycles of a generic material. Three different simulation were performed. The first treated the material as a continuum material in an effort to simulation fatigue crack growth in polycrystalline materials. The second was performed using crystal plasticity to simulate fatigue crack growth in a single crystal along the [100] direction. The third also utilized crystal plasticity to simulate fatigue crack growth in a [110] direction. Due to the large deformation in the elements near the mesh, an automated remeshing technique was used when the elements became too distorted. The model demonstrated the two principals of the blunting model of crack growth proposed by Laird and
Smith for polycrystals. First, during the loading phase the crack blunts, stretching the elements ahead of it and forming new surfaces. Upon compressive loading, the crack tip buckles upon itself, creating steps away from the surface which were identified as striation marks. This is a near perfect match to the mechanical model proposed by Laird and Smith, however the authors’ acknowledged that the remeshing technique could have influenced the buckling behavior, as the crack tip deformation state was very complicated. No mesh dependence study was performed to verify this.

On the single crystal specimens, they found that striations formed when the crack growth was in the [100] direction, but not when the crack growth was in the [110] direction in agreement with the experiments performed before by Neumann [181]. This study lends credence to the idea that striation formation could be dictated entirely by the nature of deformation at the crack tip. It also suggests variations in the orientation of the slip systems could explain the crystallographic dependence on striation formation, as opposed to variations slip band orientation in the alternate shear hypothesis.

Others have attempted to model crack growth via the ductile blunting method utilizing discrete dislocation dynamics. Specifically, dislocation dynamics models have focused on exploring the geometric changes at the crack tip and the structure of dislocations emitted from it. Bjerken and Melin [195] simulated a 2-dimensional BCC material in plane strain with a notch for 14,115 cycles. They found that crack advance was due to the shape change at the crack tip consistent with the Laird and Smith theory. Furthermore, they tied the shape change to the emission of geometrically necessary dislocations. The dislocations were emitted along the planes of maximum shear and formed two shear bands ±45° from the crack growth direction. The shape changes at the crack tip produces steps on the crack flank consistent with the striation formation. This study suggest that the Laird and Smith model of crack growth remains the most accurate.

Another attempt at simulating plastic blunting via dislocation dynamics by Riemelmoser et al. [176] found a different origin for striation markings. They simulated a single edge notch specimen of alpha iron and found, like Bjerken and Melin, that crack advance was due to the Laird and Smith ductile blunting mechanism. The blunting was accompanied by the emission of geometrically necessary dislocation along the planes of maximum shear. However, Riemelmoser et al. concluded that after a period of crack advance, the dislocations would self-organize into stable slip bands, contrasting with the work of Bjerken and Melin who found that the dislocations would remain
along the shear direction. Furthermore, Riemelmoser et al. found that these slip bands would impact the crack flank, creating periodic slip traces away from the surface; these formed the striation markings. Riemelmoser et al. also proposed that the minimum striation spacing was due to the long range slip band interaction producing a minimum spacing on the slip bands. These suggestions posit that the crack continues to grow with each loading cycle, instead of incrementally through a burst of crack advance associated with striation markings. This is directly contradicted by the experimental work of Davidson and Lankford [173, 174], who found that the crack continued to grow incrementally.

An atomistic MD study of the dislocation nucleation surrounding a large-scale crack under fatigue loading was performed by Machová et al. [196]. They examined how the dislocation structure evolved in BCC iron due to crack tip deformation. An atomically sharp edge crack along the (001) plane was simulated for 6 loading cycles at stresses as high as a 3 GPa. It was found that dislocations were emitted along the slip systems with the highest shear stress, but the high strain distorted that lattice so much that parts spontaneously transitioned from BCC to FCC.

### 2.17.2 Modeling of the Crack Propagation Rate

Current physically-based models of crack propagation are based on the concept of dislocation emission from the crack tip. This was shown to leave a permanent displacement equal to the magnitude of the Burgers vector of the dislocations by the work of Rice and Thomson [117]. The collective Burgers vector created via dislocation emission per cycle is therefore equal to the crack growth rate. The current state-of-the-art models have been discussed in a recent review by Chowdhury and Sehitoglu [197]. Broadly, these models break down into three categories: those concerned with solely dislocation emission, those that incorporate dislocation blocking, and those that consider slip irreversibility.

The first category are models concerned solely with dislocation emission, and assume that no obstacles to slip occur. The differences between the models are the factors used to approximate the number of emitted dislocations. The earliest models, including those of Weertman [198, 199] and Rice [200], related the dislocation emission to global factors including the stress intensity factor, the yield stress, and a critical hysteresis energy. Later models, such as those of Yokobori et al. [201, 202], utilized individual dislocations to relate the rate of dislocation emission to local factors including the velocity and density of dislocations. The most recent work in the field is that
of Deshpande et al. [203, 204] who utilized discrete dislocation dynamics to investigate how the microstructural evolution affected dislocation emission. They found that Peach-Kohler forces would cause dislocations to congregate into slip bands, with the stress necessary to emit a dislocation dependent upon the density of the dislocation within the slip band. In addition, they claimed that slip bands were individually associated with striation marks on the fracture surface.

The second category of models, focus on how the interaction of the emitted dislocations and slip barriers affect the rate of dislocation emission at the crack tip. Tanaka et al. [205, 206] modeled crack growth as solely due to the emission of dislocations with a single slip band, with each dislocation generated advancing the crack by a single Burgers vector. A frictional stress acted on the dislocations in the slip band promoting dislocation glide. Obstacles to dislocation glide, in the form of grain boundaries, had a given and constant resistance to dislocation glide. This model predicted that crack growth would slow as these barriers were approached, due to the lower frictional stress for dislocation glide. This was an attempt to model small crack growth, where grain boundaries have been shown to be barriers to crack growth. To unify the small and large crack growth models Navarro and de los Rios [207, 208] incorporated a term which lowered the resistance of grain boundaries to dislocation glide as the number of dislocation that passed through the barrier increased. This model predicted that the retardation effect provide by grain boundaries would lessen as more grain boundaries were crossed, providing an explanation for the differing growth rates of short and long cracks.

The final category of models consider only irreversible slip as creating permanent crack extension, as dislocations that return to the crack tip during the compressive phase are considered to not advance the crack. A model of how irreversible slip occurs at the crack tip was proposed by Frong and Thomas [209, 210] and Wu, Koul and Krausz [211]. This model proposed that during the tensile phase the blunting crack emits dislocations along one slip system, which glide back toward the crack tip during the compressive phase. The glide back toward the crack tip activates a second parallel slip system, ending the operation of the first slip system. This second system is active through the rest of the compressive phase, and serves to sharpen the blunted crack. Since the amount of emitted dislocation and the number of dislocations that return to the crack tip are unequal the crack advances.
Another model of irreversible plastic slip was proposed by Pippan [212, 213]. In this model, dislocation are emitted on a single slip plane ahead of the crack during the tensile phase. The dislocations then attempt to reverse direction, and glide back to the crack tip during the compressive phase. However, the crack tip deformation during the compressive phase results in the emission of another set of dislocation along the same slip plane, but with opposite Burgers vectors. The second set of dislocations is inherently less numerous than the first, resulting in annihilation of some of the returning dislocations and a net accumulation of Burgers vectors at the crack tip resulting in crack extension. An implication of this model is that there would be a dislocation free zone ahead of the crack tip created by the mutual annihilation.

The most recent irreversible slip model was proposed by Chowdhury et al. [214-216]. This model examines how the dislocation interact with obstacles to produce irreversible slip. During the tensile phase, dislocation are emitted from a crack tip and slip (forward motion) until encountering a grain boundary, where upon they transmit across the boundary and continue to slip. During the compressive phase, dislocations attempt to slip back to the crack tip (reverse motion). The grain boundary imposes frictional stresses on both forward and reverse dislocation motion; the amount of frictional stress the grain boundary imposes was estimated by the magnitude residual Burgers vector created in the grain boundary by dislocation transmission across it. The levels of forward and reverse slip are different due to a difference in the frictional stresses resisting dislocation motion. This leads to a net accumulation of Burgers vectors at the crack tip per cycle resulting in crack advance.

A common feature of these models is that the number of slip bands and interactions considered are very small. Additionally, they assume that no preexisting microstructure, whether from cyclic loading or the plastic zone of the crack tip is present, and as such dislocations are capable of easily slipping away from the crack tip. The accuracy of these assumptions needs to be verified through detailed microstructure examinination.

**2.18 Dislocation Structures Surrounding Stage II Crack Growths**

Understanding the dislocation structures around a growing crack is essential to resolving questions about the mechanism of crack growth. Unfortunately, the dislocation microstructure surrounding the fatigue cracks can only be examined via TEM or electron contrast channeling imaging (ECCI). Two techniques have been employed to produce TEM samples. In the first technique, the crack is
arrested and the sample is thinly sliced along the direction of crack growth. Then the region containing the crack tip is punched out of the thin slice and thinned to electron transparency via jet electropolishing techniques [217]. In the second technique, the sample is fatigued until final fracture. Material is then electroplated onto the fracture surface until enough has been deposited so that a disc with the fracture surface at its center can be punched out. This disc is then thinned via conventional TEM techniques such as jet electro polishing or dimpling and ion polishing [218].

Both techniques have major drawbacks. The punch out method can result in material loss in the area immediately next to the crack flank. The technique is also unsuitable for cracks with a substantial amount of opening. The electroplating technique only works with pure material or simple alloys as the electroplated material must be identical to the base material to avoid preferential polishing rates during the thinning to electron transparency.

Recently, the proliferation of dual beam FIB instruments has enabled a third way of producing samples from fracture surface. FIB machining allows for specific locations on the fracture surface to be chosen, while having no requirements as to the material type. The drawback to the FIB preparation are the long preparation time per sample, the limited visible area, and the induced radiation damage [219, 220].

The microstructure surrounding fatigue cracks in FCC materials depends on the type of slip. For wavy slip materials, such as copper, the primary structure surrounding crack flanks have been dislocation cells [218, 221-223]. Dislocation cells decrease in size with an increase in stress intensity factor. The dislocation cells also decrease in size in proximity of the crack flank. The misorientation of the cells increases nearer the crack flank with a corresponding increase in the hardness [222]. These dislocation cells appear on both single and polycrystalline materials.

Recent work by Haung et al. [224] demonstrated that in polycrystalline copper the structure next to a fatigue crack tip was dependent on the stress intensity factor. In their experiment, the crack growth rate, which is linked to both the stress intensity factor and the size of the reversed plastic zone, was varied between $5 \times 10^{-5}$ mm/cycle and $5 \times 10^{-7}$ mm/cycle. At the higher crack growth rate the microstructure surrounding the crack consisted of dislocation cells, while at the lower crack growth rate the dislocation microstructure consisted of loop patches. When the crack growth rate was switched the microstructure transitioned between the two structures. This research suggests that the microstructure near the crack tip is solely dependent on the microstructure formed by
cyclic loading, and is unmodified by the either the plastic zone of the crack tip, or the deformation inherent to crack advance via ductile blunting. This stands in contradiction to the earlier effort to map the plastic zone, where large increases in strain were noted near the crack tip; and the efforts to understand ductile blunting, wherein slip traces were observed being emitted from the crack tip at the preferred angles.

The dislocation structure of planar slip material has received less attention. Immediately adjacent to the to the crack flank, fine dislocation cells or subgrains have been found in 70-30 brass [225], 2024 Al [226], Al-Cu-Mg alloys [227, 228], and Al-Mg-Cu-Zn[229]. There is some evidence that these fine dislocation features have a size sensitivity to the stress intensity factor and that they become larger further away from the crack flank. When examining the crack tip, Awatani et al. [225] found that the dislocation cells did not occur ahead of the crack tip, suggesting that they were formed as part of crack closure. Away from the crack tip, the microstructure give way to intense deformation bands. Bowles and Broek [230] have claimed that these intense deformation bands correspond with each striation marking on the surface. Planar slip deformation can be seen outside these intense bands.

In summary, while the dislocation structures surrounding the crack tip have been imaged, the formation of the structure are unknown. Work has not been done that correctly isolates the contributions of the three strain components: the strain from the cyclic loading, the strain created by the yielding in the crack tip plastic zone, and the strain associated with the crack tip deformation. By not isolating out how these elements interact, it is also currently impossible to directly tie a microstructural feature found near the crack tip or beneath the fracture surface to an individual crack advance increment or striation marking.

2.19 Key Questions Remaining

Based upon an extensive review of the background literature, I have identified several areas where the relationship between fatigue crack growth and the microstructure remains unclear:

1. While the size and shape the plastic zone of the crack tip is well known, recent research has shown that there are substantial inhomogeneities both between the bands and within the individual bands. Such inhomogeneities are attributed to the microstructure of the underlying material, but a direct comparison to the dislocation structure is lacking.
2. The dislocation structure surrounding fatigue cracks is known, but how the dislocations structure comes about and what strain inputs contribute to the dislocation structure are unclear.

3. While microstructure features have been tied to both crack advance increments and the striations markings found on the fracture surface, no convincing correlation between such features has been shown.

4. Current physically-based models of fatigue crack propagation are based around the interactions of a small number of slip systems with a small number of obstacles. The relevance of the obstacles and what is simulated remains questionable. Improving the knowledge of the microstructure at crack tips, including the exact microstructure that crack tips propagate into and the effect that such propagation has on that model, could lead to improved physically-based models of crack growth and more fatigue resistant materials and structures.

2.20 References


CHAPTER 3
MATERIALS AND METHODS

This chapter describes the experimental procedures that were utilized for this work on understanding the evolved microstructure surrounding fatigue cracks. Descriptions are provided of the following: first, the materials used and preparation techniques; second, the techniques used for testing samples and the cyclic loading conditions; third, the techniques used for producing transmission electron microscopy (TEM) samples from the fatigued specimens and the locations from which the TEM samples were extracted; and finally, the microscopy techniques used to analyze the samples.

3.1 Materials

Five different FCC metals were used to study the fatigue crack growth. These materials broadly fall into two categories: metal alloys, which included Haynes 230, 316 austenite stainless steel and Nitronic 40 stainless steel; and pure metals, which included copper and nickel. The primary material of interest was Haynes 230, a solid-solution-strengthened planar-slip Ni-alloy. The composition of Haynes 230 is given in Table 3.1. Planar slip in Haynes 230 does not arise from the stacking fault energy, but from short-range order within the solid solution [1]. The Haynes 230 alloy had previously undergone a solution heat treatment and was used in the as-received condition.

Two other metal alloys were studied, 316 stainless steel and Nitronic 40 stainless steel; the composition of each is given in Table 3.1. 316 stainless steel exhibits wavy slip under monotonic loading, but transitions to planar slip under cyclic loading [5]. In contrast, Nitronic 40 deforms by planar dislocation slip initially, and later transitions to deformation twinning [6]. The two pure metals studied were Oxygen-Free High Conductivity copper and Ni-200, commercially pure nickel. For the sake of simplicity, the latter two will be referred to as copper and nickel throughout this document.

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<td>-</td>
<td>0.06</td>
<td>0.3</td>
<td>0.03</td>
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3.2 Fatigue Bar Production

Single edge-notched fatigue bars were produced via electro-discharge machining (EDM) from the materials of interest. The dimensions of the bars were adjusted to accommodate different load frames used. Table 3.2 details the size of the bars by the material used.

<table>
<thead>
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<th>Material</th>
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<th>Height (mm)</th>
<th>Gauge Length (mm)</th>
<th>Notch Depth (mm)</th>
<th>Notch Width (mm)</th>
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<tr>
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<td>0.5</td>
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<tr>
<td>316</td>
<td>9</td>
<td>4</td>
<td>50</td>
<td>1.5</td>
<td>0.15</td>
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<tr>
<td>nickel</td>
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<td>50</td>
<td>1</td>
<td>0.15</td>
</tr>
</tbody>
</table>

After fabrication, the bars made from pure nickel and copper were annealed in a vacuum furnace at a pressure of approximately 10⁻⁶ torr for one hour at 700°C and 450°C, respectively. No post-EDM anneal was employed on the metal alloys; these were used in the as-received condition.

All samples were polished by mechanical grinding, employing progressively smaller grits down to a final 1 µm diamond solution polish. Initially, the grinding was necessary to remove the oxidation created by the EDM process, with further polishing to remove the deformation layer imposed by the mechanical polishing. One side was further mechanically polished through finer grits to a final 0.02 µm colloidal silica solution so that electron backscatter diffraction (EBSD) analysis could be performed.

3.3 Fatigue Testing and the TEM Sample Locations

Prior to cyclic loading, a TEM sample was extracted from each material to verify the starting-state dislocation structure. The bars were cyclically loaded using either an Instron 8008 servo-hydraulic system or an Instron E10000 electrodynamic system. The cyclic loading conditions are presented in Table 3.3 for each material; some samples were loaded to failure while others were arrested during crack growth. Some of the arrested crack samples were reloaded in monotonic tension. Crack growth and plastic zone size were monitored in situ via DIC for some samples. The initial loading data and the list of experiments performed by material are summarized in Table 3.3. A special case occurred during the loading of copper. The cyclic hardening of copper caused crack...
arrest relatively quickly. Since the crack growth was monitored in situ each time that the crack arrested itself, the upper and lower stress values were raised until either final failure or crack arrest occurred. Correspondingly, the two delta sigma values shown are at the initial loading and at the final failure.

Table 3.3: Cyclic loading conditions

<table>
<thead>
<tr>
<th>Material</th>
<th>$\Delta\sigma$ (MPa)</th>
<th>R-ratio</th>
<th>DIC used</th>
<th>Failure</th>
<th>Arrested Crack</th>
<th>Monotonic Loading</th>
</tr>
</thead>
<tbody>
<tr>
<td>Haynes 230</td>
<td>270</td>
<td>0.05</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>copper</td>
<td>23.75 - 95*</td>
<td>0.05</td>
<td>✓</td>
<td>✓</td>
<td></td>
<td></td>
</tr>
<tr>
<td>316</td>
<td>200</td>
<td>0.1</td>
<td></td>
<td>✓</td>
<td>✓</td>
<td></td>
</tr>
<tr>
<td>nickel</td>
<td>188</td>
<td>0.05</td>
<td></td>
<td></td>
<td></td>
<td>✓</td>
</tr>
<tr>
<td>Nitronic 40</td>
<td>225</td>
<td>0.05</td>
<td>✓</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Loading stress increased until test end to overcome work hardening

The first studies were conducted on the free surface of Haynes 230, following the cyclic loading, the plastic zone ahead of an arrested crack was examined via EBSD. The slip trace orientations and density were assessed via SEM at specific points both inside the plastic zone and outside the plastic zone. TEM samples were then extracted via FIB machining at these locations so that the microstructural evolution in the plastic zone could be assessed. Finally, to complete the understanding of the microstructural evolution, a TEM sample containing the crack tip was extracted. All other materials were limited to extraction of a single TEM sample containing the arrested crack tip, as the microstructure evolution was either well documented, in the case of copper and nickel, or inferred from the assessments of Haynes 230.

For the samples cycled to failure, the fracture surface was first investigated via SEM. The structures and crack path on the fracture surfaces were assessed to find striation markings. TEM samples were then extracted from regions of the fracture surface showing striations; this provides an assessment of the microstructural evolution in both plane strain and plane stress strain states.

In order to assess the microstructure at the crack tip away from the free surface, some of the arrested cracks were reloaded in monotonic tension to open the sample while preserving the crack tip. These samples are listed in Table 3.3. These tests were performed by pulling the sample using an Instron E10000 at a constant displacement rate of 0.001 mm/s. This rate corresponds to an initial
strain rate of 3x10^-5 /s. TEM samples were extracted from the region subjected to the change in loading mode change.

3.4 SEM and TEM Sample Production Techniques

Prior to fatigue testing, the initial microstructure was assessed using both EBSD and TEM analysis. Two SEMs were used to perform EBSD: a JEOL 7000F fitted with an HKL EBSD software system and a Zeiss 1530 “Leo” fitted with a TSL EBSD system. The EBSD scan was slightly different on each machine, as the HKL system used a square grid to sample points whereas the TSL system used a hexagonal system of points. The samples were scanned for crystallographic orientation mapping and the Kernel Average Misorientation (KAM). Orientation mapping provided the average grain size, the fraction of grain boundaries that were twins, and evidence of any texture in the material. KAM analyzed the surface strains, demonstrated that there was no case hardened layer at the surface, and provided a base for comparison to post-deformation EBSD scans of the plastic zone around cracks. Spurious points, i.e. those indexed at an orientation at odds with all of their nearest neighbors, were removed via a noise reduction algorithm within the EBSD software.

To create the TEM samples from either the free or fracture surface, Focused Ion Beam (FIB) micromachining was utilized to extract samples normal to the surface and to thin them to electron transparency. Three different dual-beam FIB instruments were used to extract samples: an FEI DB235; an FEI Helios NanoLab 600i; and a Zeiss Auriga. The major difference between the FIBs was the material used for Ion Beam-Induced Deposition (IBID): both of the FEI FIBs using an amorphous Pt-C composite, while the Zeiss FIB used amorphous C. Thus, the deposited material is visible in TEM micrographs of samples made by the FEI FIBs, but largely invisible in the samples created using the Zeiss FIB, due to the higher Z of the Pt deposition. Figure 3.1 details the process by which samples were created. While the Zeiss and FEI FIBs have slightly different geometries for performing the extraction, the general process is similar.

First, an area of interest is found using the electron beam functionality of the dual beam FIB, Figure 3.1a. Second, IBID was used to deposit material on the surface. This deposition did not alter the surface and served to protect the surface from geometry changes and radiation damage in the subsequent steps. This cap is indicated with an arrowhead on Figure 3.1b. Next, the ion beam was used to ablate the material on either side of the region of interest, leaving a small cross section, Figure 3.1c. The sample was then cut away from the sides, leaving a small bridge of material to
hold it in place, as can be seen by the cuts made to the sides and bottom of the sample in Figure 3.1d. A tungsten needle was inserted and attached to the sample via IBID, seen attached in Figure 3.1d. The bridge of remaining material was then cut away and the sample transferred to a copper TEM grid inside the FIB, Figure 3.1e. The sample is welded to the copper grid via IBID and the needle milled off. Finally, the sample was milled to electron transparency, Figure 3.1f.

![Figure 3.1: The process by which a TEM sample is created using the FIB; a: shows the area of interest; b: shows the deposited protective material, this is indicated with an arrowhead; c: shows the results from milling the surrounding material. d: shows how the samples is cut out of the surrounding material and attached to the needle; e: shows the needle attaching the sample to copper grid, while f: shows the material after thinning to electron transparency.](image)

The polished surface of the arrested crack samples was studied via the EBSD and FIB techniques described earlier. The fracture surface morphology of the failed samples was assessed using the SEM mode of the FIB machines with samples extracted from points of interest for TEM evaluation.

### 3.5 TEM techniques

The samples produced by FIB micromachining were examined using a TEM and a scanning transmission electron microscope (STEM). Either a JEOL 2010 “LaB₆” operating at 200 kV or a Tecnai TF30 operating at 300 kV was used for diffraction contrast TEM imaging and for obtaining Selected Area Diffraction Patterns (SADP) of the microstructure. Only the Tecnai TF30 was used
for diffraction contrast STEM imaging. Diffraction contrast TEM images were formed by placing the sample into a two-beam diffraction condition, in which only the transmitted beam and a single diffracted beam were excited, and collected by an UltraScan CCD. The diffraction contrast STEM images were obtained following the techniques developed by Phillips et al. [7]. In the STEM technique, the crystal was aligned such that the converged beam was parallel to a zone axis. The beam was then scanned over the area of interest, and resulting diffraction patterns were captured both by the bright-field detector and the high angle annular dark-field detector to construct BF and HAADF images simultaneously. The STEM technique was used as there was better penetration of thick samples, the contrast difference at the surface was lessened, and a larger field of view was possible. Diffraction contrast micrographs and SADPs were captured with Gatan manufactured CCD cameras with the JEOL microscope capturing at 2004x1336 pixels and the Tecnai capturing at 2048x2048 pixels. Diffraction contrast STEM images were obtained using the bright-field STEM detector and captured at a resolution of 1024x1024 pixels. TEM images were converted to TIFF images using Gatan Digital Micrograph software, while STEM images were converted to TIFF images using ImageJ. Subsequent image adjustments were limited to level and curve adjustments in Adobe Photoshop CS6. Large area-of-view images were constructed by manually aligning the images and using the auto-blend features in Adobe Photoshop CS6 to blend together multiple images.

3.6 References

CHAPTER 4

EXPERIMENTAL RESULTS

In this chapter, the results of the investigation of the microstructure in pure FCC metals, copper and nickel, are compared with that generated in FCC alloys, Haynes 230, 316 austenitic stainless steel, and Nitronic 40. This results section is organized according to the material being tested. Results from the testing of Haynes 230 are presented first at these were the most comprehensive set of experiments. The microstructural state in the other alloys was explored to identify similarities and differences.

4.1 Studies of the Microstructural Evolution on the Free Surface in Haynes 230

The goal of this investigation was to determine the evolved microstructural state produced at the crack tip in the surface region, and compare it to that generated beneath striations. This information would allow for the determination of the evolved microstructure attributed to cyclic loading, the plastic zone of the crack, and the propagation of the crack. Consequently, this section is organized by the various experiments performed on Haynes 230. First, the microstructure of the undeformed material is discussed. Second, the observations on the evolved microstructural state as a function of distance from an arrested fatigue crack after 53,355 cycles, and a stress intensity factor of 35 MPa√m, are presented. The third set of experiments determined the evolved microstructural state produced in the interior of the sample under fatigue loading. The fourth, and final, set of experiments compared the evolved microstructural state developed beneath striations and microvoids.

4.1.1 Undeformed Microstructure

After the polishing was complete, but before any loading, EBSD scans of the surface were created to determine the initial microstructure of the sample. Figure 4.1a shows the orientation map of the sample surface. The step size used to create this map was 1.5 µm while the probe size was on the order of 10-20 nm. From this orientation map, the average grain diameter was 50 µm and 54% of the grain boundaries were Σ3 twin boundaries. To create the Kernel Average Misorientation (KAM) map, Figure 4.1b, the misorientation angle between a point and each of its nearest neighbors was determined and then averaged to a single value. Misorientations higher than 5° were excluded from these averages, as these were assumed to be grain boundaries. The small
misorientations from this map arise from geometrically necessary dislocations slightly distorting the orientation in a given grain. A weakness of this method is that it does not provide information on stored dislocation dipoles, as they induce no net crystal deformation [1]. Figure 4.1b suggests there are few geometrically necessary dislocations within the sample. The line of highly misoriented points shown by the white arrowheads in Fig. 4.1b is due to a defect that was not removed by the polishing. This map also serves as a baseline for comparison with ones generated after crack growth.

Figure 4.1: An EBSD scan of the polished surface: a: orientation map and b: the Kernel Average Misorientation map. The white arrowheads in b indicate the location of a surface defect, and the black arrow indicates the location of the machined notch.

To examine the existing dislocation microstructure prior to any deformation, a sample was extracted from the specimen away from the notch and crack path by FIB machining, and thinned to electron transparency. The sample contained a low dislocation density, with the dislocations arranged in planar arrays. A representative bright-field electron micrograph of the dislocation
structure is presented as Figure 4.2. The low dislocation density, along with the lack of any additional deformation at the surface, demonstrates that the polishing was sufficient to remove any damage created by the EDM machining used to make the fatigue specimen. The black dots are small dislocation loops created by the ion beam process which was used to extract the sample and thin it to electron transparency. The dislocation loops are concentrated near the sample surface in a layer ~30 nm wide. This damage is unlikely to modify the underlying microstructure in any significant way.

Together these results show that the starting microstructure consisted of large grains with a low dislocation density.

**4.1.2 Assessment of the Surface Strain**
This fatigue test was conducted in collaboration with Prof. H. Sehitoglu’s group at the University of Illinois. During the fatigue test, the crack growth was monitored *in situ* with periodic assessment of the strain ahead of the crack tip utilizing DIC. Sehitoglu and coworkers have used this method to follow the buildup of strain with the number of cycles as the crack advances [2, 3]. The speckle pattern necessary for DIC was applied to one side of the sample while the post-mortem EBSD was performed on the other side. Figure 4.3 shows the DIC strain map after 53,000 cycles. As expected,
the strain is concentrated into two lobes approximately 45° from the direction of crack growth. The map suggests the strain within each lobe and the degree of non-uniformity within each lobe is different. From the map, it appears that the strain in the uppermost band is greater than in the lower band. In both lobes it appears that the strain is high and relatively uniform at distances between 200 µm and 500 µm from the crack tip. Within the band, significant non-uniformities in the strain are evident. Examples of non-uniform strain locations are indicated by arrows in Figure 4.3.

![DIC map of the strain field ahead of the crack tip after 53,000 cycles. The black arrow indicate the locations of non-uniform strain, while the white arrow indicates the location of the crack tip.](image)

Following the acquisition of DIC data shown in Figure 4.3, the sample was subjected to an additional 335 cycles. This resulted in a total crack extension of 1.1 mm, and a total increase in the stress intensity factor from 7 MPa√m to 35 MPa√m. This is the condition of the sample used for post-deformation analysis of the microstructure as a function of distance from the arrested crack, and for the load mode change experiment described in section 4.3.
The post-mortem EBSD map of the free surface ahead of the arrested crack is presented as Figure 4.4. Two scans, with a step size 1.5 µm and a probe size on the order of 20 nm, were obtained ahead of the arrested crack to map the plastic zone. The two scans were combined to make the KAM map, Figure 4.4a. This map demonstrates that the strain forms into two lobes oriented approximately 45° from the direction of crack growth, which is consistent with the DIC map. The strain lobes are visible as the concentrations of the higher misorientations, which is shown as a
mix of green, yellow and red over the blue background. The strain lobes start off narrowly confined
to an area of roughly 50 µm at the crack tip but gradually expand outward with increasing distance
from the crack tip, this spreading of the strain over a larger area was less evident in the DIC map
presented in Figure 4.3. The intensity of the misorientations decreases with distance from the crack
tip. The KAM shows that the strain is not as homogenous as indicated by the DIC map and that
significant local variations in strain occur. The orientation map, Figure 4.4b, shows that the
microstructure of the material does not undergo any substantial change. Both the grain size and
the twin percentage are comparable to Figure 4.1. Figure 4.4c and e highlight the region near the
crack tip of both the KAM and orientation map, respectively. The areas of highest misorientation,
red in the KAM map, tend to correlate with the location of the grain boundaries, though regions of
high misorientations can also be seen in the interior of grains. This concentration of strain occurs
at both twin boundaries, as indicated by the back arrow, and random high-angle grain boundaries,
indicated by the white arrow in Figure 4.4c. The higher misorientations at the grain boundaries
may be related to the accumulation of dislocations along the grain boundary by the formation of
dislocation pile-ups or the accumulation of lattice dislocations in the grain boundary itself. The
specific microstructure associated with grain boundary microstructure will be discussed later.

In addition to the distribution of the strain, the KAM map gives some insight into the nature of the
dislocation structures that occur. Slip traces are distinguished from the background as lines of
higher misorientation. An area containing a slip trace, indicated by the black arrowhead, is shown
in the KAM and the orientation maps, presented as Figure 4.4d and f, respectively. The slip trace
continues across the grain boundary suggesting that dislocation transmission is occurring.

To further understand the strain evolution at and ahead of the crack tip, a finite element model was
created by Dr. Mohsen Dadfarnia, the result of which is presented as Figure 4.5. The model, of
which only half is shown due to symmetry conditions, showed that the preferential strain lobe was
in general agreement with the DIC and EBSD data. The strain continuously increased as the crack
tip was approached and was entirely homogenous. Additionally, the FE model demonstrated that
the entire uncracked ligament was undergoing plastic deformation when the crack was arrested
due to the loading, albeit at a low strain level. This model does not capture the complexities at the
dislocation level that the EBSD scan did, but in general the results are consistent.
In general, the surface measurement techniques were in agreement with one another. The plastic zone associated with the crack tip was concentrated in lobes oriented 45° from the crack growth direction. The amount of plastic deformation continuously increased. With increasing microstructure sensitivity, the plastic deformation became less homogenous, both between the upper and lower lobe and within the lobes. In particular, the KAM map shows that grain boundaries were preferential sites for strain accumulation.

### 4.1.3 The Dislocation Microstructure on the Free Surface ahead of an Arrested Crack

To correlate the surface strain measurements with the underlying microstructure, samples were taken from the free surface at varying distances, inside and outside the lobe, with respect to the crack tip. Figure 4.6a, a secondary electron SEM image, shows an overview of the surface with the locations from which TEM samples were extracted highlighted. Each of the locations is referenced with a number for the subsequent TEM analysis. The locations were selected to investigate the dislocation structures inside and outside the strain lobes. This can be visualized
more clearly in Figure 4.6b, which shows the SEM image with the KAM map overlaid and the sites of TEM sample extraction indicated. Locations 1-3 occur in an arc approximately 800 µm away from the crack tip, with locations 1 and 2 from areas within the strain lobe, and location 3 outside the lobe. Locations 4 and 5 are approximately 400 µm away from the crack tip, with location 4 inside the strain lobe, and location 5 outside. Locations 6 and 7 were both taken approximately 80 µm from the crack tip, with 6 within the lobe, and 7 outside. Location 8 was taken directly from the crack tip.

Figure 4.6: The free surface ahead of the crack tip: a: SEM image of the free surface ahead of the crack tip, with locations from which the TEM samples were extracted highlighted; and b: an image of the free surface ahead of the crack tip with the KAM map superimposed.

The surface at each of the locations was examined prior to sample extraction, and the slip trace density and number of activated slip systems was evaluated. The slip trace density and number of different slip systems activated are shown as a function of distance from the crack tip in the series of SEM images presented in Figure 4.7. While each of the images presented in Figure 4.7 were taken from locations within the strain lobe, the images are representative of the slip trace structure at locations both inside and outside of the strain lobe. Figure 4.7a shows that at a distance of 800 µm from the crack tip, Location 2, the slip trace density was low with no obvious pattern established. Two slip trace directions were identified, indicating two active slip systems. The slip traces are straight and evidence of double cross-slip from one active slip system back to the original system can be seen; an example of such an event is indicated by the black arrow on Figure 4.7a. At location 4, which corresponds to 400 µm from the crack tip, the density of slip traces and the number of different directions has increased; this is shown in Figure 4.7b. The white arrowheads
indicate a twin boundary. To the left of this boundary, slip traces are seen running in only one direction, though occasional cross-slip events are observed; an example is indicated by the black arrow. On the right side of the twin boundary, two different directions of slip traces are seen with the vertical slip traces occasionally showing evidence of cross-slip into a third direction. These areas of cross-slip are indicated with black arrows.

Figure 4.7: SEM micrographs of the surface as a function of distance from the crack tip; a: location 2, with the arrow indicating a cross-slip event; b: location 4 where the black arrowheads indicate cross slip events and the white arrowhead indicating the location of a twin boundary; c: location 6, with the white arrow indicating locations at which a slip trace crossed the grain boundary and the black arrow indicating the location where a slip trace was blocked by the grain boundary; and d: location 8, the black arrow indicates a line of cross-slip events and the white arrowhead indicates brighter slip traces indicative of higher deformation.
At 80 µm from the crack tip, location 6, three slip trace directions are seen and the slip traces are curved, see Figure 4.7c. Both of these are indicative of higher strain. There is no evidence of cross-slip in the dislocation traces at this location. Some of the slip traces, indicated by the black arrow, are continuous across the grain boundary, running through the center of the sample, while others, highlighted with the white arrowheads, are discontinuous across the boundary. Location 8, presented as Figure 4.7d, shows that the crack tip experiences higher strain. Three different slip trace directions are visible as well as a line of cross-slip events; examples of these events are highlighted by the black arrow. Such cross-slip events are consistent with the direction of the plastic zone. Several slip traces, highlighted by white arrowheads, appear brighter than others, which is attributed to these slip traces extending further out from the surface than other slip traces. In other words, the dislocation activity on the slip systems associated with these slip traces was higher than on the other traces.

![Figure 4.7: TEM micrographs showing the dislocation structure beneath Location 1; a: immediately beneath the surface; b-c: away from the surface. The black arrow on a indicates the location of a surface slip trace. The white arrows on b and c indicate the different planar slip band directions seen, and the black arrow on b shows where two different slip bands meet.](image)

As shown in the bright-field electron micrograph presented in Figure 4.8a, the dislocation structures beneath location 1, 800 µm directly in front of the crack, consists primarily of planar slip bands. The slip bands impacting the surface can result in steps at the surface, an example of a
surface step is shown in Figure 4.8a and is indicated by the black arrow. The height of this step is 12.7 nm, which would require 72 dislocations, assuming the lattice parameter in Haynes 230 is equal to 0.352 nm, that of pure nickel [4]. These steps would form the slip traces viewed in the SEM images seen in Figure 4.7. The micrographs away from the free surface, Figures 4.8b and c, show that the slip band density is not significantly affected by the free surface. The average slip band spacing in the dominant direction was 135 nm. Additionally, while one direction of planar slip banding is dominant, two other planar slip band directions are observed, indicated by the white arrows in Figure 4.8b and c. Intersections of slip bands with differing orientations produce small dislocation pile-ups, indicated by the black arrow in Figure 4.8b.

Location 2, located within the strain lobe 800 µm from the crack tip, has a similar density of planar slip bands as location 1, which was from outside the strain lobe. This is evident in the TEM micrographs from that area that are presented in Figure 4.9. Figure 4.9a shows that three different planar band slip systems are active. The arrows in Figure 4.9a indicate where steps on the free surface occur with the large step being the product of multiple parallel planar slip bands intersecting with the surface. The upper step, indicated by the black arrow, was 73 nm high and would have required 406 dislocations to have intersected the surface. The lower step, indicated by the white arrow, was 22 nm high and would have required 125 dislocations. Figure 4.9b shows the
planar slip bands away from the free surface. The average spacing of the dominant slip bands was 111 nm. The dislocations structures of the planar slip bands are revealed in Figure 4.9c, a micrograph of an inclined slip band. The average dislocation spacing was 46 nm. When viewed end on, the dislocations would form the planar slip lines seen in Figure 4.9b.

Figure 4.10: TEM micrographs from location 3; a: at the surface; b: away from the free surface and c: across a high-angle grain boundary. The black arrow indicates a slip band-grain boundary interaction and the white arrowheads indicate two different slip bands originating from the interaction location.

The final location in the arc 800 µm from the crack tip, location 3, occurs outside the strain lobe. The observed microstructure is presented as bright-field TEM micrographs in Figure 4.10. From Figure 4.10a it is seen that the density of planar slip bands is similar to Locations 1 and 2. Large steps are not seen at the surface because the primary slip band direction is parallel to the sample surface. Figure 4.10b shows the planar slip bands at a higher magnification. The average slip band spacing here was 101 nm. Once again, the intersection between different slip band systems produces small dislocation pile-ups. Figure 4.10c shows the planar slip band interactions with a grain boundary. The planar slip bands in the left grain appear correlated with planar slip bands from the right grain. The intersection of a slip band with a grain boundary, indicated by the black arrow, shows that a single slip band in the right grain correlates to the location of two planar slip bands in the left grain, indicated by the white arrowheads. Although the direction of slip is
unknown in this case, it is possible that the single slip band impinging on the grain boundary generates two slip systems in the adjoining grain as strain is transferred across the boundary.

![Figure 4.11: TEM micrographs from Location 4; a: planar slip bands inclined to the beam direction; b: parallel to the beam direction, and c: enlarged view of the boxed region in showing cross-slip of dislocations. In b the blue arrow indicates a location where it is difficult to assess the band spacing, the white arrow indicates a location showing the inclination of the bands. In c the planar array under investigation has the dislocations numbered 1-5.](image)

The microstructure observed at location 4, located 400 µm from the crack tip and inside the strain lobe, is shown in the bright-field TEM micrographs presented in Figure 4.11. No significant difference in the planar slip band spacing or the dislocation density was noted. The average distance between the dislocations contained within the planar slip bands is 66 nm, Figure 4.11a. The planar dislocation arrays visible in Figure 4.11b have an average spacing of 101 nm. Additionally, the bands here are slightly inclined to the surface, seen most vividly by the band indicated by the white arrow. This makes assessing which of the lines are separate bands and which are part of the same band difficult. An example of this difficulty is indicated by the blue arrow. Additionally, dislocations are seen cross-slipping from one planar array onto another. This is the focus of Figure 4.11c, an enlarged image of the boxed region from Figure 4.11b. Near the top of the image is a planar array composed of five dislocations, indicated by the white arrowhead. While the upper portion of the planar array has the appearance of one of the large planar bands slightly inclined, the dislocations within this array are seen cross-slipping to other bands. Two of the dislocations, numbered 3 and 4, are straight and form the backbone of the planar array. Two others,
numbered 1 and 2, cross slip from the large planar array that is visible in the lower left of the image. The fifth dislocation, numbered 5, comes into the planar array from the other side and does not appear to originate from a planar array. This gives two indications into the nature of slip in this alloy. First, dislocations not contained within the planar band structure are not independent as dislocations can cross-slip out of one planar band, into another. Therefore, when interpreting these micrographs, the dislocations that appear to be independent, i.e. not associated with a planar slip band, may be in the process of cross-sliping into other planar bands. Second, dislocations can come from multiple sources to form the planar slip bands spontaneously. Both of these implications highlight the difficulty of interpreting the dislocation structure from a single micrograph, which is a projection of the 3-dimensional dislocation structure on the electron exit surface of the sample.

Figure 4.12: Overview of the twin boundary found in location 4; a-b: twin boundary running through Location 4 with the boundary indicated by the white arrowheads; c: indexed SADP with the matrix diffraction spots indicated by the subscript M and twin spots by the subscript T. The black arrows in a indicate the directions of the primary slip bands. The black arrows in b show a planar band crossing the twin boundary

In addition to the dislocation structures described in Figure 4.11, Location 4 also contained a twin boundary. This boundary, and the dislocation structure that form around it, is the focus of Figure 4.12. Figure 4.12a and b, show the boundary interaction with the primary planar slip bands. Since the primary slip bands in the right grain, indicated by the black arrow, are oriented nearly parallel
to the twin boundary interactions between the primary bands and the twin boundary are not observed. In the left grain, a planar array of dislocations all with one end attached to the twin boundary, is observed. An interaction between a planar slip band and the twin boundary can be found in Figure 4.12b. A planar band can be seen on either side of the twin boundaries, indicated by the black arrows. The planar band crosses the boundary, changes direction and continues. Since the test was not performed in situ it is impossible to know the direction of the slip through this boundary, and therefore analysis to determine the slip planes and Burgers vectors of the dislocations was not performed. Figure 4.12c shows the SADP across the boundary confirming the boundary orientation. The diffraction spots are indexed with the matrix spots designated by the subscript M, and the twin spots by the subscript T.

To confirm that the dark lines were planar slip bands composed of dislocations, an electron tomogram was created to visualize the dislocation structures in three dimensions. Figure 4.13a is a bright-field TEM micrograph of the area of interest. From the still micrograph, it is not possible to determine if the dislocations not in a slip band are randomly distributed, or form another band when viewed in a different direction. To answer this question, this area was tilted ±40° in the x

Figure 4.13: Three dimensional analysis of the planar slip bands; a: bright-field TEM micrograph from location 4; b-c: views of a tomogram created of the same area at different angles. The white arrows indicate the same line of dislocations.
direction while maintaining a diffraction vector of [110]. An image was acquired at every 1° of x tilt. These images were aligned and reconstructed using Em3D (http://em3d.stanford.edu/) software and visualized using UCSF Chimera [5]. Figure 4.13b and c demonstrate this reconstruction at two different tilts. The planar slip band indicated by the white arrow in Figure 4.13b appear as separate dislocations. However, when viewed at a different angle of tilt, Figure 4.13c, it begins to collapse to a single band. This example, along with the cross-slip evident in Figure 4.11c, demonstrates that the spatial distribution of dislocations is difficult to interpret from an electron micrograph, which is simply an image of the structure as projected on the electron exit surface of the sample. Free dislocations seen in these images, might be part of a planar slip band that is not obvious, or the dislocations might be in the process of cross-slip from one planar slip band to another. This creates difficulties when trying to compare the dislocation distribution and densities.

Location 5, located outside the strain lobe 400 µm from the crack tip, has a similar dislocation density as location 4, which is located within the strain lobe. This can be seen by comparing the electron micrographs presented in Figure 4.11 and 4.14. Figure 4.14a shows an overview indicating that two directions of planar slip bands are active. The average spacing between the slip bands was 111 nm, while the average dislocation spacing within the slip bands was 47 nm. Figure 4.14b shows the dislocation structures within the planar slip bands in greater detail. Of particular note is the band highlighted with the arrow. This band appears to be collapsing into a single line as it moves toward the arrow.

In addition, to the dislocation structure this sample included a carbide allowing for the dislocation-carbide interaction to be visualized. Figure 4.14c presents the dislocation structure between the surface and the carbide. Only one slip band appears between the surface and the carbide, indicated by the black arrow, with most of the dislocations taking the form of tangles. Dislocations are also seen attached to the surface of the carbide. Interaction between the planar slip bands and the carbide are visible in Figure 4.14d. Planar slip bands are visible approaching the carbide from the bulk, but cross-slip before intersecting it. Several white arrows point to these cross-slip events which occur approximately 500 nm from the carbide. No planar slip bands are seen directly interacting with the carbide. Between the cross-slip location and the carbide, a dense tangle of dislocations exists with some dislocation half loops (indicated by the black arrow) coming off the
carbide. This latter observation suggests the carbide-matrix interface can act as a source of dislocations either directly or in response of lattice dislocations interacting with the particle [6-8].

Figure 4.14: Bright-field TEM micrograph from Location 5; a-b: dislocation structures in the bulk; and c-d: planar slip bands interacting with a carbide and the surface. The black arrows in b and c indicate planar bands of dislocations, and the black arrow in d indicates dislocations attached the carbide. The white arrows in d indicate locations where planar bands cross-slip upon interacting with the strain field of the carbide.

The microstructure from Location 7, 80 µm from directly in front of the crack tip, is presented in Figure 4.15. This microstructure is discussed before that of Location 6 because it is broadly similar to that of Locations 4 and 5. The surface is visible in Figure 4.15a, where it is evident that neither the planar slip band density nor the dislocation density is notably higher than at location 4 or 5, which are located 400 µm away from the crack tip inside and outside of the strain lobe, respectively. Away from the sample surface, Figure 4.15b, the dislocation structure is similar to that near the fracture surface. The average planar band spacing seen in these images, was 158 nm, while the average dislocation spacing within the bands was 83 nm. Only two directions of planar bands are seen, however multiple planar bands can be seen running in the same direction. Figure 4.15c shows the structure of the planar band indicated by the white arrow. The planar band is
composed of multiple dislocations aligned with each other. Dislocation can be seen cross-slipping into, or out of the planar band, indicated by the black arrowheads. Since the test was not performed \textit{in situ}, the direction of dislocation motion was unknown.

![Figure 4.15: Bright-field TEM micrographs from Location 7; a: at the free surface; b-c: within the bulk. The box on b demonstrates the location where c was obtained of the. The white arrow on c shows the location of the planar slip band while the black arrowheads indicate dislocations that have cross-slipped into or out of the slip band.](image)

The bright-field TEM micrographs presented in Figure 4.16 show the microstructure at location 6, located 80 µm from the crack tip and within the strain lobe. There is a definite increase in strain as compared to locations 4, 5 and 7, with evidence for three active slip systems visible in Figure 4.16a. The average slip band spacing was 85 nm, the lowest of any position. The points where slip bands intersect each other results in the formation of dislocation tangles; examples are indicated by the black arrows on Figure 4.16a. The white arrow in Figure 4.16a and Figure 4.16c indicates places where slip bands intersect the surface to create slip traces. The slip trace in Figure 4.16a is 27.5 nm in height which would have required 158 dislocations impacting the surface. The slip trace in Figure 4.16c is 58.6 nm tall and would have required 333 dislocations to create it. Not every slip band that impacts the surface leaves a slip trace, as seen in Figure 4.16b, and the size of the slip traces is not uniform suggesting that the activity on the planar slip bands is not uniform. Figure 4.16c also includes a low-angle grain boundary, indicated by the black arrowheads. Slip
bands oriented parallel to the grain boundary to appear within 20 nm of the grain boundary, while the intersecting slip bands have corresponding areas of slip in the other grain.

Figure 4.16: a-c: TEM micrographs from Location 6, 80 µm from the crack tip in the strain lobe. The white arrows indicate the locations of surface steps consistent with slip traces, while the black arrows in a indicates the locations of large dislocation pile-ups due to slip band interactions. The black arrowheads in c indicate the location of a grain boundary running through the sample.

The dislocation microstructure showed a general agreement with the surface strain mapping techniques. As the crack tip was approached, the dislocation content of the extracted volumes generally increased. However, at the furthest arcs, comprised of locations 1-3 and 4-5, little difference was seen in the dislocation content from the samples inside and outside the strain lobe. This is evident in the similar spacing of the planar slip bands and the dislocation distribution contained therein, compare Figures 4.8-4.11, 4.14. This does not conform to the DIC map, Figure 4.3, which showed strains of up to 5% at nearly 1.5 µm from the crack tip within the band, but low strains outside of the band, nor does it match the strain increase suggested by the KAM map, Figure 4.5a, which shows the strain is much more diffuse than the DIC map. The lack of higher strains seen in the TEM samples from locations 1, 2 and 4 could be attributed to the higher strains being more localized, and extracted samples missing these regions of higher strain. While the KAM map was used as guidance for the sample extraction locations, the overlay could be inaccurate by several microns and the KAM map lacked the detailed features necessary to further orient the sample location. Since the surface area of the extracted samples was approximately 20 µm by 100
nm, it could be that the small highly strained regions were missed in the sample preparation process. Alternatively, the electron micrographs could simply show that the dislocation distribution is less homogeneous than suggested by either the DIC or the KAM map. In other words, these surface techniques may be overestimating the degree of strain developed and distributed within the band.

The locations close to the crack did show a large difference between the dislocation densities as shown in Figures 4.15 and 4.16. This change has several contributing factors. First, the surface strain is generally higher nearer the crack tip. This statement is supported by the FE calculation which shows a continuous increase of strain as the crack tip is approached, Figure 4.4. Second, the areas of high strain are larger. This is apparent on the KAM map where the high misorientation regions go from being on the order of 3-9 µm² to 24-36 µm². Third, the distance between the areas of higher misorientations are lower. These three factors all combine to ensure that a sample extracted from this region would be more likely to have higher strains.

It is important to note that while the distribution of the dislocations and planar slip bands may change as the crack tip is approached, the microstructure does not change. Each location showed planar slip bands, with no notable change between slip bands near the surface and in the bulk of the sample.

Figure 4.17: SEM images of the preparation process for the crack tip TEM sample; a: crack tip with the Pt strip, black arrowheads, deposited over the volume to be extracted; b: the sample volume after the trenches are dug showing the crack; and c: the extracted sample. The white arrow indicates the crack tip on the surface, the black arrow in b and c indicates a continuation of the crack; the black arrowhead in b and c indicates the same location; and the line marks the approximate position of the crack front.

To compare the dislocation structure formed by the crack tip at the free surface with the structures formed ahead of the crack, a sample was extracted directly from the crack tip, indicated as Location
8 in Figure 4.6. The process of producing this sample is detailed in Figure 4.17. To create this sample, a Pt strip was deposited on the sample surface along the crack direction so that half the Pt strip covered the region ahead the crack tip and half was along the crack in the growth direction. This is seen in Figure 4.17a, where the divot in the Pt strip, indicated by the white arrow, shows the location of the crack on the sample surface, while the black arrowheads indicate the edge of the Pt strip. Channels were then machined on either side of the Pt strip. The cross section of the sample can be seen following the milling of the trenches, Figure 4.17b. This image shows the cross-section on the side, indicated by the black arrowheads in Figure 4.17a. Moving along the crack growth direction, the crack can be seen curving deeper into the sample away from the surface. While this appears to indicate that the crack at the surface lags behind the interior, this actually arises from the nature of this cross-section. While the FIB cross-section is exactly 90° from the sample surface, the crack does not penetrate into the sample perfectly normal. Therefore, when performing a sample extraction such as this, the crack appears in the sample at the location the cross-section intersects the crack flanks. For this sample, the crack nearer the surface can be attributed to the fact that the crack turns at the sample edge. This is indicated by the white arrow in Figure 4.17a, with the same location in Figure 4.17b. The crack is longer away from the free surface, indicated by the black arrow in Figures 4.17b and 4.17c.

Upon subsequent thinning to electron transparency, portions of the crack flanks on the right side were removed. The final sample is shown in Figure 4.17c. The white line on this image shows the approximate location of the crack, prior to the final thinning step. The black arrowheads on Figure 4.17b and 4.17c indicate the same bend in the crack flanks that was preserved, while the black arrow points to the lower portions of the crack also shown in Figure 4.17b. Thus, crack flanks can be captured by this technique, though the three-dimensional change and changes of the crack path make it difficult to directly capture the crack tip in the two-dimensional cross-section.

The dislocation structures immediately ahead of the crack tip are explored in Figure 4.18. Locations from where the three TEM micrographs were taken are shown in the SEM micrograph presented in Figure 4.18c. Figure 4.18a show the area between the main crack running through the sample and the portion of the crack indicated by the black arrow in Figure 4.17b and c. The main crack flank is to the right of the image, while the black arrow indicates the locations of the secondary crack within the image. The region between the cracks consists primarily of ultra-fine
sub-grains. As the crack flank is approached the sub-grain size reduces to approximately 20 nm. The inset diffraction pattern shows that the diffraction spots are split, indicating that rotations exist between the sub-grains. The splitting of the diffraction spots measures approximately 20°. The area immediately in front of the main crack, Figure 4.18b, shows a similar refinement in a more limited range. This refinement only occurs within 400 nm of the crack flank. The inset diffraction pattern shows that the rotations exist between the sub-grains, with peak splitting on the order of 18°. Further away from crack tip the material returns to a planar banded structure, Figure 4.18d. The planar bands are packed more tightly than those seen elsewhere on the surface with an average separation distances of 47 nm. The inset diffraction pattern demonstrates that the rotations are drastically reduced in this area consistent with the change in microstructure from sub-grains to planar bands.

Figure 4.18: a, b and d: Bright-field TEM micrographs of the dislocation structures ahead of the crack tip. c: approximate locations from which the micrographs were acquired. The black arrow on a shows the location of the crack referenced by the black arrow on Figure 4.17b and c.
The dislocation microstructure behind the crack tip is explored in the micrographs presented in Figure 4.19, with the locations of the micrographs indicated in Figure 4.19c. In area a, Figure 4.19a, immediately next to the crack tip, a region of equiaxed sub-grains averaging 20 nm in diameter is found. The inset diffraction pattern shows that misorientations occur between the sub-grains with peak splitting on the order of 20°. The region of equiaxed sub-grains extends 500 nm away from the crack surface where it gives way to a banded structure that lies parallel to the crack surface. This region is imaged in Figure 4.19b; the arrow indicates the same location as the arrow in Figure 4.19a. The white dashed line in Figure 4.19b indicates where the bands bend, corresponding to a bend in the crack flank rather than a rotation due to the presence of a grain boundary. The planar bands do contain some sub-grains, which exist entirely within a band; examples are highlighted with white arrows in Figure 4.19b. The inset diffraction pattern shows a
splitting of the diffraction peaks on the order of 6°. The transition between the equiaxed sub-grains and parallel bands is not instantaneous, but occurs over a distance of roughly 400 nm. Two major microstructural features are noted in the transition zone. First, the sub-grains within the transition region are larger than those in the equiaxed region, averaging roughly 100 nm. Second, sub-grains exist within the planar bands of the material, similar to those highlighted in Figure 4.19b. This is most evident at the location indicated by the white arrow in Figure 4.19a, where the sub-grains bend around the line that the planar bands do. Thus, the transition region has planar bands, but significant rotations and sub-grains exist within the planar bands.

Approximately 2 µm from the crack flank, the microstructure is a dense planar band structure, as seen in Figure 4.19d. The average spacing between the planar bands is approximately 95 nm, though this measurement is complicated by the heavy dislocation content outside of the bands. Additionally, the planar slip bands no longer conform to the direction of the crack flank and two directions are seen, similar to dislocation microstructure seen in Figure 4.16, though significantly more dislocations exist outside the planar structure.

The sample taken from the crack tip shows that the planar slip band microstructure does not extend all the way to the crack flank. Instead, immediately at the crack flank there exists a region of sub-grains on the order of 20 nm that gradually increase in diameter until transitioning back to the planar slip band structure. This transition occurs within 400 nm of the crack flank. The sub-grains are a transformation of the existing microstructure due to the strain associated with fatigue crack advance. Since the sub-grains only exists very close to the crack tip, they would be nearly impossible to observe with either DIC or EBSD. These microstructures occur at the free surface, where the sample is in a plane stress state, and they need to be compared to the microstructure generated in the interior of the sample where the sample is in a plane strain state.

**4.2 The Microstructural Evolution beneath the Fracture Surface in Haynes 230**

In this section, the morphology immediately beneath the fracture surface at different values of $\Delta K_I$ are examined in detail. The examination also includes the effect of features such as twin boundaries, grain boundaries, and carbides on the evolved microstructural state. The locations from which samples were extracted are indicated, and the details of the microstructure revealed from the cross sectional extraction are described.
An overview of the fracture surface is shown in the SEM fractograph presented in Figure 4.20a. Striations are visible and are prominent in the flat central portion of the image, indicated by the white arrow, but are also visible in the various ridges. The other prominent features on the fracture surface are carbides, which are primarily visible as dark marks on the sample surface. The interaction between carbides and crack growth was not the focus of this thesis and not extensively studied. From images such as this one, it appears that carbides resist crack growth in some manner,
making crack growth occur around them. An example of this phenomenon is highlighted by the black arrows in Figures 4.20a and b. The $\Delta K_1$ at this location was approximately 42.2 MPa$\sqrt{m}$.

Figure 4.20b shows a Pt strip on striations from the center of Figure 4.20a. The strip lies parallel to the crack growth direction and covers numerous striations. The striations are evenly spaced, although measurement of the spacing required TEM examination. The striations visible on the flat portion are continuous, with striations visible in the background and foreground ridges indicated by the black arrows, suggesting a continuity of the crack growth across the various ridges of the sample. The extracted volume with the protective Pt strip on top is shown in Figure 4.20c. Planar slip bands extend from surface to the end of the sample, indicated by the white arrow. Figure 4.20d shows a section of the fracture surface of the extracted volume at higher magnification; the corresponding location is indicated by the black arrow in Figure 4.20c. Striations are visible on the fracture surface before giving way to regions of varying contrast consistent with sub-grains showing minor rotations; the black arrow points to this region. Below this region, planar slip bands are seen moving away from the surface roughly 30° from the vertical, and slip bands that lie parallel to the fracture surface are visible. An example of these bands is indicated by the white arrow in Figure 4.20d.

The microstructure beneath the striations on the fracture surface is shown in the electron micrographs presented in Figure 4.21; the location from which the samples were extracted is shown in Figure 4.20. The same location is shown in the bright-field and dark-field TEM micrographs presented as Figure 4.21a and b, respectively. These show that the area immediately beneath the fracture surface consists of ultrafine sub-grains. This gives way to a region of parallel planar slip bands that are inclined with respect to the fracture surface. These bands are spaced approximately 200 nm apart, while the average striation spacing is approximately 500 nm. The white and black arrows in Figures 4.21a and b show the same location across the grains. Figure 4.21c shows that areas between the slip bands have a high density of dislocations. The micrographs detail that the extent of the refined region is approximately 400 nm. Variations in the depth are not correlated to individual striations. Two or more sub-grains can occupy the volume of the refined zone. The inset diffraction patterns in Figure 4.21c confirms that splitting of the diffraction peaks, about 15°, occurs near the fracture surface within the refined zone. This confirms that the sub-grains are rotated with respect to each other. In contrast, the diffraction pattern from the region
showing the planar bands exhibits little to no splitting of the diffraction spots, indicating that the misorientation between the bands is small.

![Figure 4.21](image_url)  
Figure 4.21: Microstructure beneath striations detail in Figure 4.20. *a:* bright-field micrograph and *b:* dark-field micrograph of the same area; and *c:* bright-field micrograph with inset diffraction patterns; The white and black arrows indicate the same location in *a* and *b,* while the black arrow in *d* indicates the presence of planar slip bands running parallel to the fracture surface. The inset diffraction patterns on *c* detail the amount of peak splitting at the surface and bulk of the sample.

To confirm that the microstructure detailed in Figure 4.21 was consistent across the fracture surface, a second sample was extracted. The second sample was extracted from a region at a lower stress intensity factor (34.4 MPa√m) that was not as flat as the previous region. An SEM image, Figure 4.22a, shows the Pt strip on the region of the fracture surface to be extracted. The microstructure beneath the fracture surface is shown in Figure 4.22b. A refined layer exists immediately beneath the fracture surface and it extends to a depth of 300 nm. Below this layer a parallel planar banded structure exists. The striation spacing was approximately 500 nm, while the planar band spacing was approximately 100 nm. The inset diffraction patterns in Figure 4.22c compare the near surface with the banded structure. As before, the diffraction spots are split and rotated in the near surface region but not in the planar banded region. On closer examination of the diffraction spots from the refined layer it is noticed that the diffraction spots align with spots from the parallel band structure at certain points. These points are indicated by the arrows. This comparison shows that the splitting and rotation of the diffraction spots is not uniform about the
spot but that it is rotated in the clockwise direction by 38°. This suggests that the rotations are likely caused by shearing in one direction, and the sub-grains are formed from the deformation of the planar banded structure. The electron micrograph presented in Figure 4.22b demonstrates that some of the parallel planar band structure penetrates through the refined layer to the sample surface; examples are indicated by the black arrows. However, most of the bands terminate at the refined structure.

Figure 4.22: A second sample extracted at the sample surface at 34.4 MPa√m. a: an SEM image showing the location from which the sample was extracted. b-c: Bright-field TEM micrographs showing microstructure beneath striation at this location. The black and white arrows in b indicate locations where the planar slip band microstructure bends in the refined zone and continues to the surface respectively. The inset diffraction patterns on c shows the rotational differences between the refined region and the bulk, with the white arrows indicating the location \{111\} spots within the bulk.

The dislocation structures beneath the surface parallel to the striations are explored in Figure 4.23. The fracture surface from which this sample was extracted is shown in Figure 4.23a. A twin boundary runs through the center of the image. Striations are continuous across the boundary, but there is a 40° difference in their angle. The dotted line indicates the approximate location from which the sample was extracted. The surface of the extracted sample, seen in the bright-field TEM micrograph presented as Figure 4.23b, has slight variations in the sample height of less than 20 nm, suggesting that the sample is slightly off parallel to the striations. Immediately beneath the surface, there is a continuous dislocation structure showing planar slip bands. There is no evidence
of sub-grains nor a refined layer at the surface. There is a surface region that extends for approximately 1 µm away from the fracture surface that is misoriented from the bulk of the sample by approximately 15°. This misorientation is evident from the diffraction patterns. While the bulk of the sample is oriented along a [111] zone axis, the surface is significantly off that axis. Away from the surface, the sample continues to show planar slip bands, as seen in Figure 4.23c. The boundary imaged in Figure 4.23a is also presented in Figure 4.23d. The inset diffraction pattern shows that this is a twin boundary. Both sides of the boundary show planar slip bands with a minor change in the direction of the planar slip bands on either side of it.

![Image](image.png)

Figure 4.23: Microstructure beneath striations as viewed parallel to the striations. **a:** SEM image of the location which the sample was extracted. The dotted line, indicates the approximate sample location. **b:** Bright-field electron micrograph with comparison of SADP from specific locations; **c:** planar slip bands in the bulk; **d:** Bright-field electron micrograph at a twin boundary.
4.3 The Microstructure of an Arrested Crack on the Fracture Surface in Haynes 230

Figure 4.24: SEM images of the fracture surface during the change from FCG to MVC at \(a\): 0° and \(b\): 54° of tilt. The arrows indicate the crack propagation directions. The dotted line in \(b\) indicates the area from which a TEM sample was extracted.

In the series of electron micrographs presented in Figure 4.18 and Figure 4.19, the microstructure around a crack from the free surface was examined. To demonstrate that the microstructure ahead of the crack was similar on the free surface and within the bulk, the arrested crack was loaded in tension to failure. The working hypothesis was that the fast rupture should leave the microstructure immediately ahead of the fatigue crack intact. However, before examining that sample in detail, a sample loaded in fatigue to failure was examined to establish a baseline expectation of the microstructural evolution across loading modes. The morphology of the fracture surface is shown in the fractograph presented in Figure 4.24a. For a region over 300 \(\mu m\) long, both microvoids and striations are visible. Along the direction of crack growth, indicated by the arrow, the microvoids increase in both number and area density, while the striations become broader and less uniform. Each microvoid has a carbide associated with it, suggesting that carbides cause local stresses high enough to initiate microvoid formation, but in the absence of microvoids, crack growth reverts to the striation mechanism. This conclusion is supported by the SEM fractograph presented in Figure 4.24b, which is a tilted view of a portion of the 300 \(\mu m\) long transition zone. The striations in the center of the image are separated by a large microvoid wall associated with the string of carbides; examples of the carbides are marked with arrowheads.
Figure 4.25 An overview of the microstructure of the MVC region. *a*: an SEM micrograph showing the Pt strip on the surface; *b*: a bright-field STEM micrograph overview; and *c*: a bright-field STEM image of the surface. The white arrow in *b* indicates where a planar slip band extends from the bulk all the way to the surface.

The microstructure beneath striations has already been presented, Figures 4.21 – 4.23. The microstructure beneath a microvoid was determined by extracting a sample from within the microvoid; the exact location from which the sample was extracted is shown in Figure 4.25a. The microstructure beneath this sample is shown in the micrographs presented in Figure 4.25b and c. The bright-field diffraction contrast STEM micrograph, Figure 4.25b, displays an overview of the microstructure. Immediately beneath the fracture surface, an area of refined sub-grains exists; these sub-grains are shown at higher magnification in Figure 4.25c. These sub-grains are on the order of 20 nm. Below this refined microstructure, the material moves to planar slip bands elongated parallel to the fracture surface. This is a departure from the typical microstructure beneath striations in which the planar slip bands are oriented ~15° off perpendicular from the fracture surface. This region extends for a distance of 2.5 µm away from the fracture surface where it transitions to another region featuring planar bands. These slip bands are oriented 15° off perpendicular as seen beneath striations. Some of these bands penetrate through the other parallel banded structure, indicated by the white arrow in Figure 4.25b. These images suggest that the MVC region has extensive plasticity that is locally confined to the region near the crack surface before returning to the planar band structure typical of the fatigue response of the material.
Figure 4.26: Bright-field TEM micrograph of the microstructure beneath the transition zone in the sample failed under fatigue loading. The crack growth direction is from left to right on this image. The extracted volume is indicated on Figure 4.24b by the dashed line. The white arrowhead indicates a line of dislocation structures separating two different planar band structures. The boxes indicate regions examined in detail in Figure 4.27.

Figure 4.27: a-c: Bright-field TEM micrographs comparing the microstructure in the boxed areas in Figure 4.26. The diffraction patterns from both the surface and bulk are shown below each micrograph. The black arrowheads indicate the location of the line of dislocation features separating the planar slip structures, while the black arrows indicate the crack advance direction.

With the microstructure beneath striations and microvoids identified the microstructure in the transition zone of the sample failed under fatigue loading was examined. The location from which this sample was extracted is indicated by the dashed line on Figure 4.25b. An overview of the underlying microstructure is shown in the montage of bright-field electron micrographs presented in Figure 4.27. The striations are visible on the surface and increase in both height and spacing.
along the crack growth direction, left to right. The striations are initially 650 nm apart and 150 nm in height and this gradually increases to 2.5 µm apart and over 400 nm in height.

Three distinct regions are evident in the montage. The first consists of refined sub-grains immediately next to fracture surface. The second region consists of parallel planar slip bands; this region is similar to the regions seen beneath other fracture surface. The parallel planar slip band structure initially extends all the way to refined surface layer. This is gradually replaced by the third structure which consists of curved slip bands moving in multiple directions. The line separating the two is the series of dislocation features indicated by the white arrowheads.

Closely examining the boxed areas from Figure 4.26 allows for a more detailed view of the changes in microstructure that occur along the crack growth direction. Figure 4.27a, a bright-field electron micrograph, shows three different structures. The structures consist of a refined sub-grain structure that starts immediately at the fracture surface and continues for a distance of 200 nm, less than was seen underneath striations. Following this structure, an area of curved planar band structure is present. This structure is separated from the bulk parallel planar band structure by a line of dislocation cells. The line separating the parallel banded structures from the curved structure is indicated by the arrowheads. The line originates at the bottom of the step seen on the left side of the surface, indicated by the first black arrowhead. The line initially runs parallel to the fracture surface but after 2 µm it begins to slope away from it. The diffraction patterns show minor peak splitting at the fracture surface on the order of 5°, but little in the bulk indicating a relatively low level of plasticity.

The difference between the curved structure and the parallel slip bands is more obvious in Figure 4.27b. The curved planar band region extends 3 µm from the fracture surface. The line of dislocations separating the two banded structures is indicated by arrowheads. The refined microstructure at the surface now penetrates to an approximate depth of 500 nm. The diffraction patterns from the surface and the bulk both show an increase in the amount of diffraction spots splitting from their counterpart in Figure 4.27a, with the surface diffraction pattern showing an increase to approximately 14° of spot rotation and the bulk showing approximately 5°.

The bright-field TEM micrograph presented in Figure 4.27c demonstrates that the surface strain continues to increase along the crack growth direction. The line of dislocations separating the planar band structures is no longer visible, it is only the curved structure that is imaged in Figure
4.27c. This region now extends over 5 µm into the sample. The depth of the refined surface region remains at 500 nm, but the diffraction pattern from this region shows a further increase in strain, as the pattern forms a ring. However, regions of higher spot intensity exist, suggesting that there are preferred orientations within the refined region. The bulk diffraction pattern shows a minor increase in strain as the peak splitting is roughly 6°, slightly higher than that seen in Figure 4.27b. These results suggest that the increase in strain associated with the larger striations is primarily concentrated at the surface and the near surface region. The change from the parallel planar banded structure to the curved structure could be an influence of Stage III crack growth mechanics.

The results presented in Figures 4.18, 4.19, 4.21 and 4.22 all show the dislocation structure around the crack flank, both from the free surface (4.18 and 4.19) and the interior of the sample (4.21 and 4.22). In those samples, it was found that the microstructure transformed to a planar slip band structure with increasing distance from the crack flank. This structure had two possible modes of formation: as part of the crack advance, or following the crack advance due to the fracture surfaces rubbing together during plasticity-induced crack closure. The TEM sample extracted from the transition between the FCG region and the MVC region was an attempt to examine the crack tip without the influence of crack closure. The high stress intensity factor of these sample and the extended transition period between crack growth modes prevented a direct comparison between the microstructure of these samples and those from the FCG regime. To solve this issue and directly compare the fracture surface samples with a crack tip that did undergo crack closure, the arrested crack described in Figures 4.3-4.19 was reloaded in monotonic tension. The fracture surface resulting from this reloading is presented as Figure 4.28.

Figure 4.28 Fracture surface following the change in loading mode. a: overview fractograph; b: higher magnification of the boxed region; and c: an examination of the area indicated by the black arrow in b tilted at 54°. The white arrows in a indicate the point where the crack growth mechanism changed from fatigue crack to microvoid coalescence. The white arrows in b indicates the point where the transition began. The dotted line in c represents where the transition between growth modes occurred.
Figure 4.28a shows the fracture surface over the area where the change in loading mode occurred. The lower portion of the figure shows striations consistent with fatigue crack growth and the upper has primarily microvoids with an abrupt shift between the two features indicated by the white arrows. The boxed area across the transition zone from Figure 4.28a is shown enlarged in Figure 4.28b. When viewed in detail, the sample now shows evidence of a transition between the two fracture morphologies. The striations from the FCG region end at the white arrow and this is accompanied by an abrupt change in contrast. The striations leading up to the transition zone show no change or broadening akin to that observed in the sample failed under cyclic loading conditions. This indicates that the end of the striations is the final location of the crack tip on crack arrest, which occurred at 53,335 cycles and a ΔK of 35 MPa√m, and thus, the point at which monotonic loading was initiated. Between this location and the start of the microvoids, there are several ripples on the fracture surface that are similar in appearance to, although less regular than, striations. These ripples are seen most clearly in Figure 4.28c, which is a view of the area indicated by the black arrow in Figure 4.28b but tilted 54°. The change in the loading mode is outlined by the dotted line; the striations leading up to it are relatively uniform in spacing and height while the ripple features are varied in both spacing and height. This image also shows that the change in the loading mode is accompanied by an increase in the height of the fracture surface.

![Figure 4.29](image)

Figure 4.29: The transition between striations and microvoids following the change in loading mode. a: fractograph of the fracture surface in the transition region; b: higher magnification of the boxed region in a; and c: the extracted volume. In b and c, the black arrow indicates the location of the loading mode change, the white arrow indicates the location of the microvoid wall and the numbers reference each of the large ripples.

To achieve the twofold goal of confirming the dislocation structure at the crack tip was the same at the sample surface and the bulk, and interrogating the microstructure of the transition region to examine the cause of the ripple features, a TEM sample was extracted directly from the transition
region such that it contained all three crack growth morphologies. Figure 4.29a, a SEM image, shows an overview of the area from which the TEM sample was extracted. Striations are plainly visible in the lower left of the image and these give way to microvoids in the upper portion of the image. The boxed area indicates the location from which the sample was extracted and it is explored in further detail in Figure 4.29b. The black arrow in Figure 4.29b indicates the location at which the striations end, which marks the loading mode change. This location also has a contrast change with the striations appearing notably darker than the ripple features, which is consistent with the macroscale change in surface reflectivity between the two crack growth modes [9]. Following the end of the striations, ripples are visible on the fracture surface. There are 5 large and prominent ripples spaced approximately 3 µm apart leading up to a microvoid wall, which is indicated by the white arrow. As is evident from Figure 4.29c, these features are preserved in the extracted volume following thinning to electron transparency; the black and white arrows point to the same features as in Figure 4.29b.

![Figure 4.29a](image1.jpg) ![Figure 4.29b](image2.jpg) ![Figure 4.29c](image3.jpg) ![Figure 4.29d](image4.jpg)

Figure 4.30: Microstructure beneath the fracture surface across the transition zone associated with the change in the loading mode. a: Bright-field overview image; b: fatigue crack growth; c: transition region; and d: microvoid regime. The black and white arrows indicate the same locations as in Figure 4.29. The black arrowhead indicates the line of dislocations separating the fracture surface microstructure from the planar band microstructure.
The microstructure of the extracted volume from Figure 4.29c is presented as Figure 4.30. The montage of the bright-field TEM micrographs presented as Figure 4.30a explores the dislocation structure associated with the three different fracture surface morphologies seen in Figure 4.29. The FCG region, which is examined in detail in Figure 4.30b, shows striations with an average spacing of 410 nm and a height of 60 nm. The final striation mark is indicated by the white arrow. Underneath the striations the sample shows a refined microstructure similar to that seen in Figure 4.18; here it is limited to within 300 nm of the fracture surface. This refinement at the surface extends approximately 150 nm past the peak of the last striation. The surface microstructure is then replaced by a region showing no distinct dislocation cells or sub-grain boundaries. Later investigations, detailed in Figure 4.32c, suggest that the surface layer past the refined region consisted of tightly packed dislocation cells. Beneath the refined layer, there exists planar slip bands approximately 200 nm apart, similar to those seen in Figure 4.21.

Following the last striation peak, the crack enters the transition morphology which extends for 17 µm. On the fracture surface, the ripple features identified in Figure 4.29 remain visible and are labeled on Figure 4.30a. These ripple features are spaced 3 µm apart and have a height variation of 250 nm; this spacing of the ripples is consistent with that measured from the fractograph, Figure 4.29. Across the transition zone, the microstructure has two distinct regions: the near surface region and the planar banded region. These two regions are separated by a line of dark contrast, analogous to the line of dislocation features separating the parallel and curved planar banded regions in Figure 4.27. This line, indicated by the black arrowhead in Figure 4.30a, extends across the transition region, staying parallel to the crack growth direction until it reaches the 3rd ripple where it suddenly changes direction and penetrates into the sample before disappearing at the 4th ripple. This change in the line direction coincides with a change in the bulk dislocation structure away from planar slip bands oriented parallel to each other and 70° to the fracture surface, to one showing planar slip bands oriented in three different directions that are prominently curved. This microstructure is similar to the curved planar band structure observed in Figure 4.27. While the parallel banded microstructure extended approximately 12 µm from the end of the FCG to the 3rd ripple feature, the microstructure that replaces it extends only 6 µm until the fifth ripple feature where it is replaced by a microstructure that contains a mix of dislocation cells and planar slip bands, likely caused by the plasticity of the approaching microvoid.
The microvoid causes changes in the microstructure due to the nature of stress around it. In fatigue crack growth the stress is almost always oriented along the direction of crack growth. This creates the microstructure seen in Figure 4.21, in which the prominent planar bands are oriented in the crack growth direction. However, the local stress field associated with a growing microvoid is spherical. This creates stress with respect to the direction of the microvoid walls. Examination of Figure 4.29 suggests that the microvoid wall is oriented 35° with respect to the direction of the extracted volume. Moreover, it assumes this direction at the same location as the 4th ripple. Since the microvoid wall is not parallel to the sample it will create significant stresses and strains out of the plane of the sample. This will result in planar slip bands oriented with respect to the microvoid wall. This explains why the microstructure begins to deviate from the transition zone microstructure at the 4th ripple, as now there is a significant component of the strain associated with the formation and growth of the microvoid. The amount of out of plane strain increases as the microvoid wall is approached, likely creating planar slip bands along this direction. Since the planar slip bands are oriented out of plane with the sample, they appear to be spread over multiple nanometers creating the microstructure associated with the microvoid wall.

The line separating the surface structure and the parallel slip bands is the focus of Figure 4.30c, which is a higher magnification image of the area leading up to the first ripple feature. The line is distinguished as a series of dark features. Planar slip bands are visible coming from the bulk of the sample, but these terminate upon hitting this line. While this line does not appear to be continuous, see the location indicated by the white arrow, the planar slip bands do not penetrate through these gaps. The origin of this line can be found in Figure 4.30b, where it begins slightly after the last striation peak. This suggests that the line is entirely a product of the monotonic loading of the sample.

The final microstructure presented is that associated with the microvoid wall, indicated by the white arrow on Figure 4.30a, and featured in Figure 4.30d. The bulk of the sample consists of dislocation cells on the order of 500 nm, however, past the peak of the microvoid wall, the fracture surface features a series of equiaxed sub-grains on the order of 20 nm.
To examine the evolution of strain along the surface of the transition zone, selected area diffraction patterns were taken across the sample at the surface and in the bulk. The locations from which these patterns were acquired are highlighted in Figure 4.31a. The SADPs from the surface regions, Figures 4.31b, c and d, all show evidence of splitting of the diffraction peaks. The amount of peak splitting increases along the transition zone, it measures 19.5°, 23.5° and 26° at locations b, c and d, respectively. This indicates that the surface strain increases across the transition zone. The diffraction patterns taken from further away from the fracture surface, Figures 4.31e, f, and g, also show evidence of peak splitting, but it does not vary with location; the rotation of the diffraction peaks measures 8°, 8.5° and 8° at locations e, f and g, respectively. This suggest that while the strain increases at the sample surface across the transition zone, it remains uniform in the bulk despite the change in microstructure associated with the microvoid wall.

To determine the microstructural features that account for the distinct dark band seen in Figure 4.30c, this feature was further examined and was found to be composed of dislocations. The bright-field STEM micrograph presented in Figure 4.32a provides an overview of this distinct line. The line is indicated by the arrowhead in Figure 4.32a and its distance with respect to the fracture surface gradually increases with distance along the transition zone. As the planar slip bands approach this line, they curve in the direction of crack growth. This curvature is highlighted by comparing the black and white lines in Figure 4.32a; the black line traces the original slope of a planar slip band, while the white line traces the actual path. The deviation begins approximately 1 µm from the fracture surface and continues until the planar slip band impacts the dark line.
Figure 4.32: Identification of the structure of the line of dark contrast. a: Bright-field STEM micrograph at the initiation point; b: enlarged view of boxed region in a showing the interaction of the planar slip bands with the dark contrast line; and c: sub-grains near the surface close to the 3rd ripple in the transition zone. In a, the arrowhead marks the initiation point of the line of dark contrast, the black line lies parallel to a planar slip band whereas the white line shows the actual curved path.

This line is shown at higher magnification in Figure 4.32b. This micrograph shows the line contains features resembling dislocation cells. The planar slip bands are seen to terminate at the dislocation cells that form the line and do not penetrate them. The planar slip bands are very tightly spaced with an average spacing of 10 nm.

The final micrograph, Figure 4.32c, shows the near surface microstructure from the location just beyond the 3rd ripple. This microstructure consists primarily of sub-grains with dimensions of less than 100 nm that appear to share the same curvature as the planar slip bands as they approach the line of dislocation cells. Even though parts of this area are now in contrast, significant portions are out of contrast, such as the cell indicated by the black arrow. This observation suggests that large rotations exist between the sub-grains. Since sub-grains were found on the surface, and are the primary component of the line separating the surface region from the planar slip, this suggest that the surface layer of the transition region is primarily composed of sub-grain structures, but they are not in contrast in these images.

In summary, an arrested crack was reloaded under monotonic tension until failure. The location of the arrested fatigue crack tip was found on the fracture surface and the microstructure beneath the transition zone determined by TEM analysis. At the location of the crack tip, the microstructure was similar to that found under striations, suggesting that it was a product of fatigue crack growth.
Beneath the ripples, the bulk microstructure was similar to that beneath striations marks, but the surface microstructure was now composed primarily of dislocation cells. With increasing distance along the fracture surface, the microstructure continues to increase in complexity as additional slip systems are activated.

4.4 The Microstructure of Copper Fatigue Cracks at the Free and Fracture Surfaces

The microstructure developed on fatigue loading is perhaps best understood in copper [10-14]. The microstructure beneath fatigue striations in copper was examined to determine if it exhibits similar microstructural features as observed in Haynes 230.

![Figure 4.33](image.png)

Figure 4.33: *a*: SEM image of the free surface contain the crack tip; *b*: SEM micrograph of the extracted volume with the crack tip; *c*: a bright-field TEM micrograph of the crack tip, and *d*: a bright-field TEM micrograph further away from the crack tip. The white arrow in *c* indicates a dislocation cell multiple microns in diameter, and the white arrowhead in *c* and *d* indicates the same location.

Figure 4.33a, a SEM image, shows the area surrounding a crack tip in copper. Prominent slip traces are visible, though only one direction appears visible in each grain. A sample was extracted directly from the crack tip in a process similar to that detailed in Figure 4.17. As such, the same concerns
involving the reduction of a three-dimensional figure to a two-dimensional sample apply here. The extracted volume is shown in the SEM image, presented as Figure 4.33b. Variations in contrast are visible across the volume due to slight rotations of the crystal. As the crack tip is approached, the contrast variations decrease in size and increase in intensity, suggesting the near crack tip region is more highly deformed. Additionally, the crack tip is branched, with one branch indicated by the black arrow and the other by the white. The cause of this branching was not determined. The microstructure associated with the branch indicated by the black arrow is shown in the bright-field TEM micrograph presented as Figure 4.33c. This micrograph shows that the dislocation microstructure surrounding the crack tip is composed entirely of dislocation cells. In contrast to the Haynes 230, heavy refinement of the microstructure is not see at the crack flank, though the size of the dislocation cells does decrease as the crack tip is approached. The dislocation cells next to the crack are approximately 250 nm in diameter, but cells away from it can be multiple microns in diameter, one such dislocation cell is indicated by the white arrow on Figure 4.33c. The area between the two branches of the crack is explored in Figure 4.33d; the upper branch, indicated by the white arrowhead, shows the same location on both Figure 4.33c and 4.33d. The size of the dislocation cells is smaller between the crack branches than the bulk, averaging 500 nm, indicative of higher strains. The inset diffraction patterns from the crack flank and the bulk of the material suggest that there is slightly more deformation at the crack tip than further away, since the diffraction spots in the SADP from the crack flank had 11° of peak splitting compared to 3.8° in the bulk.

The microstructure from the arrested crack tip on the free surface was compared to the microstructure beneath the fracture surface. The SEM micrographs of the fracture surface, Figure 4.34a and b, show the surface state. While Figure 4.34a might appear to show striations, closer inspection reveals that these are primarily slip traces. This is because the bands run in three directions and are irregularly spaced. Each of the different directions are indicated by the black arrows. The actual striations, visible in Figure 4.34b, are more tightly spaced than the slip bands and appear very faint, with TEM observation showing the striation spacing being approximately 250 nm and the height 20 nm.
The bright-field TEM micrograph presented as Figure 4.34c shows that the primary structure away from the fracture surface is dislocation cells. The lines running across the dislocation cells are artifacts from the FIB preparation process. The dislocation cells did not have any particular orientation. The dislocation cells decreased in size as the fracture surface was approached. Figure 4.34d shows the fracture surface, with numerous deformation twins originating from the fracture surface extending 300 nm into the bulk of the sample, as confirmed by the inset SADP. The inset diffraction pattern also suggests that little to no rotation of the diffraction spots occurs at the fracture surface, in contrast to the results obtained in Haynes 230, see Figure 4.21. The deformation twins were only found in one sample and were oriented 90° with respect to the fracture surface, suggesting that a preferential grain orientation was necessary before the twins would form off the surface. Figure 4.34e, a bright-field TEM micrograph, shows the area beneath the fracture surface in a sample that did not have deformation twins. The size of the dislocation cells decreases from
500 nm to less than 200 nm as the fracture surface is approached. This cell size decrease initiates roughly 500 nm from the fracture surface. The inset diffraction patterns show that little peak splitting occurred in the bulk of the material, but that about 12° of peak splitting occurs at the surface, in contrast with the region showing deformation twins.

Figure 4.35: The change in misorientation of the dislocation cells near the fracture surface; a: Bright-field electron micrograph of the surface with the boxes highlighting region where SADPs were obtained; b-d: SADP from the highlighted areas.

To investigate the amount of strain localization that occurred at the fracture surface diffraction patterns were taken at intervals away from the fracture surface at one location. The area is shown in Figure 4.35a. The dislocation cells on the fracture surface are similar in size to those found on Figure 4.34e, being approximately 250 nm in diameter. Three different SADPs were obtained such that one overlapped the fracture surface and the others were acquired 150 nm and 300 away from it, respectively. The SADPs showed a continuous increase in the strain as the fracture surface was approached. The SADP from the region at 300 nm from the fracture surface, Figure 4.35b, shows the diffraction spots are rotated by 7°. At 150 nm from the fracture surface, the SADP, Figure 4.35c, the rotation of the diffraction spots increases to 14°. The final location, directly over the fracture surface is shown in Figure 4.35d and had 21° of peak splitting. Interestingly, the peak splitting occurring in Figure 4.35d is not continuous like in the other spots, but instead discrete spots are see at the different points. This suggests that the rotations associated with the peak splitting are primarily due to rotations of the dislocation cells with respect to each other and not rotations of the interior of the cells. In addition, the location of the (200) spots has rotated by approximately 30° from the diffraction pattern shown in Figure 4.35b.
Figure 4.36: The results from the Transmission EBSD study on the crack tip: 
a: the crack tip with the orientation map overlaid to give context for the approximate scan position; 
b: the orientation map of the crack tip; 
c: the KAM map of the area near the crack tip; 
d and e: misorientation profile maps obtained along the directions indicated by the arrows in b; the arrows on e indicate a pair wild spike due to misindexing of several points.
To further investigate the rotation of cells a Transmission EBSD (T-EBSD) scan was obtained from the crack tip featured in Figure 4.33. The benefits of T-EBSD include smaller step sizes and higher diffraction pattern quality [15-17]. The T-EBSD scan was obtained utilizing a JEOL 7000F microscope and Oxford instrument HKL Software for reconstruction. The sample was tilted 20° in accordance with techniques noted in literatures [15-17] and the step size was 20 nm.

Figure 4.36 shows the results of T-EBSD investigation into copper. Figure 4.36a shows the area near the crack tip with orientation map overlaid, to provide context for the location of the scan in relation to the crack tip. The two branches of the crack tip are indicated with arrows; these location are indicated on the subsequent images. Figure 4.36b shows the orientation map near the crack tip. The bulk orientation, seen away from the crack tip, has a plane normal between {100} and {111}. As the crack tip is approached, this plane normal changes into a {110} type above the crack, while changing into a {100} type below the crack. The change in the orientation is due to misorientations between the dislocation cells. Dislocation cells are visible in Figure 4.36b as areas of similar orientations. These are separated between each other by white spaces, where the software was unable to index properly. These white spaces are the dislocation cells walls. The indexing failure was due to the high density of dislocations contained within the cell walls. The KAM map, Figure 4.36c shows that the misorientations within the dislocation cells were low, while the misorientations near the cell walls were much higher. This is consistent with the TEM results that showed few dislocations inside the cells. Two misorientation profiles are presented as Figure 4.36d and e. These misorientation profiles show that change in misorientation that occurs relative to the origin point as the crack tip is approached. The locations from which these misorientation profiles were obtained are indicated on Figure 4.36b. The first misorientation profile, Figure 4.36d, shows follows the change in misorientation in front of the crack. Three different dislocations are capture in this profile; the first as a misorientation of 4° from its neighbor, while the second (nearer the crack tip) has a misorientation of 8°. The second misorientation profile traces the changes behind the crack tip, crossing numerous dislocation cells. Several spikes are see in the misorientation profile; these spikes are likely due to misindexed points and are disregarded with respect the following discussion of how misorientation between the cells change. The misorientation are low between the cells (between 2° and 5°) until the point approximately 1.5 µm from the origin where the misorientation becomes on the order of 10-15°. Both these misorientation profiles indicate that the rotations between the dislocation cells becomes higher as the crack tip is approached.
In summary, copper showed a consistent decrease in the dislocation cell size as the crack tip was approached and an increase in the rotations of the cells with respect to each other. Both of these are indications of higher strain near the crack. In particular, the diffraction patterns suggest that the rotation of the dislocation cells was relatively small until within 300 nm of the fracture surface suggesting that strain is localized to the crack tip. This rotation of the dislocation cells was also noted to occur ahead of the crack tip.

4.5 The Microstructural Evolution of a Fatigue Crack on the Free Surface in Nickel

![Image](image)

Figure 4.37: Surface crack in nickel. a: SEM image of the crack; b: SEM image of the crack tip with prominent slip bands visible; and c: SEM image of the cross section of the extracted volume showing the crack tip. The white arrowheads in b indicate steps out of the crack growth plane in the direction of the slip traces seen in the lower grain. The white arrow in c indicates the area showing varying contrast indicative of local rotations.

To corroborate the localization of strain seen in copper with another FCC material that exhibited wavy slip, single edge notched tension bars of pure nickel were fatigued. When the crack had grown to a sufficient length, the test was halted, arresting the crack, and a sample was prepared from the crack tip following the method described in Figure 4.17. Figure 4.37a, an SEM micrograph, shows the crack after 225,000 cycles. The crack has grown 300 µm from the notch and the stress intensity factor is 18.7 MPa√m. The crack tip is shown in Figure 4.37b. The crack path is jagged with frequent steps, which are indicated by the white arrowheads. These steps are typically in the direction of the slip traces seen in the lower grain of this image. The crack and an annealing twin are visible in Figure 4.37c, which is a SEM image of the volume extracted for analysis in the TEM. Surrounding the crack tip are contrast variations consistent with small local rotations due to strain. The dislocation structures surrounding this crack tip are further examined in Figure 4.38.

The dislocation structure surrounding the crack tip detailed in Figure 4.37 is explored in Figure 4.38. Figure 4.38a shows the crack tip and the dislocation cells that surround it. The dislocation
cells immediately next to the crack tip average 300 nm in diameter, with the cell diameter increasing to 500 nm away from the crack tip. The inset diffraction pattern from directly next to the crack flank shows that a moderate amount of peaking splitting, about 5°, occurs, while the inset diffraction pattern from the bulk shows no visible peak splitting. Ahead of the crack tip, Figure 4.38b, the dislocation cell size increases to approximately 750 nm. This size suggests that the crack advance refines the dislocation cell size from this to the levels seen in Figure 4.38a. The dislocation cells next to the crack flank are the focus of Figure 4.38c. This micrograph confirms that the dislocation cells are approximately 300 nm next to the crack tip and these increase to 500 nm a short distance from the crack flank. The minor variations in contrast between the dislocation cells support the inset diffraction pattern from Figure 4.38a, suggesting that the rotational difference between the cells is small.

Figure 4.38: Dislocation structures surrounding the surface crack tip in nickel following 225,000 cycles; a: ahead of the crack tip; b: on the free surface away from the crack tip; and c: structure directly next to the crack flank.

Figure 4.39: The crack tip in a nickel after 500,000 cycles. a: overview SEM image; b: crack tip showing intense slip traces ahead of it and in association with the striations on the fracture surface; c: and the extracted volume. A grain boundary runs through the extracted volume. The black lines running through these images are the result of a software error during acquisition
The arrested crack following 500,000 cycles is shown in Figure 4.39a; the crack has grown 1.9 mm and the stress intensity factor is 34.7 MPa√m. The tip of the crack is shown in greater detail in Figure 4.39b. The crack tip is visible and prominent slip traces lie in the direction of crack advance. Interestingly, the lower edge of the crack is bent in such a manner that the striations on the fracture surface are visible. This allows for a comparison between the slip traces on the specimen surface and the striations on the fracture surface. Within the boxed region there were 4 visible striations but 7 visible slip traces suggesting that multiple slip traces are associated with each striation mark. The spacing between striations here is on the order of 1.2 µm suggesting that very large strains are present. Since the area containing a closed crack tip is small, the sample was extracted perpendicular rather than parallel to the crack growth direction. The extracted volume is shown in Figure 4.39c; this image is oriented such that the left is the same direction as down from Figure 4.39a and b. While the crack tip was not captured in this sample a grain boundary was seen running through the center of it and its location makes it very near the crack tip. The high levels of strain are confirmed in this micrograph as there are large contrast variations across the sample.

Figure 4.40: The dislocation structure from the arrested crack in nickel. Bright-field electron micrograph of the microstructure in a: the left grain; b: the grain boundary; and c: the right grain.

The dislocation structure associated with the highly strained crack tip detailed in Figure 4.39 are presented in Figure 4.40. To maintain consistency between Figure 4.40 and Figure 4.39c, the images are organized with the right grain as Figure 4.40c and the left grain as Figure 4.40a. However, understanding Figure 4.40a requires first understanding the dislocation structure in Figure 4.40c and consequently it is described first. In the right grain, Figure 4.40c, the dislocation cells are elongated vertically toward the free surface and are visible throughout the grain, including
the right side of the grain boundary in Figure 4.40b. The cells are approximately 250 nm wide. The inset SADP shows that small rotations exist between the cells. The cell elongation in this direction, as opposed to the equiaxed cells seen in Figure 4.38 is likely due to the higher strain of the sample and the direction of the slip traces. Examining Figure 4.39b suggests that the slip traces in this grain primarily ran out of the plane of this sample, and are tightly packed in the horizontal direction. This would form a three-dimensional structure where the dislocation cells are elongated both perpendicularly to the free surface and parallel to the direction of crack growth, but tightly packed perpendicular to the crack growth direction. While the elongation parallel to the crack growth cannot be confirmed with this sample, the other aspects support this conclusion.

The left grain of this sample, imaged in Figure 4.40a and Figure 4.40b, shows less distinct features than Figure 4.40c. The sample has varying contrast indicative of minor variations in the orientation of the grain. These regions show an orientation that is perpendicular with respect to the free surface, just like Figure 4.40. However, the slip bands in this region are not oriented parallel to the direction of crack growth but instead are oriented 60° with respect to it. If the dislocation cells are elongated vertically and in the slip trace direction, the elongation out of the plane is no longer perpendicular to the plane, causing the cell boundaries to appear diffuse and indistinct. Thus, it is likely that both samples have highly elongated cells, but the direction of elongation is different because of the misorientation across the grain boundaries.

### 4.6 The Microstructure of 316 Stainless Steel across Two Loading Modes

![316 Stainless Steel Fracture Surface](image)

Figure 4.41: SEM images from the 316 stainless steel fracture surface: a: An overview of the fracture surface, b-c: SEM micrographs of the areas highlighted in a.
To determine if the microstructure evolution associated with the change in loading mode, cyclic to monotonic, observed in Haynes 230 was unique to the material, a similar test was conducted on a 316 stainless steel. The overview of the stainless steel sample is presented in Figure 4.41a. The sample was subjected to 80,000 cycles, which resulted in crack growth of 3.07 mm with a corresponding stress intensity factor of 73.1 MPa√m. The black arrows indicate the line where the loading mode was changed to monotonic, and the boxed areas are shown enlarged in Figures 4.41b and c. Leading up to this line, the material showed striations and fatigue crack growth, while after this line the material showed microvoid coalescence. Additionally, following the loading mode change, the sample showed considerably more strain as the fast fracture region is roughly 1 mm narrower than the FCG regime. The line indicating the location of the loading mode change is in the center of Figure 4.41b. The marked boxes indicate the locations from which samples were extracted. These areas were chosen as they offered similar flat areas that had experienced each loading type. Figure 4.41c shows another area, with the arrow indicating the location from which the sample was extracted. Both Figures 4.41b and c suggest that the transition in stainless steel is not instantaneous. While the transition from Figure 4.41c seems to occur over 100 µm, the flat areas seen past the transition line in Figure 4.41 suggest that the transition can be spread out over several hundred microns.

Figure 4.42: The microstructure associated with striations and FCG in 316 stainless steel: a: SEM image of the fracture surface where the TEM sample was created: b-c: Bright-field micrographs detailing the dislocation microstructure along the crack growth direction. The crack growth direction is from left to right in all images, and the inset diffraction patterns in b and c show larger amounts of peak splitting near the surface.
Figure 4.42 details the microstructures associated with crack growth due to cyclic loading in 316 stainless steel. The evolved microstructure beneath the fracture surface in 316 stainless has been extensively studied elsewhere [18-20] and so only a brief overview of the dislocation structure is provided here. Figure 4.42a, an SEM micrograph, show the location from which the sample was extracted and that striations are the dominant surface feature. These striations are visible in the bright-field image presented in Figure 4.42b and have an average spacing of 175 nm and an average height of 30 nm. Immediately beneath the fracture surface, a refined layer that extends 70 nm from the crack surface exists. This transitions to a banded structure, with the band spacing being between 100 and 200 nm. These bands could possibly be twins or strain-induced martensite [19, 20]. Since strain-induced martensite is more prevalent at lower stress intensity factors it is likely they are deformation twins. The inset diffraction patterns show that peak splitting occurs both in the refined structure and the banded structure. In the refined structure, the diffraction spots...
are rotated approximately 30° whereas in the bands it is 10°. The same microstructure is seen in Figure 4.42b, though the refinement of the surface now extends 375 nm from the fracture surface. The bands are also less distinct, though this is likely due to a difference in the imaging conditions. The peak splitting at the surface, visible in the inset diffraction pattern is limited to 15°, while it is about 9° in the bulk.

The microstructure associated with the transition between loading modes in 316SS is shown in Figure 4.43. Figure 4.43a, shows the fracture surface at the loading mode change. The contrast change running through the center of the image coincides with the change in loading mode. The transition region here is 30 µm wide. Striations are visible leading up to the loading mode change, while after the change, ripples are present. The montage of bright-field images from the fracture surface is presented as Figure 4.43b. This demonstrates that the fracture surface is relatively flat leading up to the loading mode change, but afterwards it slopes upward at a 45° angle. The total change of height in this sample is 16 µm, but since this sample does not capture all of the transition zone it is likely that the total height change of the transition is over 30 µm. The letters on the montage indicate areas that are more closely examined in Figures 4.43 c-f.

Before the transition, Figure 4.43c, the microstructure has the typical microstructure associated with striations. Striations are visible on the fracture surface and have a spacing of 1 µm with a height of 350 nm. Beneath the striations, the refined structure extends 375 nm from the fracture surface. Beneath this region the sample once again exhibits planar bands spaced 300 nm apart.

The change in loading mode is associated with an intense series of planar bands penetrating through the sample, detailed in Figure 4.43d. These planar bands have a stronger contrast than the bands on either side of them and the spacing is reduced to 150 nm. The inset diffraction patterns suggest that the level of peak splitting at the surface is extremely high, approximately 33°, while within the planar bands below the surface it is 10°.

Following the loading change, ripple features appear on the fracture surface. These have a spacing of 2.5 µm. The microstructure underneath these ripples is detailed in Figure 4.43e. There is a layer of refined sub-grains on the order of 75 nm in diameter which transitions to a number of planar features. Examination of Figure 4.43f, which was taken further along the crack growth direction shows that the only planar features remaining are those that curve to be parallel to the crack growth surface. This suggests that the band structure in Figure 4.43c is formed from the bands produced
by cyclic loading ahead of the crack tip. However, the extensive plasticity due to the loading changes make it difficult to substantiate this claim.

Figure 4.44: The microstructure in the flat region following the loading mode change; a: SEM micrograph of the region from which the TEM sample was extracted; b-d: bright-field TEM micrographs detailing the microstructure along the crack growth direction. The white arrowheads in c and d indicate the dislocation line separating the curved planar band from the straight bands.

Figure 4.44 explores the microstructure formed in the flat region after the loading mode change. No microvoids are seen on the surface of this sample, but irregular ripple-like features are visible in the SEM micrograph presented as Figure 4.44a. The microstructure has a refined zone immediately beneath the fracture surface that extends at most 100 nm, before transitioning to the planar band structure similar to Figure 4.42b. The inset diffraction patterns demonstrate that at the surface the diffraction spots are rotated 18°, but no rotation of the diffraction spots occurs in the bulk. However, in Figure 4.44c, the planar band structure no longer extends to the surface, but is replaced by a curved planar band structure. There is a line composed of dislocation structures that separates the curved section from the planar section similar to that found on the transition microstructure in Haynes 230, see Figure 4.30. The line is jagged with numerous steps indicated by white arrows. Figure 4.44d shows similar features but the curved planar band has decreased in depth from the fracture surface from several microns to under 300 nm. The line of dislocations separates the surface features from the bulk planar features and is indicated by the white arrowheads. The inset diffraction patterns suggest that the curved band area has much higher strain with diffraction spots rotated by 30°, see inset diffraction patterns in both Figure 4.44c and d.

Overall, this surface microstructure is consistent with the results from the transition zone in Haynes 230. However, this sample was created far from the change in loading mode. This suggest dislocation structures such as these are typical of the crack advance under monotonic loading.
Figure 4.45: Assessment of the microstructure beneath a flat area on fracture surface following the change in loading mode in 316SS. *a-b*: SEM micrographs of the fracture surface and the extracted volume; *c-e*: Bright-field electron micrographs detailing the microstructure beneath this fracture surface. The white arrow on *a* indicates the location of the loading mode change, and the black arrow the location from which the TEM sample was extracted. The black arrow on *b* indicates the line separating the bulk microstructure from the surface microstructure. The black arrow on *c* and *d* indicate the same location separating the surface microstructure from the bulk.

To verify the microstructures seen in Figure 4.45, another sample was extracted from a flat region of the fracture surface but one that existed closer to the transition; this sample was extracted at a distance of 60 µm from the transition line compared to 100 µm for the sample described in Figure
4.4. The SEM micrograph of this area, Figure 4.45a, shows similar ripple features as Figure 4.44. The transition is indicated by the white arrow on Figure 4.44 while the black arrow indicates the location from which the TEM sample was extracted. The SEM image presented in Figure 4.45b shows the extracted TEM sample in profile; the ripples on the surface are plainly visible. The bulk of the sample shows contrast variations consistent with crystalline planar defects and it is separated from the surface by a line of planar defects, which are indicated by an arrow. The bright-field TEM image presented as Figure 4.45c, shows that beneath the fracture surface an equiaxed refined sub-grain structure exists. This structure extended for 300 nm before giving way to planar bands. The planar bands near the fracture surface are curved in the direction of crack advance. Figure 4.45d shows that the planar bands in the bulk of the material are not curved, but curvature initiates 3 µm away from the fracture surface. The black arrows mark the same location across Figure 4.45c and d, allowing for the continuation of the bands to be noted. There is a line of defects similar to that separating the curved and parallel regions in other materials; the bands begin to curve before intersecting this line of defects, whereas in Figure 4.44 this line marked the separation of the curved and straight regions. Directly at the surface, the microstructure is refined into sub-grains as seen in Figure 4.44e. The SADP, inset pattern in Figure 4.45c, from the equiaxed sub-grain region shows rotation of the diffraction spots by approximately 18° whereas diffraction spots from a region in the bulk of the sample show little peak splitting. Both of the 316SS samples taken from flat areas past the change in the loading mode show that the planar bands curve strongly in the direction of crack growth near the surface. This suggests that there is extensive plasticity in the crack growth direction near the fracture surface.

4.7 The Microstructure of Nitronic 40 Fatigue Cracks at the Free and Fracture Surfaces

To complement the above work on determining the microstructure surrounding fatigue cracks in planar slip materials, the microstructure produced in Nitronic 40 was determined. The crack was arrested after 80,000 cycles. Figure 4.46 shows the crack tip in the sample. The crack growth in this sample shows a strong hook indicating that the crack growth in not entirely uniform. Following the initial milling around the crack, Figure 4.46b, the crack tip can be seen at the bottom of the sample volume. This indicates that the interior crack tip was not directly aligned with the surface crack tip. This could be due to either the surface crack lagging behind the interior crack or the interior crack having a different direction from the surface crack. When the sample was thinned to just the volume to be extracted, Figure 4.46c, the crack can be seen running across the sample,
while the tip at the bottom of the sample become more complicated. The crack running across the surface of the sample can be seen to branch in three different directions at the bottom of this sample. This branching is even more evident on Figure 4.46d, which is after thinning to electron transparency. Some of the surface was lost during the thinning process, however the crack flank was protected.

![Figure 4.46: The microstructure surrounding a free surface crack tip in Nitronic 40; a: SEM image of the surface crack tip; b: TEM sample following the milling of a trench with the crack below the surface seen extending ahead of it; c: branching crack evident after milling both trenches; and d: the thinned TEM sample showing loss of some of the surface and preservation of the crack.](image)

The extracted volume is again shown in Figure 4.47a with areas from which the electron micrograph was acquired. The subsequent TEM analysis revealed that the dislocation structure consisted of refined sub-grains immediately next to the crack flanks, which were replaced by planar slip bands further away from them. This is especially evident in Figure 4.47b, where the refined sub-grains extend approximately 350 nm on the lower side of the crack flank as indicated
by the white arrows. The sub-grains then give way to planar features elongated parallel to the crack flank. The sub-grains are equiaxed and have a diameter of 200 nm. The upper side of the crack flank, marked by the black arrow, was the region seen between the crack flank and the branching crack path in Figure 4.46c. The inset diffraction pattern from this region suggests about 13° of diffraction peak splitting.

Figure 4.47: The free surface arrested crack tip in Nitronic 40; a: SEM image of the thinned surface crack demonstrating locations of interest for TEM analysis; b-c: TEM micrographs showing the dislocation microstructure surrounding the crack flanks of the main crack; and d: TEM micrograph showing the dislocation microstructure surrounding one of the branching cracks.
The difference between the microstructure next to the crack flank and the bulk microstructure is explored in Figure 4.47c. The refined structure extends approximately 350 nm into the sample and the sub-grains have a diameter close to 100 nm immediately adjacent to the crack flank and 200 nm slightly further away from it. The inset diffraction patterns show that 15° of peak splitting occurs in the refined zone and 9° occurs in the planar banded region. This transformation of the microstructure is also seen in Figure 4.47d, which captures the crack tip of one of the branching crack paths. Sub-grains are visible along the crack flank, with the ones immediately adjacent to the crack flank having a diameter of 100 nm while the next set has a diameter of 200 nm. This sub-grain formation is visible at similar distances immediately ahead of the crack, however the difference in contrast is lower, suggesting that the subgrains are not rotated as severely with respect to each other as the ones along the crack flank. The inset diffraction pattern from immediately in front of the crack tip shows peak splitting of 13°, which is slightly lower than that seen in the crack flank in Figure 4.47c. This suggests that the sub-grain formation occurs ahead of the propagating crack.

The microstructure beneath the striations is explored in Figure 4.48. The SEM micrograph, Figure 4.48a, shows the striations on the fracture sample with the area from which the TEM samples was extracted indicated by the box. Striations are visible in the bright-field TEM micrographs, Figures 4.48b and c, and have a spacing of approximately 500 nm. In the bulk of the sample, banded features oriented away from the fracture surface, and spaced approximately 200 nm apart, are visible. Analysis of the inset diffraction pattern in Figure 4.48b demonstrated these bands are deformation twins, with secondary twins inside the primary twins. Additionally, there is a layer of refined sub-grains extending 375 nm away from the fracture surface; this layer is most evident in Figure 4.48c. There is a distinct contrast change between the bulk planar twin structure and the refined surface structure. The primary deformation twins run through the refined layer to the fracture surface. These twins are bent in the direction of crack advance within the sub-grain layer, indicating they were present prior to crack advance. No specific relationship was seen between the striation marks on the surface and either the refined region or the deformation twins. The inset diffraction pattern from the sub-grain layer shows peak splitting of approximately 15° occurs. Overall the results from Nitronic 40 agree with the results from 316SS and Haynes 230. All the planar slip alloys show that the fracture surface and the crack flanks had a region of refined sub-grains that transitioned into a planar band region. Beneath the fracture surface, the planar bands
tended to be oriented strongly away from the surface, and when interacting with the sub-grain layer, the planar bands would bend strongly in the direction of crack advance.

Figure 4.48: The microstructure beneath the striations parallel to the crack growth direction; a: Fracture surface with the position from which the TEM sample was extracted. b-c: bright-field TEM micrographs detailing the microstructure beneath these areas. Inset diffraction patterns from regions indicated.

### 4.8 References


Chapter 4 presented the experimental results of the evolved microstructural state following fatigue loading for select FCC pure metals, which all exhibited wavy slip, and alloys, which exhibited planar slip. Specifically, the evolved microstructure in the vicinity of an arrested crack was compared to and contrasted with that found beneath striations on the fracture surface. The key findings from this work were:

1. In the planar slip alloys, the evolved microstructural state beneath the free surface increases in complexity as the crack tip is approached. This evolution was not simply a continued increase in complexity of the far-field microstructure, but involved the activation of additional slip systems and the formation of a sub-grain microstructure in the vicinity of the fracture surface. In contrast, in the wavy slip systems the microstructural refinement at either the crack tip or fracture surface was subtler and involved a continued refinement of the existing microstructure.

2. In pure metals, the microstructure beneath striations was composed of dislocation cells that decreased in size and increased in misorientation between neighboring cells as the fracture surface was approached. In the alloys that deformed by planar slip, the evolved microstructural state transitioned from a region of refined sub-grains of a few hundred nanometers in diameter to a microstructure that consisted of deformation bands and in some cases deformation twins and possible strain-induced martensite. This microstructure was consistent with the far-field microstructure although the degree of deformation decreased with distance from the fracture surface.

3. No specific microstructural feature was associated with striation markings in either planar or wavy slip materials.

4. By changing the mode of loading from cyclic to uniaxial tension, it was shown that the failure path transitioned over some distance that corresponded to additional modes of deformation being activated and dominating the evolved microstructural state. This result gives insight into the interplay between striations and microvoid failure.
5. There is a lack of correlation between the microstructure predicted by strain mapping techniques and the actual crack tip microstructure. Although this was not an extensive component of the thesis, the comparison does provide insight into our ability to interpret microscale results in terms of specific deformation processes.

This chapter is organized as follows:

1. The ability to interpret surface strains as assessed by using EBSD and / or DIC in terms of specific evolved dislocation structures is discussed. It will be shown that in very general terms this is possible, but in terms of specific and local deformation processes the correlation is weak at best as we have insufficient knowledge of dislocation processes in regions of high strains.

2. A discussion of the evolution of the deformation microstructure generated either by crack closure events or by the actual propagation of the crack through an early generated deformation microstructure. In other words, the evolved microstructural state is assessed in terms of contributions from the cyclic loading, the plastic zone of the crack tip, crack closure and crack propagation. It will be demonstrated that the evolved microstructural state observed in this work is a consequence of all possible contributors except crack closure plasticity.

5.1 Surface Strain Mapping and the Underlying Dislocation Structure in Haynes 230

Mapping of the surface strains by using either EDSD or DIC measurements has been used to follow the evolution of strain during cyclic loading as a means to assess the degree of plasticity [1, 2]. These results are often interpreted in terms of the deformation processes and interactions that are believed to have occurred in the material. However, the evolved microstructural state has not been directly correlated with either EBSD or DIC strain maps.

Although EBSD and DIC analysis were not used extensively in this work, they were employed to map the deformation field generated ahead of an arrested fatigue crack in Haynes 230, see Figure 4.3. The two methods yield results that are in qualitative agreement. This result agrees with the work by Carroll et al. [3] that demonstrated that the strain evolved with the number of loading cycle at some distance ahead of the crack tip, and is concentrated, although not necessarily equally, into two lobes oriented approximately 45° from the crack propagation direction. Although both
the DIC and EBSD maps presented in this work share the same general lobe formation, a more detailed examination reveals differences, especially in the magnitude and homogeneity of the strain within the lobes. In general, the DIC map shows more homogeneous strain distributions than the EBSD map. The difference in the degree of homogeneity can be attributed to how measurements determine the strain. For example, EBSD analysis compares the average misorientation, resolution limited to 0.75° [4], between the step size points, in this case 1.5 µm, and its nearest neighbors. It is important to note that the step size is not equivalent to the probe size, typically 10-20 nm, which determines the area from the data is collected. This measurement reflects the amount of geometrically necessary dislocations responsible for the rotation of the crystal and not statistically stored dislocations [5]. In contrast, the DIC map examines how the surface deforms and captures the contributions from all the deformation mechanisms, including grain boundary motion and deformation twinning [6]. This also means that the strain measured by DIC cannot be related directly to specific microstructural features. The DIC spatial resolution used in this work was on the order of 5-10 µm, although higher resolution (0.15 µm) has been demonstrated in other works [7].

From the analysis conducted in this study, it is possible to compare the data in the EBSD map and the underlying microstructure since TEM samples were extracted from the surface from which the EBSD map was generated; no direct correlation is possible with the DIC data as it was generated from the opposite surface. Since the dislocation structure consisted of planar slip bands throughout the plastic zone, the relative amount of plastic of strain must be evaluated by examining the spacing of the planar slip bands and the dislocation spacing within them. Characterizing the latter proved difficult as many planar slip bands consisted of long tightly packed dislocations preventing an accurate measurement of the dislocation spacing.

In general, the EBSD map showed that at locations equidistant from the crack tip plastic strain was highest within the deformation lobe, and that strain decreased with increasing distance from the crack tip. The evolved microstructure was characterized at distances of 800 µm, 400 µm and 80 µm from the crack tip, see Figures 4.8–4.16. In contrast, microstructural assessment by TEM did not find significant differences in the microstructure from inside and outside of the deformation lobes at distances of 800 µm and 400 µm from the crack tip. Only at the distance of 80 µm from
the crack tip was there a noticeable difference in the deformation structure inside and outside the band, with the planar band spacing inside the lobe reduced by 46% compared to outside the lobe.

There are two possible explanations why the difference in strain between the deformation lobe and the bulk of the material was only noticed from the samples taken closest to the crack tip. The first is a remark on the intensity of the deformation. As the crack tip is approached, plastic strain within the deformation lobe rises, while plastic strain outside the lobe stays relatively constant. Thus, the pair of samples nearer the crack tip will show a more intense difference in plastic strain, observable which is under TEM examination. The second possible explanation involves the degree of strain localization. The EBSD map suggests that concentrations of plastic strain are highly localized even within the deformation lobe. Since the sampled area for each EBSD point and the step size are different by two orders of magnitude, the possibility exists that the strain is even more highly localized than suggested by the map. Such a localization could mean that the TEM samples extracted from the deformation lobe simply did not contain the highly strained regions. This second explanation is unlikely since intense sub-micron strain inhomogeneities would not be energetically favorable.

Part of the goal of the EBSD work was to connect changes in the measured surface strain with changes in the microstructure. These microstructural changes manifested as modifications to the planar slip structure, except at the crack tip, where the microstructure underwent a transformational change. This change was not reflected on the EBSD scan of the surface for two likely reasons. The first was that the size of the transformation zone was much lower than the step size of the EBSD scan. However, even if a smaller step size was utilized, this region may not be detectable via EBSD techniques due to the significant out-of-plane strain at the crack tip. This strain creates tilts in the sample surface away from the flat plane necessary for accurate indexing of the EBSD pattern. The tilt intensifies as the crack tip is approached, making it more difficult to index the areas next to the crack flank.

When comparing the EBSD maps and the microstructure, no direct correlation could be made other than increased deformation was associated with higher strains. While no comparison between the DIC map and the microstructure was performed, it would be reasonable to assume that the results would be similar and no direct correlation between the measured strain and the microstructural arrangements would exist. In addition to examining the general correlation between the measured
strain and the microstructure, it is important to attempt to correlate the microstructure and strain surrounding specific microstructural features, namely slip traces, grain boundaries, and carbides.

At low strain levels, slip traces appeared on the EBSD map as long lines with higher misorientation. The density of slip traces found by EBSD was lower than that imaged on the subsequent SEM images, likely due to EBSD only detecting misorientations due to large concentrations of dislocations. Electron micrographs capturing the free surface including slip traces showed that the traces were the result of multiple parallel planar slip bands impacting the free surface. At higher strains, individual slip traces were no longer visible on the EBSD map. This likely arises from the increased density of dislocations preventing the contributions to the misorientation from single slip traces from standing out.

![Figure 5.1: Comparison between the strain map and the dislocation structure at twin boundary; a: EBSD KAM map showing the strain at the twin boundary; and b: the dislocation structure at the twin boundary. The arrow indicates the approximate location from which the sample was obtained.](image)

The EBSD map also demonstrated that strain was accumulating at grain boundaries and that this accumulation extended 4-5 pixels from the grain boundary, which corresponds to a distance of 7.5 µm. However, no changes to the dislocation structure around grain boundaries, which would be consistent with strain extending this distance, were observed in the TEM analysis. Figure 5.1 compares an EBSD strain map and the dislocation structure observed at a twin boundary. The TEM images was shown in greater detail in Figure 4.12. Areas of higher strain, visualized as the orange and yellow, are plainly visible along the grain boundary and these extend 2-3 µm from the boundary, Figure 5.1a. These strain fields are discontinuous along the boundary indicating that some locations are subjected to higher strains than others. The black arrow indicates the approximate location at which the TEM sample was extracted. The electron micrograph of a region of the same boundary, Figure 5.1b, shows that the planar slip bands do not change near the grain.
boundary. The dislocations associated with the grain boundary are limited to within 40 nm of the grain boundary. Planar slip bands that impact the grain boundary show no increase in the dislocation density compared to other bands examined. Overall the TEM analysis shows little to no evidence of higher dislocation activity associated with the twin boundary on the length scale found by the EBSD analysis.

There are several possibilities for why the grain boundary strain concentration found on the EBSD strain map was not seen in the TEM analysis. First, there is the issue of the resolution of the different techniques. Second, the discontinuous nature of the strain along the grain boundary may make it difficult to capture the variation in any individual TEM sample. Third, the strain could be formed as an increase in the density of dislocations within the planar slip bands, and not an increase in the density of the planar slip bands themselves; such an increase would be difficult to image. This explanation would rely on the planar slip bands forming pile-up structures near the grain boundary, which was not noted in the planar slip bands that interacted with the twin boundary. In general, the extensive strain field around the grain boundary suggested by EBSD is not supported by the TEM observations.

Figure 5.2: Comparison between strain map and dislocation microstructure at a carbide; a: EBSD KAM map showing the strain associated with a carbide; and b: the microstructure between a buried carbide and free surface. The black arrow in a points the the strain concentration associated with the carbide.

The other microstructure features examined for correlations in Haynes 230 were carbides. M₆C carbides are found throughout the sample and are visible in both surface SEM images and on the fracture surface. A comparison between the EBSD map and the TEM micrograph is made in Figure 5.2. In Figure 5.2b, the carbide was shown to drastically affect the dislocation structure. Local to the carbide was a tangle of dislocation half loops coming off the carbide. The base planar slip structure did not directly interact with the carbide, instead it cross-slipped away from the carbide.
at an approach distance of 500 nm. The EBSD strain map from this area, Figure 5.2a, shows an increase in strain at the approximate location of the carbide, indicated by the arrow. As the carbide is under the surface all of the sampled points were indexed and the carbide itself was not directly observed on the EBSD map. The strain field of the carbide creates the extensive cross-slip seen in Figure 5.2b. This demonstrates that EBSD can detect the microstructural change created by the stress field of the carbide and a direct correlation can be drawn. Other areas which feature seemingly random increases of strain away from grain boundaries may be due to the actions of carbides.

From the above, it can be concluded that a direct correlation of the strains computed by EBSD (and by corollary DIC) with specific deformation states cannot be made. This makes it difficult to interpret EBSD and DIC data in terms of specific deformation mechanisms as the underlying mechanisms are more complex than generally assumed. In other words, there is a need to understand the evolution of dislocations structures with strain, the self-organization of dislocations into low energy configurations, and how these configurations evolve as a function of strain before surface strain maps can be interpreted in terms of specific dislocation structures.

### 5.2 The Microstructure around Crack Tips in Planar Materials

One of the challenges of determining the microstructure generated by the propagation of a fatigue crack is separating the contributions of the evolved microstructural state from the cyclic loading, the plastic zone of the crack tip, and the deformation processes involved in crack advance. To isolate the individual contributions, the microstructure of an arrested fatigue crack was examined as function of distance from the crack tip on the free surface, as well as beneath striations and voids on fracture surfaces. This result also distinguishes whether the crack tip microstructure was formed by the large plastic strains involved in the process of crack advance [8, 9] or from the repeated instances of crack closure behind the crack tip [10].

From the examination of the microstructure beneath the free surface, Figures 4.6-4.16, ahead the crack in the Haynes 230 alloy it is known that the far-field microstructure consists of dislocation slip organized into planar bands. This can be attributed to the actions of the cyclic loading. The effect of the plastic zone is to increase the density of slip traces, activate multiple additional slip systems, and increase the density of the planar slip bands. Dense planar slip bands were found beneath the striation markings and near arrested crack tips on both the free surface. This suggests
that the planar bands structure exists ahead of the crack under plane stress and plane strain conditions.

Additional support for the existence of this banded structure ahead of the propagating crack is found in the microstructure reported in 316 austenitic stainless steel and in Nitronic 40. In 316 stainless steel, the microstructure consisted of dislocation cells with twins superimposed. In Nitronic 40 the microstructure consisted of deformation twins superimposed on a high density of planar slip bands. The twins in the stainless steel showed similar orientation to the slip bands in Haynes 230, suggesting a common origin. The banded structure was found near the arrested crack tip from the free surface in Nitronic 40, and ahead of the interior arrested crack in 316 stainless steel. The similarity demonstrates that this microstructure is commonly formed by different planar slip metals and is unaffected by 3-dimensional strain state. The planar slip band structure presents a microstructural barrier to crack tip dislocation emission.

Based on the evidence presented in Chapter 4, the refined microstructure surrounding crack tips results from a combination of cyclic loading, the plastic zone produced ahead of the crack tip, and crack-tip blunting. Several observations lead to this conclusion:

1. The refined microstructure existed on both sides of the crack flanks of the arrested crack in Haynes 230. Additionally, there was a smooth transition between the refined microstructure found at the surface and the planar band structure, suggesting progressively higher strain toward the crack tip.

2. The refined microstructure was found beneath features on the fracture surface, with the rotation of the diffraction spots suggesting that the refinement was due to an intense strain in one direction.

3. The refined microstructure was observed at the arrested crack tip away from the free surface until the point where the loading mode was switched from cyclic to monotonic. Upon the change in loading mode, the refined microstructure was replaced by dislocation cells, while the planar band structure continued.

4. The refined microstructure was present across the different planar slip materials investigated.

The refined structure observed can be considered analogous the fatigue process zone, which is an area within the crack plastic zone directly affected by the dislocation emission associated with
crack advance. Estimates of the size of the process zone have been varied. Works have sited the size of the process zone to be on the order of several hundred nanometers [11], tens of micrometers [12], or hundreds of micrometers [13] for steels and aluminum alloys. This study found that the refined structure, ergo the process zone, was limited to between 300-500 nm across a range of planar slip alloys and stress intensity factors.

Based on the microstructure of the sample, the refined microstructure forms from intense shear strain in one direction near the crack tip. There are three main factors indicating that the microstructure was created by shear strain and suggesting the directionality of the strain:

1. The planar slip bands that penetrate into the refine structure are curved in the direction of crack propagation.
2. The selected area diffraction patterns indicate that the refined microstructure causes a splitting and rotation of the diffraction spots in one direction only. Such rotation indicates that the refined structure has been developed from the planar slip structure by the action directional strain field.
3. The rotation and refinement at the surface was not visible when the fracture surface was examined parallel to the striations markings.

Taken together, these three points would suggest that the refined fracture surface is rotated strongly in the x- and y-directions while having little rotation in the z- direction. The two leading mechanisms for crack advance, the Laird and Smith [14] blunting model and the Neumann [15] alternating shear model require dislocation emission along the planes of maximum shear. The refinement and the attendant rotation observed are indicative of very high strains occurring at the fracture surface, agreeing with the established mechanisms. It is therefore likely the refined structure is formed ahead of the crack by the large shear strains involved in crack advance processes, while the planar slip structure is formed due to the actions of the crack tip plastic zone surrounding the crack. The rotation of the surface occurs as the planar slip structure is restrained to cross slip in order to accommodate the shear strain associated with crack advance.

The refinement is a response to this shear strain. Due to the influx of a large number of dislocations emitted by the blunting crack tip, spaces between adjacent planar slip bands begin to rotate to accommodate additional dislocations. The planar slip bands act as barriers to dislocation motion, confining the emitted dislocations near the crack tip. As more dislocations are emitted and
incorporated into subgrain boundaries the size of the subgrains decreases, while the rotations between the subgrains increase. Near the crack, where the dislocation density is highest, the subgrains are smaller than the preexisting planar band spacing, but further away, evidence of the transition between the two structures can be seen. This is most evident on Figure 4.19, where the microstructure next to the crack consists of small equiaxed subgrains which smoothly transition to a structure where planar bands are still evident, but the spaces between the planar slip bands have small rotations and misorientations.

It is unlikely that the structure was formed due to the actions of crack closure and rubbing of the fracture as previous literature had suggested [8]. First, no indications of rubbing deformation, such as tire track markings, were observed on the fracture surface. Second, the microstructural indications suggest that the refined structure was formed by the actions of an intense shear strain operating in one direction, while crack closure and rubbing would apply a compressive stress to the fracture surface. Third, the refined microstructure was consistently found near arrested cracks on both the fatigue and fracture surface; the refined microstructure in these locations was comparable to the microstructure found beneath striations. The arrested cracks would not have been subjected to repeated instances of crack closure, so the crack tip microstructure should be preserved.

While the strain within the refined zone cannot be quantified from the data here, the dislocation structure can be compared to deformation structures generated through other means. Similar refinement has been generated through high pressure torsion experiments in nickel alloys. In a nickel base Ni-Cr-Al alloy it took a strain of 7 to reduce the grain size from 60 µm to 34 nm [16]. In a Ni-20%Cr alloy it was found that a strain of 2.5 was necessary for the production of nanoscale grains from large grains [17]. While not directly comparable to the material studied herein, these results do suggest that very high strains are necessary to produce the crack tip microstructure.

This microstructural evolution, i.e. the planar bands into refined sub-grains, is much more complex than would be predicted by models of crack growth described in Section 2.17.2. All of the physically-based models use an accumulation of Burgers vectors at the crack tip due to the emission of dislocations. A recent review by Chowdhury and Sehitoglu [18] highlights many of these models. These models all utilized simple interactions to model the resistance to dislocation emission, or the irreversible slip. Of particular note were the models by Deshpande et al. [19, 20],
who utilized dislocation dynamics to study slip band evolution. The spacing between slip bands found in this study are comparable to Deshpande et al.’s study, however the slip band evolution in their study was due to the actions of dislocations emitted from the fatigue crack and not the preexisting microstructure as was found here.

Other models attempt to relate the amount of irreversible slip to crack growth. These models currently simulate a single slip system interacting with a grain boundary [21-23] or two parallel slip systems interacting with each other [24, 25] or a dislocation-free zone caused by dislocation with opposite Burgers vectors slipping on the same plane at different points in the loading cycle [26, 27]. Such models do not account for the microstructural complexity noted in this study. Each of these models relies on reverse slip toward the crack tip, while this study demonstrates that dislocations emitted from the crack tip will be accommodated within the preexisting microstructure and will not be capable of reverse slip back to the crack tip. In particular, this study refutes the concept of a dislocation free zone ahead of a crack tip.

Furthermore, none of the models deal with the interaction of emitted dislocations with the preexisting microstructure. This study suggests that both the planar slip structure, and the dislocation cell structure act as strong barriers to slip. Modeling of such interactions could be a promising potential route for a physically-based crack growth model to accurately predict both the resistance to dislocation emission and the mechanism of slip irreversibility, as well as more accurately simulate the conditions of growth fatigue cracks.

### 5.3 The Microstructure around Crack Tips of Wavy Slip Materials

In general, the dislocation structure formed in wavy slip materials was very different from that produced in planar slip materials. The most important finding was that the refined layer does not occur immediately beneath the fracture surfaces. The findings on the type of dislocation structure found for wavy slip materials are in agreement with those reported in the literature [8, 28-30].

Dislocation cells were found around fatigue cracks for both copper and nickel. The dislocation cell structure was found ahead of the crack, surrounding the crack flanks and beneath the fracture surface. In general, the dislocation cells are not oriented in any particular direction. However, at a
high stress intensity factor, the nickel crack had slip bands in the same direction as slip traces on the free surface, Figure 4.40.

There was evidence that the strain near the crack tip was higher than strain in the bulk of the sample in copper. As the crack tip was approached, the dislocation cell size decreased and deformation twins were formed in favorably oriented grains; both observations are indicative of higher strains near the surface. Furthermore, the cells immediately adjacent to the fracture surface have a strong difference in orientation, which is a strong indication of higher strains, and the degree of misorientation between the cells diminishes with increasing distance the fracture surface.

Taken together, this evidence suggests that there is an increase of strain in the immediate vicinity of the crack tip, similar to that found in the planar slip alloys. However, the refinement at the surface was not seen in the wavy slip alloys due to the difference in dislocation structures; Copper and nickel are able to contain the shear strain associated with crack advance within the existing dislocation structure by rotating the preexisting dislocation cells to decreasing the cell size and increasing the misorientation between adjacent cells.

The lack of transformation microstructure also prevents an overt measurement of the process zone associated with fatigue crack growth. Instead the process zone must be estimated by examining how quickly the high cell misorientation reverts to the bulk cell misorientation, and observing the depth at which the deformation twins propagate. These observations suggest that the process zone associated with crack advance in copper is comparable to the process zone found on the planar slip materials, i.e. between 300 and 500 nm from the fracture surface.

5.4 The Microstructure Associated with Striations and Crack Advance

In general, this microstructural study was unable to determine the association of striation marks, and hence crack advance, with specific microstructural feature. Since the crack advance was not monitored in situ for most of the samples, determining whether the crack grew intermittently or cycle by cycle was not possible.

In planar slip alloys, it has been suggested [20, 31, 32] that each band in the planar banded structure was associated with the formation of a single striation. Such a correlation was not found in this study. In Haynes 230, two to three intense planar bands were observed beneath each striation, see for example Figures 4.22 and 4.23. A similar result was found in 316 stainless steel and Nitronic
40. Given the current hypothesis that the planar banded structure was formed due to the plastic zone of the crack tip, it is unreasonable to expect each band to be associated with one striation.

In addition to the planar slip bands, the refined structure did not show any association with striations in any of the alloys tested. Despite the height change connected with the striations, the refined structures stayed at a relatively constant penetration into the material. Changes to penetration were not noted to correspond to striations. Sub-grains did not correspond to individual parts of striations either.

In the wavy slip materials tested, copper and nickel, no microstructural features corresponding to individual striations was found. In copper, the dislocation cells were smaller than the striation marks and cell boundaries did not correspond to striation features. This is in agreement with the existing literature [28, 33-35]. When deformation twins were present, between one and five twins existed for every striation mark. When striations and surface slip traces were present in nickel, a close examination of the crack tip revealed that multiple slip traces existed for each striation mark.

This evidence does not confirm nor deny the tradition model of one fatigue striation being created by a single crack advance in response to a loading cycle, although such a link is well established at the relatively high stress intensity factors used in this study [14, 36, 37]. What has not been observed is any correlation between the underlying microstructure and the striation markings. This would suggest that the striation markings are not correlated with individual loading cycles. This is consistent with studies at lower stress intensity factors where multiple loading cycles were necessary to create single crack advance event [38, 39].

Thus, in both the wavy and planar slip materials three was no correspondence between the microstructural features and the striation markings on the surface, nor is there any microstructural features that would correspond to a single crack advance event. This suggests that the surface striation features are relatively independent of the microstructure features.

**5.5 The Influence of Loading Mode on Microstructural Evolution and Fracture Pathway**

Having addressed how the microstructure under striations evolves, it is now important to examine how changing the loading mode impacts the microstructure development and the transition from striation formation to microvoids. Following the change in loading mode, the microstructure at the fracture surface immediately changes to a series of tightly packed dislocation cells. The far-field
microstructure remains broadly similar across this change. The fracture surface microstructure becomes separated from the far-field banded structure by a band of dislocations. The far-field banded structure only changes when it enters the region near the microvoid wall.

The formation of the ripple features is likely due to the actions of the crack tip. Following the cyclic loading the arrested crack tip closes due to the reverse plastic zone leaving a sharp crack tip in the material. This crack has a critical stress intensity factor necessary to once again advance. Upon loading, the stress intensity factor gradually rises. When the stress intensity factor reaches a critical value, the crack propagates. During propagation, the crack tip will begin to plastically blunt, lowering the effective driving force for crack growth. If the crack blunts enough, the crack may temporarily stop until the driving force becomes high enough again. When the stress intensity factor reaches the fracture toughness, the primary M6C carbides within Haynes 230 will begin nucleating microvoids, which is where the sample leaves the transition region and enters the microvoid coalescence region. The ripple markings are points where temporary pauses occurred in between rounds of the crack growth via plastically blunting.

The transition from the far-field banded microstructure to the MVC microstructure is accompanied by a large increase in plasticity. The microstructure changes from the neatly organized planar slip bands to a less organized region showing local variations in contrast. This section is likely caused by the change in the stress state as the microvoid region is approached. While a sharp crack would likely have a similar plastic field as the fatigue crack, a two-dimensional plane strain state, the microvoid wall will have a spherical three-dimensional strain state. This spherical strain state would be in the direction of the microvoid wall, which is oriented 45° from the direction this sample is located. Since the deformation is now oriented partially out of the plane of the sample, the boundaries are not distinct, creating the microstructure associated with the microvoid wall.

The near surface microstructure in the transition region consists primarily of dislocation cells, as imaged in Figure 4.33c. These cells appear to have divided based on the prior locations of planar slip bands. In addition, planar slip bands leading up to the cell structure are bent forward, with dislocation cells being bent more. It is likely that this structure is formed as the material at the crack tip plastically stretches in the direction of crack advance. This creates a substantial amount of strain in the forward direction, bending the slip bands and forming the dislocation cells. Since this microstructure is not produced during fatigue loading, the substantial shearing strains are
replaced by a higher normal strain in the direction of crack advance. This normal strain extends further away from the fracture surface than the localized shearing of the crack tip in fatigue, forming the microstructure.

5.6 References


CHAPTER 6
SUMMARY AND CONCLUSIONS

The microstructure evolution surrounding fatigue crack tips for a variety of FCC metals and alloys was examined in detail using strain mapping techniques, SEM imaging, and TEM examination. The main findings of the work were:

1. There are distinct differences in the microstructural evolution between wavy slip pure metals and planar slip alloys. In wavy slip alloys the microstructure consists of dislocations cells, while in planar slip alloys the microstructure consisted of bands which were refined into sub-grains near the crack flank.

2. In planar slip alloys the contributions of each plastic process were identified. The cyclic loading resulted in planar slip bands. The plastic zone of the crack acted to increase the density of the planar slip bands. The crack tip deformation further refined the planar slip structure into a sub-grain structure.

3. The microstructural complexity found near fatigue cracks is much higher than that predicted by current physically-based models of crack growth. The current models cannot accurately predict this microstructural evolution because they do not consider interactions with the dislocation emitted by the crack with pre-existing dislocation structures. A possible future model, one that uses accurate knowledge of the pre-existing microstructure, was identified.

4. Highly strained regions were found near the crack in both planar and wavy slip materials. In planar slip alloys it was estimated to be the size of the refined zone between 300-500 nm. In the wavy slip metals, it was estimated from the extent of highly misoriented dislocation cells. The highly misoriented cells only extended 300-500 nm from the crack tip. This size was considered to be the process zone size.

5. No microstructural feature was found that corresponded to striations marks. In the planar slip alloy, neither the bulk banded structure nor the local refined structure had any correlation to the striation markings. The dislocation cells and deformation twins found in the wavy slip alloys did not correspond to the striation markings either. This suggests that there is no inherent microstructural process that ties loading cycles to striations markings.
and, hence, crack advance. This also suggests that models that attempt to link striation formation to the presence of slip bands impacting the fracture surface are incorrect.

6. The correlations between strain mapping techniques and the microstructure within a planar slip alloy were investigated. It was found that increases in strain associated with the plastic lobes and grain boundaries did not have a corresponding increase in the strain visible in the microstructure. However, second phase particles did show a strong correlation, having a localized increase in strain directly associated with a change in the dislocation structure.