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The Role of Microstructure on Deformation and Damage Mechanisms in a Ni-based Superalloy at Elevated Temperatures

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THE ROLE OF MICROSTRUCTURE ON DEFORMATION AND DAMAGE
MECHANISMS IN A NI-BASED SUPERALLOY AT ELEVATED
TEMPERATURES

BY

KIMBERLY E. MACIEJEWSKI

A DISSERTATION SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF
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DOCTOR OF PHILOSOPHY DISSERTATION
OF
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ABSTRACT

The overall objective of this research work is the development and implementation of a mechanistic based time-dependent crack growth model which considers the role of creep, fatigue and environment interactions on both the bulk and the grain boundary phase in ME3 disk material. The model is established by considering a moving crack tip along a grain boundary path in which damage events are described in terms of the grain boundary deformation and related accommodation processes. Modeling of these events was achieved by adapting a cohesive zone approach (an interface with internal singular surfaces) in which the grain boundary dislocation network is smeared into a Newtonian fluid element. The deformation behavior of this element is controlled by the continuum in both far field (internal state variable model) and near field (crystal plasticity model) and the intrinsic grain boundary viscosity which is characterized by microstructural parameters, including grain boundary precipitates and morphology, and is able to define the mobility of the element by scaling the motion of dislocations into a mesoscopic scale. Within the cohesive zone element, the motion of gliding dislocations in the tangential direction relates to the observed grain boundary sliding displacement, the rate of which is limited by the climb of dislocations over grain boundary obstacles. Effects of microstructural variation and orientation of the surrounding continuum are embedded in the tangential stress developing in the grain boundary. The mobility of the element in the tangential direction (i.e. by grain boundary sliding) characterizes the accumulation of irreversible displacement while the vertical movement (migration), although present, is assumed to alter stress by relaxation and, thus, is not considered a

contributing factor in the damage process. This process is controlled by the rate at which the time-dependent sliding reaches a critical displacement and as such, a damage criterion is introduced by considering the mobility limit in the tangential direction leading to strain incompatibility and failure. This limit is diminished by environmental effects which are introduced as a dynamic embrittlement process that hinders grain boundary mobility due to oxygen diffusion. The concepts described herein indicate that implementation of the cohesive zone model requires the knowledge of the grain boundary external and internal deformation fields. The external field is generated by developing and coupling two continuum constitutive models including (i) a microstructure-explicit coarse scale crystal plasticity model with strength provided by tertiary and secondary γ' precipitates. This scale is appropriate for the representation of the continuum region at the immediate crack tip, and (ii) a macroscopic internal state variable model for the purpose of modeling the response of the far field region located several grains away from the crack path. The hardening contributions of the γ' precipitates consider dislocation/precipitate interactions in terms of γ' particles shearing and/or Orowan by-passing mechanisms. The material parameters for these models are obtained from results of low cycle fatigue tests which were performed at three temperatures; 650, 704 and 760°C. Furthermore, a series of microstructure controlled experiments were carried out in order to develop and validate the microstructure dependency feature of the continuum constitutive models. The second requirement in the implementation of the cohesive zone model is a grain boundary deformation model which has been developed, as described above, on the basis of viscous flow rules of the boundary material. This

model is supported by dwell crack growth experiments carried out at the three temperatures mentioned above, in both air and vacuum environments. Results of these tests have identified the frequency range in which the grain boundary cohesive zone model is applicable and also provided data to calculate the grain boundary activation energy as well as identifying the relative contributions of creep and environment in the critical sliding displacement leading to failure. Validation of the cohesive zone model has been carried out by comparing the simulated crack growth data with that obtained experimentally. This comparison is used to optimize the different model components and to provide a route to assess the relative significance of each of these components in relation to the intergranular damage associated with dwell fatigue crack growth in the ME3 alloy. For this purpose, a set of case studies were performed in order to illustrate the sensitivity of the cohesive zone model to variations in microstructure parameters (γ' statistics and grain boundary morphology) examined within the range of temperatures utilized in this study.

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PREFACE

This dissertation is prepared using manuscript format.

Chapter 1 gives an overview of the research work. It describes the jet engine in terms of its service application (temperature and loading), material requirements and limiting life and safety factors. This justifies the use of the Nickel-based superalloy for the current research. In addition, it discusses the importance of microstructure on the time-dependent cracking process. Finally, this chapter addresses the various scales of modeling including phase field, fine and coarse crystal plasticity and macroscopic internal state variable type modeling. It also discusses the modeling requirements, advantages and disadvantages of the various approaches and the reasoning behind the model scales used in this work.

Chapter 2 describes the Nickel-based superalloy material including processing techniques, chemistry, microstructure and heat treatments. The processing routes, as well as, the advantages and disadvantages of the ingot and powder metallurgy processes are discussed. The roles of the various alloying elements are addressed. The microstructure of the Nickel-based superalloy is described including phases and grain boundary morphology. In addition, the heat treatment process and precipitate kinetics including nucleation, growth and coarsening are discussed. This chapter will also describe the characteristics of the two main alloys for the current work, IN100 and ME3.

Chapter 3 describes the dwell fatigue crack growth experiments, including test setup, crack growth rate and its correlation with loading frequency. In addition, the loading frequency is correlated with the slip band spacing and fracture mode. The

crack length, crack opening displacement and crack growth rate are examined in this chapter as a function of temperature and environment. Furthermore, the active operating mechanisms are identified through analysis of the apparent activation energy and experimental observations.

Chapter 4, which describes the dwell crack growth modeling approach. The overall simulation strategy is addressed, outlining the required model components including the multi-scale continuum model and grain boundary interface model. In this, the grain boundary deformation and damage mechanisms, including grain boundary sliding and dynamic embrittlement, are incorporated into a grain boundary cohesive zone interface model. In addition, the general grain boundary deformation and damage mechanisms including cavitations, grain boundary sliding, two stage oxidation and dynamic embrittlement are discussed.

Chapter 5 describes the continuum material deformation models which are necessary to describe the stress field surrounding the crack tip in the cohesive zone model. In this, the theory of the internal state variable (ISV) and coarse crystal plasticity (XP) models are described. In addition, the low cycle fatigue experiment setup and test results are discussed. The results are also interpreted in terms of the influence of temperature and loading conditions. Determination of the required model parameters is carried out and uniaxial one and two-dimensional simulations are performed under various strain controlled loading scenarios.

Chapter 6 then combines the continuum and grain boundary modeling efforts to simulate the dwell crack growth rate. The finite element model is described and various case studies varying temperature and environment are performed and

compared to the experimentally obtained crack growth rate. In addition, a sensitivity analysis is performed varying the oxygen partial pressure, grain boundary diffusivity and grain boundary modulus. The influence of these parameters is examined in terms of grain boundary sliding and crack growth rate.

The influence of microstructure on intergranular cracking is then addressed in Chapter 7. In this the γ' statistics are examined by explicitly modeling their size and volume fraction in the ISV and XP material model formulations. The hardening contributions of the γ' precipitates consider dislocation/precipitate interactions which have been identified in terms of particle shearing and/or Orowan by-passing mechanisms with respect to a critical particle size. The influence of γ' on continuum deformation, grain boundary sliding and crack growth rate are examined both experimentally and numerically. Furthermore, the influence of grain boundary serrations on the grain boundary viscosity, sliding and crack growth rate is examined. A portion of Section 7.2.2 will follow formatting guidelines for the *International Journal of Fatigue*. A portion of Section 7.2.3 will follow formatting guidelines for *Materials Science and Engineering A*.

This is followed by Chapter 8, outlining the main conclusions of this work and Chapter 9 detailing future recommendations.

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CHAPTER 1

INTRODUCTION

1.1 Statement of Problem

Intergranular crack growth in Nickel-based superalloys, utilized in the turbine components of aero-engines, is significantly influenced by localized factors that include aggressive air elements and temperature which could alter the flow behavior and enhance damage effects taking place in the crack tip region. Other factors influencing the long crack growth mechanisms are related to the time-dependent deformation response of the near- and far-field continuum surrounding the crack tip. The work in this thesis deals with intergranular cracking mechanisms by focusing on the synergistic effects of both localized and global factors which control the kinetics of the crack growth process along the grain boundary fracture path. These factors will be examined in terms of microstructure details including dislocation/precipitate interactions as they are influenced by thermal and mechanical loading of the crack tip region. The major goals of this research are:

- i) Development of a mechanistic based intergranular crack growth model using a cohesive zone approach. The simulation work in this approach requires the knowledge of the deformation and fracture formulations of three basic components-which are: (1) grain boundary interface representing the cracking path, (2) near-field region (an assembly of randomly oriented grains) in direct

contact with the crack path which imparts displacement and traction fields affecting the grain boundary deformation response, and (3) far-field continuum which transfers the external mechanical loading to the former two regions. In order to define the grain boundary interface laws, the active grain boundary cracking mechanisms in an air and vacuum environment are determined by performing a series of crack growth tests on the as received microstructure. These mechanisms, identified as grain boundary sliding and dynamic embrittlement by oxygen diffusion, are incorporated into the traction displacement laws governing the deformation and damage of the grain boundary interface. The near- and far-field continuum, which control the deformation behavior of the grain boundary interface, are described by a multi-scale model consisting of a coarse crystal plasticity zone representing the near-field region and a macroscopic internal state variable type model which describes the far-field material response. In order to define the necessary material parameters required for the crystal plasticity and internal state variable models, a series of strain controlled low cycle fatigue tests are performed and analyzed. Following this, the continuum and grain boundary interface models are combined into a cohesive zone model and the intergranular cracking process is validated experimentally for different temperatures and environments.

- ii) Examine the role of microstructure on the intergranular cracking process by experimentally altering the γ' precipitate statistics of the as received Nickel-base superalloy material through variations in the time, temperature and

cooling rate in the typical three stage heat treatment sequence (solutioning, stabilization and aging). The influence of microstructure on variations of internal stresses on monotonic, relaxation, and cyclic loading responses are determined by performing strain controlled uniaxial tests on both the as received and the modified microstructures. In addition, the role of microstructure on intergranular crack growth rate is examined experimentally by performing crack growth tests in both air and vacuum environments using specimens made of the modified microstructure materials. Furthermore, the role of microstructure on intergranular cracking is examined numerically, through the development of a microstructure sensitive model in which the constitutive flow behavior of the matrix material is formulated as explicit functions of precipitate size and volume fraction. The validity of this model is examined by comparing the relative hardening parameters, including isotropic and back stress, to those obtained experimentally under monotonic and cyclic loading. This model is further used in conjunction with the grain boundary interface model, to examine the influence of microstructure on the grain boundary sliding and cracking response.

Experimental and modeling work, as outlined above, were carried out on two Nickel-based superalloy materials, ME3 and IN100, which will be detailed in Chapter 2 of the thesis. The significance of the use of this class of alloys as a turbine disk material in aero-engine applications will be reviewed in the next section, followed by justification of the research carried out in this thesis.

1.2 Significance of Nickel-Based Superalloys as Turbine Disk Materials

Precipitation strengthened Nickel-based superalloys are widely used in aircraft engines, in particular in the high pressure turbine (hot section) due to their excellent resistance to creep and stress rupture, as well as corrosion and oxidation resistance at elevated temperatures. These materials are able to maintain high strengths at temperatures greater than 0.7 times the melting temperature. Damage tolerance methodologies needed in these applications require accurate predictions of the crack growth behavior under typical service conditions. Military requirements drive toward higher temperature and longer flight capabilities. As operating temperatures and hold times at maximum stress of gas turbine discs increases, time dependent material behavior becomes increasingly important. A major factor, influencing both temperature and time dependent material behavior, is microstructure. During the operation of a disc at elevated temperatures, microstructure effects such as coarsening, solubility and diffusion become critical in assessing the material behavior. As a result, it is important to study these effects by means of dwell fatigue crack growth and deformation testing.

The major components of the jet engine are shown in Figure 1-1. The gas turbine operation is summarized as follows; Air is sucked through the fan into the compressors. The compressors are a series of vanes (which rotate) and stators (which remain stationary). The function of the compressor is to squeeze the air into smaller areas, increasing the air pressure to many times that of the atmospheric pressure. The high pressure air is then mixed with fuel in the combustion chamber and ignited,

producing hot expanding gases. The hot gases leaving the combustion chamber cause the turbine to rotate. The turbine is a series of disks and blades which are connected to the compressors by a high-pressure shaft and the rotation of the turbine will drive the compressors. The hot air will then leave the engine through the nozzle, converging the flow to a fixed area, exhausting the air at high velocity, producing thrust. The temperature and pressure profile, as seen in Figure 1-2, shows that the fan and compressor regions are the cold section, while the combustion chamber, turbines and nozzle comprise the hot region. The large variations in both temperature and pressure throughout the engine give rise to a wide variety of materials for the different parts of the engine. These materials and their properties are shown in Figures 1-3 and 1-4; respectively.

Figure 1-1: The major components of the jet engine.

Figure 1-2: The (a) temperature and (b) pressure profile in a typical gas turbine engine, adapted from reference [1].

Figure 1-3: Common materials in a gas turbine engine [1].

Figure 1-4: Yield strength as a function of temperature for typical engine materials, adapted from references [1, 2].

Figure 1-3 shows the cold section consists of materials such as Aluminum, Composites, Steel and Titanium, while the hot section is mainly composed of Nickel-based superalloys. The Aluminum and Composite materials are used in the fan and compressor housing. The Steel is used in static parts of the compressor. The Titanium, which has high strength, low density, but limited temperature capabilities, as shown in Figure 1-4, is used in the rotating compressor components. The hot section components require both temperature and strength capabilities, thus, Nickel-based superalloys are commonly used.

The component immediately following the combustion chamber (see Figure 1-1) is the high pressure turbine, which consists of disk and blade assemblies (see Figure 1-5). The entry temperature in the first row of the turbine blade system approaches 1400°C and the blade temperature is cooled by a complex internal cooling system, to

approximately 930°C. The turbine blades are single crystal Nickel-based superalloys, which have much higher creep resistance than equiaxed grain structures. The operating conditions of the high pressure turbine disk are 200-300°C in the bore and 650°C in the rim, rotational speeds greater than 10,000 rpm, with stresses reaching 1000 MPa in the bore during take-off, as well as an oxidizing and corrosive environment [5, 6]. This requires a material with high yield strength, good ductility and fracture toughness, resistance to fatigue crack initiation and growth in an oxidizing environment, and high creep resistance (particularly in the rim region) [5, 6]. Thus, the disk is made of Nickel-based superalloy, due to its strength and high temperature capabilities, as shown in Figure 1-4.

Figure 1-5: Disk and blade assembly [3, 4].

The life and safety of a turbine engine relies highly on the ability of the turbine disk to withstand failure. The main stresses in the disk are summarized as follows; dwell-time loading due to normal start-flight-landing cycles (creep), high cycle fatigue due to vibrations and airflow (fatigue), centrifugal stresses (creep) and thermal stresses

(creep). Service life is characterized by crack initiation and crack propagation under these various stress conditions. Cracking usually occurs in three main regions, as shown in Figure 1-6, in bore section from small intrinsic or processing defects (inclusions, porosities, microstructure defects), in the rim section from assembly holes, or in the rim section from the fir tree roots [7, 8]. As such, accurate predictions of service life require extensive knowledge of low cycle fatigue and creep behavior, as well as crack initiation and propagation. The work in this thesis will focus on the crack propagation. The next section will discuss various factors influencing the crack propagation, in particular those related to microstructure details in the continuum at the crack tip region.

Figure 1-6: Typical cracking locations in the turbine disk, where the arrows indicate the cracking direction [7].

1.3 Thesis Justification: Influence of Microstructure on Time-Dependent Cracking Mechanisms in Nickel-Based Superalloys

A considerable amount of research has been performed on crack growth damage mechanisms and their correlations with various loading and material parameters. It has been confirmed that there are a lot of parameters which are responsible for the crack growth, mechanical parameters, including loading stresses, frequency, loading waveform, etc., material factors, including alloy chemistry, microstructure, etc., and environment, including test temperature and gas environment [9]. A review of the role of each of these factors in crack growth damage mechanisms on high temperature alloys can be found in the work of many authors, see for example references [10-15]. The general conclusion of these studies is that the fatigue crack growth fracture modes can be classified into two distinctive mechanisms, cycle-dependent damage and time-dependent damage processes. The cycle-dependent cracking takes place along slip planes and occurs at frequencies higher than the material's transitional frequency, which is a function of the load ratio, temperature, environment and intrinsic factors of the microstructure. On the other hand, time-dependent crack growth, achieved at loading frequencies below the transitional frequency, is a thermally activated process which is characterized by an intergranular fracture path [15, 16]. The transition from time- to cycle-dependent behaviour occurs as a mixed mode regime displaying combined intergranular/transgranular fracture features. The determination of the transgranular/intergranular transitional frequency and details of mechanisms associated with each of these two fracture modes are

discussed in references [14, 17]. Transition to higher frequencies eliminates the effects of such processes, such as creep and oxidation. Additional time-dependent processes at elevated temperature include dislocation climb and slip, environmental interactions, grain boundary sliding, strain-aging, stress relaxation, creep, cavity nucleation and growth, and microstructural instabilities. Typical applications of Nickel-based superalloys, particularly in aero engine designs, include hold times at elevated temperature, thus promoting intergranular fracture.

At high temperature, grain boundaries have an important role in flow and fracture of polycrystalline metals. Grain boundary phenomena include sliding, migration, diffusion creep and cavitation. Wave shape and phases along the grain boundary influence these phenomena. Grain boundary serrations are known to impede grain boundary sliding, thus decreasing crack growth rate and improving creep properties [18, 19]. Serrations also increase the path for grain boundary diffusion. Both of these factors reduce crack propagation by slowing the formation of grain boundary microcracks ahead of the main crack [18]. This is why it is of great importance to study the effects of grain boundary serration on mechanical properties of polycrystalline materials which undergo intergranular cracking at high temperatures. In the case of superalloys with high γ' volume fraction, grain boundary serrations are generated by precipitating γ' at the grain boundaries and serrations are of a sinusoidal wave profile such as that observed in IN738LC, Nimonic 115, Mar-M-247, and MERL 76 [20]. Although a triangular wave profile has also been observed in IN718, which is generated by the pinning of grain boundaries by needle-like δ -phase [20]. In addition to serrations, many studies have dealt with the role of grain boundary

phases which are viewed as important factors in the control of grain boundary sliding, cavitation and oxidation processes taking place during intergranular cracking. Carbide precipitates influence crack growth by initiating geometrically necessary steps in the crack plane. Pédrón and Pineau [21] have studied Inconel 718 with various microstructures including fine grained, coarse grained, material with a necklace structure, and material with grain boundary β phase. They have shown that at high loading frequencies of 20 Hz, the fatigue crack growth rates are the same for all microstructures. However, with the introduction of a 300 second hold time at the maximum force, the crack growth rates differ, especially in the lower stress intensity factor range. The necklace structure gave the best crack growth resistance followed by the coarse grained and fine grained. Ma et al [16] have also shown the grain boundary NiAl- β phase in Inconel 783 alloy to be beneficial to time-dependent fatigue crack growth rate at 650°C in an air environment. Knowles and Hunt [22] have studied RR1000 at 725°C in both vacuum and air environments. They found that precipitation of the sigma phase promotes crack tip cavity nucleation and thus increases the crack growth rate. Zhao et al [23], in their work on a Nickel-based superalloy showed that the size, morphology, distribution, lattice mismatch and segregation of particles along the grain boundaries control the strength of the grain boundaries, and thus, the intergranular cracking.

In addition to grain boundary phases, gamma prime phases within the gamma matrix have been examined in relation to crack growth resistance. These precipitates are quantified in terms of primary (γ'_p), secondary (γ'_s) and tertiary (γ'_t) gamma prime. The work of Pang and Reed [24] have shown that large grain size, large coherent γ'_p

precipitate size and higher volume fractions of both γ'_s and γ'_p precipitates improves fatigue lifetimes in PM Ni-base alloys. Everitt et al [25] studied RR1000, N18 and Udimet 720 low interstitial (U720Li) with a standard and large grain variant (U720Li-LG). Fatigue crack growth rates for all materials at 650°C show that RR1000 provides the best performance, followed by U720Li-LG, N18 and U720Li. The U720Li-LG, RR1000 and N18 have been solution treated relatively close to their gamma prime solvus temperature, resulting in more solutioning of γ'_p . The reduced amount of γ'_p is expected to result in a faster grain growth. However, larger grain sizes are only observed for N18 and U720Li-LG. In RR1000, other grain boundary pinning processes, such as carbides, must be in operation, regardless of the reduction of γ'_p . The decrease in volume fraction of γ'_p leaves more gamma prime forming elements available in solution to form γ'_s and γ'_t during subsequent aging processes. Several studies have shown that the increase in γ'_s and γ'_t are beneficial to fatigue crack growth. The work of Telesman et al [26] found that a larger γ'_s precipitate size, increases the resistance to hold time crack growth. Schirra et al [27] have shown that the γ'_s and γ'_t precipitates had small effects on mechanical properties of two P/M superalloy materials, KM4 and SR3, at 649°C. The strength of both alloys decreased with larger grain sizes and larger γ'_s sizes. While there was no effect of γ'_t size on strength, increasing γ'_t size had beneficial effects on increasing the crack growth resistance. Merrick and Floreen [19] have also observed that large size, uniformly distributed γ' , as well as increased grain sizes, would decrease the intergranular crack growth rate. Knowles and Hunt [22] found that crack tip relaxation is induced by dissolution of fine γ'_t precipitates and selective coarsening of gamma prime decreases

crack growth rate. Ma et al [28] in their work on IN783, did not observe a reduced intergranular crack growth rate due to the dissolution of particles, but instead due to the increase of the secondary γ' size. Recent work by Telesman et al [29] has shown that there is no influence of the grain boundary $M_{23}C_6$ phase on hold time crack growth rate and that the key influence on crack growth rate arises from the size of γ'_t . The authors suggest that the γ'_t plays an important role in reducing the crack tip driving force by crack tip relaxation, especially during long hold times. Heat treatment procedures on LSHR alloy were focused on variations in volume fraction and size of secondary and tertiary precipitates. Hold time crack growth tests were conducted with 90 second hold time period at 704°C and results showed lower remaining stress level (more stress relaxation) corresponds to a lower crack growth rate. Their work shows that the lower remaining stress is associated with larger γ'_t size and smaller γ'_s . Similarly, Schirra et al [27], Gayda and Miner [30] have shown that increasing γ'_t size had beneficial effects on increasing the crack growth resistance. On the other hand, Jackson [31] considers that in Nickel-based superalloys, neither the size nor the volume fraction of the γ' precipitates influence intergranular cracking unless they are in the direct vicinity of the carbides along the grain boundaries.

The studies mentioned above point out to different views of the role of microstructure in the intergranular cracking process. Missing in these efforts is a systematic examination of the microstructural variable on the development of the ongoing deformation characteristics at the crack tip and affected grain boundary. This difficulty stems from the fact that an attempt to examine these effects lacks two basic prerequisites. The first is that constitutive models are generally phenomenological and

do not address explicit effects of microstructure on hardening characteristics. The second is related to the deformation field along the grain boundary. This is assumed to exist on the basis of the view that intergranular crack growth, which occurs as the decohesion of the grain boundary interface in the crack tip region, is governed by the grain-boundary traction-displacement relationships in both normal and shear directions. These displacements are related, through compatibility requirements, to the deformation field in the immediate continuum surrounding the affected grain boundary path. Localization of grain boundary stress and strain would then depend on the corresponding fields in the immediate neighboring continuum. In this, the two components, normal and tangential are equally significant in the development of damage events leading to grain boundary cracks. The normal strain is important in relation to grain boundary cavitations, while the tangential strain is relevant to the grain boundary sliding. The ability to generate two strain components depends not only on the use of a continuum phenomenological approach, but rather requires the use of a single crystal formulation that is microstructure and orientation dependent. Thus, this thesis will focus on the influence of γ' precipitates on the hardening characteristics and on the deformation field ahead of the crack tip. The resulting data and analysis of this study can be used to provide needed information on the damage tolerance of P/M materials for applications involving load reversibility at elevated temperatures. A significant outcome of this study is the global constitutive flow equation and grain boundary cohesive function, which can be used to tailor microstructure features of high temperature alloys to achieve optimum resistance to damage response associated with mission loading conditions.

The next section will outline modeling approaches that can be used to describe the continuum material deformation. It will also illustrate the vast span of scales ranging from atomic to component level and the necessary models' formulations and requirements. This will be followed by the scope of the current work.

1.4 Thesis Approach

Modeling approaches of the continuum material deformation response span various level scales, as shown in Figure 1-7. The relative modeling schemes used for each scale level, are summarized by McDowell [33, 34] as: atomistic (molecular dynamics), collections of dislocations (discrete dislocation theory, kinetic Monte Carlo methods, phase field type models), sub-grain dislocation substructures (continuously distributed dislocation theory, statistical mechanics models for distributed defects, phase field type models), single grain and polycrystalline materials (fine and coarse crystal plasticity models) and macroscopic scale (internal state variable type models).

Figure 1-7: Relative length scales associated with modeling approaches for metallic materials [33].

Molecular dynamics is used to assess processes at the atomic scale such as the energies associated with defect nucleation. This type of modeling is limited to pure metals and small time scales [33]. Discrete dislocations and dislocation substructures can be modeled by discrete dislocation model calculations [35] and/or phase field type modeling [36-38]. The phase field type modeling is based on a series of energy formulations, carried out at the precipitate/dislocation scale level. Unocic et al [36] use this type of model to study the dislocation dissociation activity as a function of magnitude and orientation of an applied shear stress, intrinsic stacking fault energy (γ_{ISF}) and microstructure parameters (γ channel width, γ' size), as shown in Figure 1-8, in a γ' strengthened Nickel-based superalloy. This figure shows the interaction between a spherical secondary γ' particle and a pure screw type $a/2\langle 110 \rangle$ matrix dislocation (Burgers vector AB). The in-plane stress is oriented between -90 and 90 degrees (with respect to the Burgers vector orientation) and its magnitude, indicated on the radial direction between 0.35 and 1.41 GPa. Under the different orientations simulated, the AB dislocation may dissociate into a leading and trailing partial dislocation ($A\delta$ and δB). The various dislocation/precipitate interaction mechanisms are shown in Figure 1-8 for the different orientations. The phase field model can be used to determine the frictional stress as a function of precipitate size and channel width, which could later be used at a higher modeling scale. Furthermore, it can be used to study the influence of factors such as γ' size, shape, channel width or lattice misfit on the active deformation modes [37]. This modeling approach can also be extended to the meso-scale (lengths greater than the Burgers vector size) to study the

dislocation interaction with secondary and tertiary γ' precipitates (i.e. more than one). In this, the influence of precipitate size and volume fractions can be mapped out.

Figure 1-8: Dislocation activity diagram showing the interaction of a spherical γ' particle and a pure screw type $a/2\langle 110 \rangle$ matrix dislocation (AB). The symbol is calculated from phase field simulations and denotes activity of the shockley partial dislocations, $A\delta$ and δB after dissociation. (A) Orowan bowing of full dislocation (B) Orowan bowing of both partial dislocations (dislocation dissociation) (C) Anti-phase boundary shearing of γ' (D) Orowan bowing of the leading partial dislocation (decorrelation) (E) dislocations arrested in front of the channel [36].

Zhou et al [38] extend this further to incorporate both dislocation and microstructure evolution simultaneously to simulate rafting of γ' precipitates in single crystal Ni-Al alloys under high temperature deformation. Validation of studies at the phase field

scale required extensive transmission electron microscopy (TEM) work in order to confirm the dominant deformation mechanisms under certain loading conditions for a given alloy. In addition, it requires knowledge of various energies, i.e. stacking fault energies, which are obtained from experiments or calculations.

Following the phase field scale are the single and polycrystalline material models. These crystal plasticity (XP) models are developed at fine and coarse scales based on finite deformation theories. In each of these scales, the crystal orientation is considered and active slip systems are calculated based on the loading direction. The formulations at the fine scale are generally dislocation density based [39, 40], while the coarse scale smears the dislocation related effects into general flow and hardening formulations [41-43]. The dislocation density is generally divided into various types including mobile and immobile dislocations, statistically stored and geometrically necessary dislocations and forest dislocations [40, 44]. Each of these categories contributes differently to the hardening parameters of the constitutive flow formulations. Unlike the phase field model, the deformation mode is not characterized by the crystal plasticity model. The active mode is implicit to the model formulations. In addition, the formulations also incorporate microstructure features such as grain size, precipitate size and volume fraction. Thus, the fine scale can be used to study the influence of these microstructure features under the active deformation mode. Similar to the fine scale, the coarse scale crystal plasticity model can explicitly incorporate microstructure features, but generally does not model dislocation density. Instead, this scale follows similar hardening formulations as the macroscopic modeling approach [42, 43]. In this, the classical hardening variables, such as isotropic and kinematic

hardening are a function of orientation and plastic shear strain. Models at this scale, as well as the fine scale, can also incorporate latent hardening phenomena [41, 45-47]. Validation of either fine or coarse scale crystal plasticity models require low cycle fatigue experiments under a series of loading conditions (monotonic, stress relaxation, cyclic, various loading rates, complex loading scenarios). The fine scale requires the knowledge of many variables which can be obtained from experiments and phase field simulations. The advantage of a coarse scale crystal plasticity model (as will be described in Chapter 5 of this thesis) is that the material parameters required are fewer than that of the fine scale and can be obtained by modification of the macroscopic material parameters. Additionally, the coarse scale model, when used in conjunction with a cohesive zone model or macroscopic material model, does not require extensive work on the transition between scales.

The final scale used in this work is the macroscopic level (specimen to component scale) in which the deformation behavior is described by an internal state variable (ISV) type model [48, 49]. The ISV type model is based on non linear kinematic hardening formulations in which the total stress is the sum of the viscous, kinematic, isotropic and initial yield stress components. These formulations are based on classical plasticity theory and do not consider the grain orientation or active slip systems as the crystal plasticity formulations did. At this scale, validation requires low cycle fatigue experiments under a series of loading conditions (monotonic, stress relaxation, cyclic) and knowledge of the active deformation modes is not a necessity. This scale also requires fewer material parameters than the finer scale models, which can be obtained directly from the low cycle fatigue experiments. Microstructure

factors can be explicitly modeled at this scale, although their influence can only be examined in a global sense.

A cohesive zone approach in which the grain boundary dislocation network is smeared into a Newtonian fluid element was used to describe the grain boundary traction displacement laws controlling the deformation and damage behavior [50]. Within the cohesive zone element, the motion of gliding dislocations in the tangential direction relates to the observed grain boundary sliding displacement the rate of which is limited by the climb of dislocations over grain boundary obstacles. Effects of microstructural variation and orientation of the surrounding continuum are embedded in the tangential stress developing in the grain boundary. For this, the crystal plasticity scale is necessary, since it considers orientation effects, allowing the displacement and tractions across the grain boundary (along the crack path) to be dissociated into both normal and tangential directions. If the ISV scale is exclusively used, the tangential component is minimal. The fracture of the grain boundary element is controlled by the rate at which the grain boundary sliding reaches a critical limit. This limit is diminished by environmental effects due to oxygen diffusion. Material constants required for the grain boundary interface laws can be obtained through experiments and calculations. Validation of this model requires knowledge of the ISV and XP material behavior and extensive crack growth testing. The crack growth testing must be carried out in air and vacuum environments to obtain the corresponding loading conditions leading to intergranular fracture (temperature, environment, loading frequency). They are also used to obtain the active grain boundary cracking

mechanisms (i.e. grain boundary sliding and dynamic embrittlement). Finally, the crack growth rate is used as a means of comparing the model and experimental results.

The modeling scales described in the previous section show the various advantages and disadvantages of each approach. The main goal of this work is to examine the influence of microstructure factors, in particular γ' size and volume fraction, on the intergranular cracking process. For this, the appropriate scales are the cohesive zone model (to describe the grain boundary deformation and damage), coarse XP model (to incorporate the normal and tangential stress and displacement components acting across the grain boundary, as the active intergranular cracking mode is due to grain boundary sliding which requires the tangential components) and macroscopic ISV model (as a stepping stone to obtain material parameters from the low cycle fatigue experiments to calculate the material parameters required for the XP model).

1.5 Thesis Overview

This thesis describes the experimental, analytical and numerical work performed in order to carry out the objectives of this research, as outlined in Section 1.1. Chapter 2 describes the Nickel-based superalloy material including processing techniques, chemistry, microstructure and heat treatments. It will also describe the characteristics of the two main alloys for the current work, IN100 and ME3. Chapter 3 describes the dwell fatigue crack growth experiments, including test setup, crack growth rate and its correlation with loading frequency. In addition, the loading

frequency is correlated with the slip band spacing and fracture mode. Furthermore, the active operating mechanisms are identified through analysis of the apparent activation energy and experimental observations. This is followed by Chapter 4, which describes the dwell crack growth modeling approach. In this, the grain boundary deformation and damage mechanisms, including grain boundary sliding and dynamic embrittlement, are incorporated into a grain boundary cohesive zone interface model. Chapter 5 describes the continuum material deformation models which are necessary to describe the stress field surrounding the crack tip in the cohesive zone model. In this, the theory of the internal state variable and coarse crystal plasticity models are described. In addition, the low cycle fatigue experiment setup and test results are discussed. Determination of the required model parameters is carried out and uniaxial one and two-dimensional simulations are performed under various strain controlled loading scenarios. Chapter 6 then combines the continuum and grain boundary modeling efforts to simulate the dwell crack growth rate. The finite element model is described and various case studies varying temperature and environment are performed and compared to the experimentally obtained crack growth rate. The influence of microstructure on intergranular cracking is then addressed in Chapter 7. In this the γ' statistics are examined by explicitly modeling their size and volume fraction in the ISV and XP material model formulations. The influence of γ' on grain boundary sliding and crack growth rate is then examined both experimentally and numerically. Furthermore, the influence of grain boundary serrations on the grain boundary viscosity, sliding and crack growth rate is examined. This is followed by

Chapter 8 outlining the main conclusions of this work and Chapter 9 detailing future recommendations.

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CHAPTER 2

NICKEL-BASED SUPERALLOYS - PROCESSING, CHEMISTRY AND MICROSTRUCTURE

2.1 Superalloys

There are three major classes of superalloys, Cobalt-, Iron- and Nickel-based alloys [1, 2]. These alloys are high performance materials and are utilized at temperatures greater than 0.7 times the melting temperature. They are used in a wide variety of applications such as aircraft engines, rocket engines, submarines, nuclear reactors, power plants and petrochemical equipment, to name a few [2]. Depending on the application, Cobalt-base alloys are generally used where a high corrosion resistance or weldability is required, at lower temperatures Iron-Nickel alloys may be used, which have a reduced cost compared to Cobalt- and Nickel-based alloys, [3, 4]. Consequently Iron-Nickel alloys are only used in their wrought condition, thus limiting the grain size control and alloying content. Due to its high strength, microstructure stability and oxidation resistance, Nickel-based superalloys are used in the hottest parts of the jet engine, mainly the turbine disk and turbine blades. These alloys are not only processed in their wrought condition, but processed using powder metallurgy as well, thus increasing alloying content and enhancing the grain size control.

2.2 Processing: Ingot and Powder Metallurgy

Ingot Metallurgy: The conventional processing route is referred to as ingot metallurgy [5, 6]. The basic steps are outlined in Figure 2-1. The first step is vacuum induction melting (VIM), in which the raw materials are charged in a ceramic crucible under vacuum to form a base alloy. This base material has a high density of cracks, porosities and refractory particles of ceramics. Therefore, this is followed by secondary processes such as electro-slag refining and vacuum arc remelting (VAR) in which undesirable oxide-containing inclusions are removed. These processes produce an ingot with better mechanical properties and also reduce chemical inhomogeneities. These steps are then followed by annealing, and deformation processing such as drawing and forging. This will homogenize the composition, in addition to producing the final shape. The material produced by these steps is commonly referred to as a wrought condition.

Investment casting is another method for fabricating of components with complex shapes. In this method, a single-use wax mold with ceramic cores is used to create complex shapes such as blades or vanes [5, 6]. These castings can result in equiaxed grain structures, columnar grained or single crystal. Directional solidification casting processes, in which a preferential orientated grain growth occurs, is obtained by maintaining a unidirectional thermal gradient during the cooling process [6, 7]. Columnar and single crystal materials are optimal for blade applications in which require superior creep resistance.

Figure 2-1: Basic steps of ingot metallurgy consisting of vacuum induction melting, electro-slag refining, vacuum arc remelting, annealing and deformation processing by drawing and forging resulting in a wrought superalloy material [5].

Wrought alloys, such as IN718 or Waspaloy, have a relatively low level of strengthening elements such as Al, Ti and Nb, thus, additional cost associated with powder processing is not justified [5]. In addition, materials processed by this route contain a limited amount of alloying and as a result has significant chemical segregation [8]. For these reasons, powder metallurgy was introduced.

Powder Metallurgy: In order to overcome some of the disadvantages of the conventional ingot metallurgy route, powder metallurgy (P/M) was adopted [5, 6]. The basic steps are outlined in Figure 2-2. Similar to ingot metallurgy, P/M starts with VIM to produce a base alloy. This is then followed by gas or vacuum atomization in which the base alloy is remelted, atomized and rapidly solidified into fine powders. Sieving is used to remove oxide-containing inclusions. These powders are collected

into steel cans, evacuated under vacuum and sealed. These cans are then processed by hot isostatic pressing (HIP) or extrusion, in order to consolidate the powder. The ingots are then forged into structural components.

Figure 2-2: Basic steps of powder metallurgy consisting of vacuum induction melting, followed by gas or vacuum atomization to produce fine powders which are collected into steel cans and processed by hot isostatic pressing or extrusion followed by forging [5].

P/M alloys, such as Udimet 720 or the two alloys as part of this thesis work, Inconel 100 and ME3, have many advantages over wrought or cast alloys. The P/M process improves the compositional homogeneity by reducing the segregation and impurities from processing [5]. The extent of chemical segregation cannot exceed the

size of the powder particle [8] produced by the atomization step. In addition, P/M alloys have a smaller grain size and typically coarsen in a more controlled and uniform manner [9]. Although P/M processing increases the cost, it also allows for processing of large dimension disks made of superalloys with higher γ' fraction and/or with higher content of refractory metals (Mo, W, Nb, Ta) [5]. The material produced by P/M processing is then further optimized through various heat treatment stages to obtain a desired microstructure.

2.3 Chemical Composition

The modern superalloy contains 10+ alloying additions (plus impurities) [10], each with varying roles. The base alloy, Ni, constitutes 25-50% of the solid solution. The two main strengthening mechanisms in these alloys are solid solution hardening and precipitation hardening. The solid solution strengthening is enhanced by elements such as Co, Cr, Fe, Mo, W, Ta, and Re [10, 11], which partition to the austenitic γ matrix and have an atomic radii similar to that of Ni [5], only 3-13% different than that of Ni [1]. The addition of refractory elements such as Nb, Mo, Ta, W and Re increase the high temperature strength of the superalloy. Co has a large influence on the solubility of γ' , by raising the solidus and it may also raise or lower the solvus temperature [12]. The precipitation hardening is enhanced by γ' forming elements like, Ni, Ti and Al, which have an atomic radii larger than that of Ni [5], 6-18% different than Ni [1]. Grain boundary strengthening by carbide formation is from elements such as, W, Ta, Ti, Mo, Nb, Hf, and Cr [10] or by boride formation from elements such as

Cr and Mo [10, 5]. The elements B, C, Zr, and Hf act as grain boundary refiners. In addition, B, C and Zr segregate the grain boundary, because their atomic size is very different compared to that of Ni [5], 21-27% different than Ni [1], reducing sliding and diffusional processes [10]. Alloys such as Al, Cr, Y, La, Ta and Ce improve oxidation resistance, while Cr, Co, Si, La, and Th improve hot corrosion resistance [10]. Al forms a protective surface oxide, while Cr forms Cr_2O_3 which protects from oxygen, nitrogen and sulfur. The main effects of the alloying elements in Ni-based superalloys are summarized in Table 2-1.

Table 2-1: The roles of alloying elements in Nickel-based superalloys [11].

	Ni	Co	Fe	Cr	Mo, W	Nb, Ta, Ti	Al	C, B, Zr, Hf
Solid Solution	X	X	X	X	X			
Grain Boundary								X
Precipitation						X	X	
Carbide Formers				X	X	X		
Oxide Formers				X			X	

Additional factors, such as coherency strains at the gamma-gamma prime interfaces, lattice mismatch, etc., also add to the precipitation hardening [11]. The effects of alloying elements on grain boundary strength, as well as grain boundary phases, have been the main focus during high temperature intergranular crack growth. Thus, most studies have been focused on the effects of elements such as Nb, Ta, Hf, B and Zr, which have been directly correlated with the grain boundary strength or grain boundary phases.

Effects of Alloying Elements: Everitt et al [13] have shown that grain size alone does not account for the crack propagation rates. They show that RR1000, with an intermediate grain size compared to U720Li and U720Li-LG, has the lowest crack propagation rate. The influence of additional features, such as alloy chemistry and coherent gamma prime distributions, play a role in crack propagation at elevated temperatures. The addition of Hf to the RR1000 and N18 alloys leads to Hf enriched MC carbides, opposed to Ti or Ta enriched MC carbides. The addition of Ta to RR1000 leads to Ta enriched MC carbides. These carbides are commonly located at grain boundaries. The decomposition of the MC carbides during subsequent heat treatments and exposure at high temperatures causes the formation of carbides such as the $M_{23}C_6$. This carbide is commonly enriched by Cr and forms primarily at grain boundaries. The rate of decomposition of Ta or Hf enriched MC carbides is the controlling factor in the rate of formation of $M_{23}C_6$ carbides. Since the diffusion of Hf and Ta is slow, it is used to fine tune the chemistry of $M_{23}C_6$ carbides at grain boundaries. The formation of Cr enriched $M_{23}C_6$ carbides causes a local depletion of Cr causing a lower corrosion resistance and lower crack growth resistance in air environments. The presence of grain boundary carbides can also reduce grain boundary sliding, which in turn provides a beneficial effect to fatigue crack growth resistance, especially in a vacuum environment or during dwell loading conditions. Telesman et al [14] have designed several compositions of Alloy 10 to study the effect of Nb, Ta and Co on time dependent crack growth behavior. They believed Nb accelerates crack growth rate while tantalum retards crack growth rate. But their work has shown that the crack growth resistance is insensitive to the significant changes in

the Nb, Ta and Co content, as well as Nb to Ta ratio, in the Alloy 10 composition, within the scope of compositions investigated. These effects are illustrated in da/dt as a function of K_{max} , where the various alloy compositions collapse to a single curve for the two heat treatment conditions of subsolvus and supersolvus solutioning. This lead to inconclusive results which identify the grain boundary species responsible for the enhanced crack growth rate in oxygen environments. Increasing amounts of C refines the grain size, which in turn reduced creep and hold time crack growth rates. Huron et al [15] have shown that Hf had minor effects on lowering yield strength and reducing hold time crack growth rates, while Mg had negligible effects. However, Ta had significant effects, where increasing Ta improved both creep life as well as crack growth resistance in KM4 at 704°C.

The Effect of Boron: The element B has been commonly used to increase grain boundary strength of polycrystalline superalloys. Single crystal alloys are generally free of B since they have no grain boundaries. B has been introduced in some large single crystal components where low angle grain boundary defects are unavoidable [16]. B combines to form intergranular borides of the M_3B_3 type. B and Zr additions increase grain boundary strength, decrease grain boundary diffusivity and lower grain boundary surface energy. B has also been added at low levels to lower the alloy's solidus temperature. Zr is believed to remove Sulfur from the grain boundaries and lower grain boundary diffusion rates [17]. Small additions of B and Zr can be extremely beneficial to the creep rupture properties of Nickel-based superalloys, where they help prevent premature cavity and micro-crack formation at grain

boundaries [18]. Xiao et al [19] have studied the effect of B, ranging from 12 to 100 ppm, on the fatigue crack growth rate in Inconel 718 at room temperature and 650°C. Their results show B improves the fatigue crack propagation resistance and improved the low cycle fatigue life of IN718 [19, 20]. The fatigue threshold at 650°C increased with the addition of B. In addition, specimens tested at 650°C show a transition in the cracking mode with varying amounts of B. The cracks propagated with an intergranular mode in specimens with a B concentration of 12 ppm and by a mixture of intergranular and transgranular mode in the alloy with 29 ppm and 60 ppm B. In the specimens with even higher B (concentration of 100 ppm), mostly transgranular fracture was observed. This transition in the cracking mode with the addition of B shows that B plays an important role in increasing grain boundary strength in IN718. This demonstrates the main mechanism for improvements in crack growth resistance is due to B segregation increasing grain boundary cohesion. The segregation of B at grain boundaries can also tie up vacancies and reduce grain-boundary diffusivity, and thus improve the resistance to an Oxygen environment. Another possible mechanism for the improvement could be from the formation of boride particles. These particles impede the dislocation slip and make it more difficult for cross slip to occur, and thus enhance the fatigue crack growth resistance. At lower B concentrations, cross slip can easily occur, causing plastic deformation to be concentrated in local regions leading to crack initiation. Huron et al [15] carried out various compositional studies on KM4 and have shown that B is the most influential element, with increased B leading to grain boundary filming and increased triple point void formation and increased tendency for thermally induced porosity. Although the increase in B reduced creep

life, it was beneficial to rupture life (at 649°C), rupture ductility and to a lesser extent hold time crack growth rates (at 704°C).

The Effect of Niobium: Gao et al [21] identify grain boundary Nb carbides as the phase most susceptible to environmental attack. They proposed NbC phases at the grain boundaries decompose to produce Nb oxides which embrittle the grain boundaries. They also suggest that segregated Nb in the form of Ni_3Nb precipitates at grain boundaries further embrittles the grain boundaries [21, 18]. In addition, they suggest NbC oxidation is the rate controlling process for environmentally enhanced creep crack growth in the Inconel 718. These theories are supported by a strong correlation between environmental sensitivity, which is defined as the ratio between crack growth rate in air and the corresponding rate in an inert environment, and Nb concentration. Liu et al [22] investigated the effect of various thermal treatments on the hold-time fatigue crack propagation behavior of 718Plus alloy. The hold-time fatigue crack propagation rates of the specimens with the grain boundary δ -phase have shown improvements. Normally the δ -phase is considered to be a detrimental phase, by consuming Nb and reducing the alloy's strength. Lui et al suggest that the improvement could be from the depletion of Nb in the matrix so that fewer NbC particles are present. Antolovich has suggested that blocky δ -phase distributed along the grain boundaries decreases the environmental effects by trapping oxygen at the δ -matrix interfaces [23].

2.4 Microstructure

2.4.1 Phases

The microstructure of the Nickel-based superalloy consists of a matrix phase with various precipitated phases; the major phases present are as follows:

Gamma Matrix (γ): The gamma matrix (γ) is a disordered face center cubic (FCC) crystal structure, as shown in Figure 2-3 [5, 12]. In this crystal structure Ni atoms occupy the majority of the lattice sites, with a disordered arrangement of Al and other solutes such as Co, Cr, Mo or W.

Figure 2-3: FCC crystal structure of γ matrix phase [5, 8].

Gamma Prime (γ'): The primary strengthening phase of the Nickel-based superalloy is the gamma prime (γ') precipitate, with a composition of $\text{Ni}_3(\text{Al,Ti})$ [5, 12]. Some alloys can have up to 70% volume fraction of γ' [1]. This phase has an ordered L1_2 FCC crystal structure, as shown in Figure 2-4, where Al or Ti atoms occupy the corners and Ni atoms occupy the face.

Figure 2-4: Primitive $L1_2$ FCC crystal structure of γ' phase [5, 8].

This phase is coherent with the γ matrix, thus crystallographic planes of precipitate and matrix are continuous. In addition, there is a small lattice misfit between the precipitate/matrix phases, promoting homogeneous precipitation throughout the γ matrix. The particle-matrix misfit, δ , is a function of the lattice parameters of the γ and γ' phases given as a_γ and $a_{\gamma'}$ respectively, and is mathematically expressed as [5]:

$$\delta = 2 \left(\frac{a_{\gamma'} - a_\gamma}{a_{\gamma'} + a_\gamma} \right) \quad (2-1)$$

The misfit is strongly dependent on the partitioning of solutes between γ and γ' , as well as, the size of the γ' precipitate. Misfit is positive when γ' has a larger lattice parameter than γ and misfit is negative when γ' has a smaller lattice parameter than γ . A negative misfit stimulates the formation of rafts of γ' (layers of the phase in the direction normal to applied stress), which reduce creep rate if the mechanism involves climb of dislocations across the precipitate rafts.

The γ' precipitate as various sizes, depending on the heat treatment of the alloy. A correlation exists between particle size and particle morphology based on a minimum surface energy criterion, where small particles exist as spheres and the increase in size modifies the shape to cubes, arrays of cubes, and dendrites [5]. In

general, γ' particles occur either as a bimodal (tertiary and secondary) or a trimodal (tertiary, secondary and primary) particle distribution, as illustrated in Figure 2-5.

Figure 2-5: Schematic illustration of a microstructure with a trimodal γ' particle distribution [24].

The large gamma prime, which is generally 1-10 microns in size, is called primary gamma prime (γ'_p). This phase normally occupies the triple junctions and pin the γ grain boundary during the (sub-solvus) solutioning phase, thus restricting grain growth [5]. The intragranular γ' particles are the secondary and tertiary gamma prime precipitates. The secondary γ' (γ'_s), appear cubical in shape with a size in the range of 70-500 nm, while the smaller ones, tertiary γ' (γ'_t), seem to be spherical with a size 5-50 nm. All precipitates, including γ'_p , are coherent with the γ matrix.

Gamma Double Prime (γ''): In Nickel-Iron superalloys, such as Inconel 718, 706 or 725, with high Nb (or Va) content, the gamma double prime (γ'') phase can precipitate [5, 12]. This disc phase has a composition of Ni_3Nb or Ni_3V , with an ordered body centered tetragonal (BCT) crystal structure, as shown in Figure 2-6.

Figure 2-6: BCT crystal structure of γ'' phase [5, 8].

In these alloys, the γ'' is the primary hardening phase rather than the γ' . The γ'' phase provides higher strength up to 650°C, but is unstable above that, a decrease in hardening will result if the equilibrium Ni_3Nb orthorhombic phase (δ -phase) is allowed to form.

Carbides: In addition to the γ' precipitates, there are carbide phases which can be both beneficial and detrimental to the mechanical properties. The strongest carbide formers in order of strength are Hf, Zr, Ti, Nb, Ta, V, Mo, W. Carbides with the chemical composition MC (M = W, Ta, Ti, Mo, Nb, Hf) are large blocky, globular particles, which are undesirable [5, 25, 26]. These carbides usually form at high temperature

from the liquid phase, and one main objective of the subsequent heat treatment processes is to break down these carbides to form smaller more desirable ones. The chemical reaction, $MC + \gamma \rightarrow M_{23}C_6 + \gamma'$, occurs at lower temperatures $\sim 750^\circ\text{C}$ during the heat treatment process or during prolonged periods of service exposure [5, 26]. The resulting carbides, $M_{23}C_6$ ($M = \text{Cr, Mo, W}$) are grain boundary particles which enhance creep rupture strength. Other carbides form from a similar decomposition, $MC + \gamma \rightarrow M_6C + \gamma'$, where M_6C ($M = \text{Mo, W}$) are small platelets residing within the grains or as blocky particles in grain boundaries, these decrease ductility and stress rupture properties, but are useful for grain control [2]. Carbides of the form M_7C_3 ($M = \text{Cr}$) are discrete particles. The above mentioned carbides have an FCC crystal structure and in general, ones that form on the grain boundary are beneficial, reducing grain boundary sliding, improving creep and fatigue crack growth resistance.

Borides: Elements such as Cr or Mo can also combine with B to form borides [5, 26]. These phases reside along the grain boundaries. They form as M_3B_2 , where M is $(\text{Mo}_{0.48}\text{Ti}_{0.07}\text{Cr}_{0.39}\text{Ni}_{0.03}\text{Co}_{0.03})$ or $(\text{Mo}_{0.31}\text{Ti}_{0.07}\text{Cr}_{0.49}\text{Ni}_{0.06}\text{Co}_{0.07})$ [2, 26].

TCP Phases: Undesirable brittle phases which tie up γ and γ' strengthening elements can form in alloys containing transition metals, i.e. Ta, Nb, Cr, W, Mo [5, 12, 26]. These phases are most likely to form topologically close-packed phases (TCP) such as σ , μ or laves. The σ phase has a chemical composition of $(\text{Cr,Mo})_x(\text{Ni,Co})_y$, which is hard and plate-like. The μ phase has a similar composition to σ phase with a majority

of Mo and Co. Laves have a chemical composition, AB_2 , such as $MoCo_2$ and $TaCo_2$. These phases reduce creep strength and can act as crack initiators.

2.4.2 Grain Boundary Morphology

At high temperature, grain boundaries have an important role in flow and fracture of polycrystalline metals. Grain boundary phenomena include sliding, migration, diffusion creep and cavitation. Wave shape and phases along the grain boundary influence these phenomena. Grain boundary serrations are known to impede grain boundary sliding, thus decreasing crack growth rate and improving creep properties. This is why it is of great importance to study the effects of grain boundary serration on mechanical properties of polycrystalline materials which undergo intergranular cracking at high temperatures.

Grain boundary serrations generated by the pinning of grain boundaries by needle-like δ -phase create a triangular wave profile as in IN718 [27]. Similarly, the work of Koul and Gessinger have shown triangular shaped serrated grain boundaries, in 20-11P austenitic stainless steel, are formed by the precipitation of coarse $M_{23}C_6$ carbides that grow from one grain into the matrix of another grain [28]. The grain boundary segment b-b is shorter and has a lower energy configuration than a-a, thus, forms a serrated boundary, as seen in Figure 2-7.

Figure 2-7: Schemata for serrated grain boundary formation by precipitation of $M_{23}C_6$ carbides. Straight boundaries, a-a, are formed during solutioning, and serrated boundaries, b-b, are formed after $M_{23}C_6$ precipitates [28].

In the case of superalloys of high γ' volume fraction, grain boundary serrations are generated by precipitating γ' at the grain boundaries and are of a sinusoidal wave profile such as IN738LC, Nimonic 115, Mar-M-247, and MERL 76 [27]. Danflou, Marty and Walder's [29] work on Astroloy have shown that grain boundary serrations are formed by the precipitation of coarse γ' precipitates along the grain boundary, by heat treating above the solvus followed by slow cooling. The average wavelength of serration was found to be directly related to the size of γ' precipitates located along grain boundaries [29]. The size of these coarse γ' precipitates can be controlled by the heat treatment parameters which include homogenizing temperature, cooling rate and cooling range.

There are prerequisite conditions for serration to occur. Carbides and borides will inhibit grain boundary displacement, producing a straight grain boundary. Serrated grain boundaries can be suppressed if there are other grain boundary precipitates that precipitate before γ' (i.e. boro-carbide precipitation in Alloy APK-1). Thus, the γ' solvus must be higher than the solvus of grain boundary carbides, $M_{23}C_6$,

MC or M_6C or borides. In the case of Nimonic 105, cooling from solution 1170°C to 1000°C, $M_{23}C_6$ carbide precipitates before γ' , suppressing serrated grain boundaries and develops straight boundaries [28]. There is some speculation that grain size may play a role in serration. In ultra-fine grained material, if some carbides precipitate prior to γ' precipitation, there is a large grain boundary area and carbides will precipitate far apart allowing serrations to form in some locations where there are no carbides. The γ' precipitate size must be larger than a critical size. Lastly, serration is dependent on solutioning temperature and cooling rate from solutioning [30].

The homogenizing temperature affects both grain growth and the capability of boundary migration during the formation of serrations. A supersolvus heat treatment, as seen in Figure 2-8, which dissolves $M_{23}C_6$ carbides and γ' particles into solid solution so that the grain boundaries are free to move, is a prerequisite for development of large serrations. A subsolvus heat treatment restricts the growth of serrations to several hundred nanometers [29, 30]. Table 2-2 below summarizes solvus temperature and serration occurrence of some common Nickel-based superalloys. This emphasizes that for grain boundary serrations to occur, the γ' solvus must be higher than solvus of grain boundary carbides [28].

Cooling rate partly determines the amplitude of the serrations [29]. If the material is cooled rapidly after solutioning through the γ' precipitation range, the γ' particles are small and the boundary migration is limited [Henry et al 1993, 28]. Thus slow cooling, as seen in Figure 2-8, through the γ' precipitation range is essential to produce grain boundary serrations. Koul and Gessinger's work on Nimonic 115, that

is furnace cooled at 0.5-2.33°C/min, show serration amplitude and wavelength increase with decreasing cooling rate from 2.33 to 0.5 [28].

Figure 2-8: Schemata of temperature versus time for the solutioning heat treatment cycle. The supersolvus temperature and slow cooling rate indicated by the solid red lines are prerequisites for formation of serrated grain boundaries.

Table 2-2: Nickel-based superalloys with serrated grain boundaries, where FC = furnace cooled and AC = air cooled [28].

Alloy	M ₂₃ C ₆ solvus temperature (°C)	γ' solvus temperature (°C)	Serrated grain boundaries formed or not	Minimum cooling rate required
Nimonic 115	1080-1100	1140-1160	Yes	3°C/min
IN 738	1000	1160-1175	Yes	FC
IN 792	1000-1050	1100-1150	Yes	AC
Rene 95	Low	1120-1160	Yes	FC
Astroloy LC	1000	1120	Yes	FC
Nimonic 105	1100-1150	1020-1060	No	-

The average amplitude of serrations and distribution of serrated boundaries are influenced by the temperature at the end of furnace cooling. Amplitude and

wavelength of the serration become more uniform when the specimen is held at the temperature at the end of furnace cooling [29]. The amplitude of the serrations also increase with the misorientation between grains. The experimental results showed that serration of small amplitude were associated with high coincidence boundaries, while serrations of large amplitude with low coincidence boundaries or random boundaries [29].

Danflou et al [29] report that Koul and Gessinger's work suggested that γ' nucleates on one side of the grain boundary and due to the presence of the "open" grain boundary in contact with the γ' particle. Koul and Gessinger [28] show a net strain energy differential between interface A and B of the γ' particle provides a driving force to move the γ' particle into the direction of the grain boundary, as seen in Figure 2-9, and that high-angle boundaries migrate more easily than low-angle boundaries.

Figure 2-9: Schemata of the migration of the γ' precipitate. Where ΔG (Side A) > ΔG (Side B), providing the driving force for migration of the γ' precipitate in the direction of the grain boundary [28].

Free energy of a crystal with fully coherent spherical precipitates is given as the sum of the coherency strain energy and the chemical interfacial energy [28, 30]:

$$\Delta G_{coherent} = \Delta G_S + \alpha_{ch} A \quad (2-11)$$

$$\Delta G_S = 4\mu\delta^2 V \quad (2-12)$$

where ΔG_S is the coherency strain energy, α_{ch} is the chemical interfacial energy, A is the precipitate area (given by equation 2-4), μ the shear modulus of the matrix, δ is the particle-matrix misfit parameter, V is the particle volume (given by equation 2-3) and r is particle radius. Substituting equations (2-3), (2-4) and (2-12) into (2-11) yields a new expression for the coherent free energy expressed as:

$$\Delta G_{coherent} = \frac{16}{3} \pi r^3 \mu \delta^2 + 4\pi r^2 \alpha_{ch} \quad (2-13)$$

Semicoherent or incoherent interface relieves the effects of misfit (i.e. a grain boundary of a finite thickness, $\Delta G_S = 0$), but an extra structural interfacial energy is added to the chemical interfacial energy and the free energy is given as [28, 30]:

$$\Delta G_{semicoherent} = 0 + A(\alpha_{ch} + \alpha_{st}) = 4\pi r^2 (\alpha_{ch} + \alpha_{st}) \quad (2-14)$$

where α_{st} is the interfacial energy due to structural distortion caused by the misfit dislocations. When $\Delta G_{coherent}$ (Side A) $>$ $\Delta G_{semicoherent}$ (Side B), there is an energy differential between the two sides A and B of the γ' particle. The migration of the γ' particle will be in the direction of the lowest energy, in this case side B, the grain boundary.

There is a minimum γ' particle radius, below this the free energy balance between sides A and B is negligible. This critical particle size, $r_{critical}$, can be determined by equating the coherent and semicoherent free energies [28, 30]:

$$r_{critical} = \frac{3\alpha_{st}}{4\mu\delta^2} \quad (2-15)$$

Figure 2-10 shows for a given misfit parameter, the coherent energy and the incoherent energy as a function of particle radius. At the critical radius value, the favorable state changes (i.e. the lowest energy state). When the precipitate particle radius is smaller than $r_{critical}$, it is more favorable to be coherent, this state gives the lowest total energy. On the other hand, if the precipitate particle radius is larger than $r_{critical}$, it is more favorable to be semicoherent or incoherent [31].

Figure 2-10: Coherent and incoherent free energy as a function of particle radius for a spherical precipitate [31].

More recent work by Danflou et al [32] and Henry et al [33] have shown that serrations in a Nickel-base superalloy can also be produced by asymmetrical precipitation growth of γ' through slow cooling through the temperature range just below solvus. The precipitates grow in the direction of the incoherent boundary because of the solute diffusion from both the lattice and rapid diffusion along the grain boundary as seen in Figure 2-11.

Figure 2-11: Schemata of the asymmetric growth of the γ' precipitate, with arrows indicating the solute flux [33].

Another theory is given in the work of Wlodek, Kelly and Alden, where they document that serrations are a result of grain boundary movement during sub-solvus cooling from the solutioning temperature. They describe that serrations are not due to growth of γ' into the grain boundary, but by dynamic movement of the grain boundary into freshly nucleated γ' secondary. For constant cooling rate, the force driving this movement increases with the length of unsupported grain boundary [34].

2.5 Heat Treatment

After the P/M processing the forged material microstructure is optimized through a series of heat treatments. The solidification stage can result in non-uniform precipitate distribution or formation of secondary detrimental phases, such as MC carbides [12]. Thus, the heat treatment is essential to obtain a uniform particle distribution and break down the large MC type carbides. In addition, the heat treatment cycle is optimized in relation to the service temperature in order to obtain a stable microstructure in service. The typical heat treatment sequence for Nickel-based

superalloys involves a three stage process consisting of solutioning, stabilization and aging, as illustrated in Figure 2-12.

Figure 2-12: Schematic of typical heat treatment stages for a Nickel-based superalloy.

Solutioning: The first stage is called solutioning. The time at solutioning is typically 1-3 hours, depending on the temperature level (time decreases as temperature level increases). This step occurs at a temperature level above (super-solvus) or below (sub-solvus) the γ' solvus temperature and below the solidus temperature (temperature at which a liquid forms) [12]. The γ' solvus and solidus temperatures are highly dependent on alloy chemistry, thus, these temperatures vary from alloy to alloy. The γ' solvus for most alloys lies between 1050-1200°C [5], therefore, solutioning is typically at a temperature in the range of 900-1300°C. During sub-solvus solutioning only secondary γ' , tertiary γ' and carbides, will dissolve, while the primary γ'

precipitates remain intact [5, 35]. The primary γ' particles may also coarsen during the solutioning stage as a function of time, depending on the temperature selection. The temperature selection can also control the volume fraction of primary γ' precipitates and as a result, limit the available elements for formation of secondary and tertiary γ' during the subsequent heat treatment stages. The primary γ' particles have a grain pinning effect and restrict the γ grain growth during the solutioning stage [5]. Thus, sub-solvus solutioned materials typically have a trimodal γ' particle distribution (γ'_p , γ'_s and γ'_t) and tend to have a smaller grain size. On the other hand, during super-solvus solutioning, the primary γ' precipitates, as well as, secondary γ' , tertiary γ' and carbides, will dissolve. This process, results in grain growth and a bimodal γ' particle distribution consisting of γ'_s and γ'_t . Thus in general, lower solution temperatures produce a finer grain size resulting in better tensile and fatigue properties, while higher solution temperatures produce coarser grain size resulting in better long-term stress-rupture and creep properties, and lower tensile yield strength. During cooling from solutioning to room temperature, the secondary γ' precipitates will come out of solution. The cooling rate will control the size of the secondary γ' , fast quenching causes formation of very fine γ' , while slow cooling allows nucleation and growth to take place resulting in larger secondary γ' . During fast quenching, the secondary γ' volume fraction will increase at the expense of the tertiary γ' volume fraction. Following the secondary γ' precipitation, hyperfine γ' are also formed, but these are dissolved again in the subsequent aging steps. In addition, grain boundary serrations

may also form during cooling from solutioning, this process will be discussed in details in the following section (section 2.6).

Stabilization: The second stage is called stabilization. During this stage, the hyperfine γ' formed by cooling from solution are dissolved and the secondary γ' particles are coarsened. The temperature level is selected as to be below the primary γ' solvus and above the tertiary γ' solvus, thus, only the secondary γ' particles are controlled during this stage. The temperature and time will control the amount of coarsening that takes place. In addition, the temperature level can effect the volume fraction of secondary γ' , a higher temperature will dissolve more γ' , thereby reducing the secondary γ' volume fraction and increasing the tertiary γ' volume fraction. The volume fraction of tertiary γ' is dictated by the relative volume fractions of the primary and secondary γ' precipitates. During cooling from stabilization to room temperature, the tertiary γ' precipitates will come out of solution.

Aging: The third stage is called aging. During this stage, the kinetics are insufficient to modify the primary and secondary γ' precipitates, thus, this stage mainly controls the tertiary γ' coarsening. The temperature level selected here is below the tertiary γ' solvus temperature and above the service temperature. Furthermore, the aging temperature can also promote decomposition of MC carbides as previously discussed [5].

Precipitation Kinetics: The kinetics controlling precipitation are nucleation, growth and coarsening. Precipitation transformations can be expressed as [31]:

$$\gamma^{ss} = \gamma + \gamma' \quad (2-2)$$

where γ^{ss} is a supersaturated solution, γ' is a stable precipitate, γ is a stable solution with the same crystal structure as γ^{ss} but with a composition closer to equilibrium. During the solutioning stage, the γ' phase is soluble in the γ matrix. During cooling from solutioning, as the temperature decreases, a limited solubility exists, and thus, the supersaturated (unstable γ' in γ solution) solution will decompose into two phases and nucleation of γ' will occur. This is followed by growth of the nuclei and coarsening of the precipitates, although the three processes can overlap in time [36].

The free energy associated with nucleation has three contributions; volume free energy, interfacial energy and misfit strain energy [31]. The creation of volume, V , of γ' phase will cause a reduction in energy, $V\Delta G_V$, this is the volume free energy. This energy is a function of the time and the level of supersaturation [36]. The creation of the γ/γ' interface of area, A , will cause an increase in energy, $A\alpha$, where α is the interfacial energy. In addition, an increase in energy will occur as a function of the misfit strain energy over the volume of the precipitate, $V\Delta G_S$. For a spherical nucleus volume and area are defined as:

$$V = \frac{4}{3}\pi r^3 \quad (2-3)$$

$$A = 4\pi r^2 \quad (2-4)$$

The summation of these energies gives the total free energy as:

$$\Delta G = -V\Delta G_V + A\alpha + V\Delta G_S = \frac{4}{3}\pi r^3(\Delta G_S - \Delta G_V) + 4\pi r^2\alpha \quad (2-5)$$

Nucleation will occur when the total free energy overcomes the activation energy barrier, ΔG^* , at which a critical nucleus size exists. The corresponding ΔG^* and critical size, r^* , are given by [31]:

$$\Delta G^* = \frac{16\pi\alpha^3}{3(\Delta G_V - \Delta G_S)^2} \quad (2-6)$$

$$r^* = \frac{2\alpha}{(\Delta G_V - \Delta G_S)} \quad (2-7)$$

A nucleus is bonded by a combination of coherent, semicoherent and incoherent interfaces. Growth of the nuclei depends on the migration of these interfaces. The incoherent interfaces have the highest mobility followed by semicoherent interfaces. Thus, nuclei with incoherent interfaces will grow faster resulting in a thin disc or plate precipitate morphology [31]. The γ' will nucleate with coherent interfaces, thus, the interface migration and nucleus growth will maintain a spherical morphology. Growth is the process in which a nucleus is trying to reach thermodynamic equilibrium. This depends on the local concentration gradient of mass flow of atoms from the matrix to the particle. As the particles become larger, the gradient becomes smaller and the flux of atoms towards the particle decreases with time [36].

The microstructure of a system is unstable if the interfacial energy is not at a minimum [31]. Thus, coarsening occurs, in which the total interfacial energy of a system (i.e. sum of interfacial energy of all the particles) is lowered. This process is referred to as Ostwald ripening, in which the larger particles grow at the expense of

the smaller particles, while maintaining a constant volume fraction. A system with a high density of small particles has a higher total interfacial area than a system with a low density of larger particles. Assuming volume diffusion is the rate controlling factor, the mean particle radius, $\bar{r}$, will increase as a function of time following the law [31]:

$$\bar{r}^3 = r_0^3 + Kt \quad (2-8)$$

Where r_0 is the mean radius at $t = 0$, t is time and K is the growth rate which is proportional to the product of the diffusion coefficient, D , interfacial energy, α , and equilibrium solubility of very large particles, X_e . The rate of coarsening increases with temperature as D and X_e are exponentially dependent on temperature. It is also important to note that the coarsening rate, $d\bar{r}/dt$, is proportional to $K/\bar{r}^2$, thus a distribution of small particles would coarsen more rapidly [31].

The distribution of particle size is assumed to follow the Lifshitz-Sloyozov-Wagner (LSW) type of distribution. This type of distribution assumes the particles which have been coarsened during the heat treatment have reached a steady state distribution, P , and is given by Ardell as [37, 38]:

$$P(u) = \begin{cases} \frac{4u^2}{9} \left(\frac{3}{3+u} \right)^{7/3} \left(\frac{-3/2}{u-3/2} \right)^{11/3} \exp\left(\frac{u}{u-3/2} \right) & \text{for } u < 3/2 \\ 0 & \text{for } u \geq 3/2 \end{cases} \quad (2-9)$$

where u is the particle radius, r , normalized by the mean particle radius, $\bar{r}$. The function P has two features:

i) a maximum at $u = 1.5$ ($r = 1.5 \bar{r}$)

ii) $\int_0^{\infty} P(u) du = 1$

Below describes the process in order to plot the experimentally measured particle distribution in terms of P . First, the equivalent particle radius was measured experimentally for a minimum of 350 particles for a given microstructure. The particle distribution is plotted in Figure 2-13a as frequency (F) versus r . The axes in Figure 2-13a are converted from F versus r to $z(u)$ versus u , where $z(u) = Fu^2$. This new distribution is shown in Figure 2-13b. This is curve fit to a Gaussian type distribution of the form:

$$z(u) = Z_1 \exp\left(-0.5\left(\frac{u - Z_2}{Z_3}\right)^2\right) \quad (2-10)$$

where Z_1 , Z_2 and Z_3 are constants. This curve is shown in Figure 2-13b.

Figure 2-13: (a) Frequency versus particle radius, (b) $z(u)$ versus u and (c) experimental particle distribution with superimposed LSW distribution, P , versus u for a microstructure with an average radius of 37.9 nm.

The integral, $\int_0^{3/2} z(u) du = \frac{1}{G}$, is calculated to determine the constant G . In order to normalize the experimental data $z(u)$ is multiplied by a factor of G such that $\int_0^{3/2} Gz(u) du = 1$. This can be plotted with the distribution P as in Figure 2-13c as a function of u .

2.6 IN100 and ME3 Material Characterization

Two materials are employed in this thesis, Inconel 100 (IN100) and ME3 alloy. Both are Nickel-based superalloys processed by the P/M route, used in the disc of the jet engine. The chemical compositions of these alloys are given in Table 2-3. The additional refractory elements, W and Nb increase the high temperature strength of ME3 as compared to IN100. These elements, along with the supplementary Ta, are solid solution strengtheners, gamma prime and carbide formers [11, 12].

Table 2-3: Chemical composition (in weight %) of IN100 [39] and ME3 [40] alloys, with Ni as the balance.

Alloy	Al	Ti	Co	Cr	Mo	V	Zr	B	C	W	Ta	Nb
IN100	4.85	4.24	18.23	12.13	3.22	0.71	0.071	0.02	0.072	-	-	-
ME3	3.5	3.7	20.6	13.0	3.8	-	0.05	0.03	0.04	2.1	2.4	0.9

The heat treatment of the as received IN100 material is a three stage process consisting of: i) sub-solvus solutioning stage at 1149°C for two hours followed by oil quenching, ii) stabilization at 982°C for one hour followed by fan cooling, and iii) aging at 732°C for eight hours followed by air cooling. The average Rockwell C

hardness of the as received material is 42.7 HRC and its microstructure which is shown in Figure 2-14a, includes primary γ' (γ'_p) located at the triple points of the grain boundaries as well as within the grains and carbides, located mostly along the grain boundaries. In order to reveal the grain boundaries, the specimens were etched for 3 minutes in Kallings waterless etchant (2g CuCl_2 , 40ml HCl , 40-80ml Ethanol). Due to the sub-solvus solutioning condition, the primary γ' inhibit grain growth resulting in an average grain size of 5 μm (ASTM 12), as shown in Figure 2-14a. The grain size is reported as a mean intercept length [41], which will be detailed following the ME3 material characterization. Sizes (equivalent particle diameter) and volume fractions of the γ'_p are 1.8 μm and 24.2 %, respectively, the distribution of γ'_p particles is shown in Figure 2-14b. Micrographs revealing the precipitate phases were achieved with an AG-21 etchant for 15 seconds. The shape and distribution of the intragranular γ' particles exhibit a bimodal size distribution (Figure 2-14c and 2-14d), where the larger particles, secondary γ' (γ'_s), appear cubical while the smaller ones, tertiary γ' (γ'_t) are spherical (Figure 2-14d). Sizes and volume fractions of the γ'_s are 144 nm and 26.7 %, and the γ'_t are 9.4 nm and 7.5 %; respectively. The classification between secondary and tertiary γ' particles was determined using a threshold size of 50 nm. The precipitate sizes are measured using Sigma Scan image analysis software and are reported as equivalent particle diameter. Their distributions are also shown in Figure 2-15.

Figure 2-14: (a) Secondary electron micrographs (SEM) of the as received IN100 microstructure, etched for 3 minutes in Waterless Kallings etchant, showing γ'_p and carbides located along the grain boundaries and γ'_s in the matrix. SEM of as received IN100, swabbed with AG-21 etchant for 15 sec, showing (b) γ'_p located along the grain boundaries and (c) γ'_s in the matrix. (d) Transmission electron micrograph (TEM) of the as received IN100 showing details of the γ'_t .

Figure 2-15: The (a) secondary γ' and (b) tertiary γ' particle distributions in the as received IN100 material.

The ME3 material used in this study was provided by GE Aviation and Pratt & Whitney [42]. Each provided material which was atomized, compacted and extruded

into a billet form. These billets were then cut and isothermally forged at 1052°C at a strain rate of 0.010 s⁻¹. The final forgings were flat pancakes with a thickness of 1.85 in and diameter of 32.39 in. The forged disks were subjected to a subsolvus heat treatment at 1127°C for 2 hours followed by oil quenching with a 45 second transfer time from furnace to oil. The forged disks were then subjected to conventional stabilization and aging cycles. Following this, specimen blanks were extracted from the pancakes. Two different size blanks (for URI's low cycle fatigue and compact tension specimens) were extracted (as shown in Figure 2-16), a block 1.45 in x 1.45 in x 0.675 in and a rod with a length of 4.25 in and diameter of 0.675 in. In addition, rods with a length of 2.5 in and diameter of 0.6 in were extracted for Ohio State's creep testing. These blanks were then heat treated by GE Aviation, using a standard three stage heat treatment including solutioning, stabilization and aging. Heat treatment of the rod type blanks were carried out in a vacuum furnace in a fixture, as shown in Figure 2-17, carrying 10 specimens at a time, where 2 were dummy specimens containing embedded thermocouples for monitoring the cooling rate. Similarly, the heat treatment of the block type blanks were carried out with dummy blanks for temperature monitoring and were set directly in the furnace on a baffle plate. Controlled cooling rates were carried out using an inert gas backfill and a delayed activation of a fan system for gas circulation. The two forgings provided material for two microstructure conditions; M1, when heat treated results in planar grain boundaries and M2, which has serrated grain boundaries, where the two heat treatment cycles for these microstructures were varied. The M1 supersolvus solutioning stage consisted of heating the samples to 1171°C, holding for 1 hour, followed by a direct

quench cooling with a rate of 111°C/min to room temperature. The M2 supersolvus solutioning stage consisted of heating the samples to 1171°C, holding for 1 hour, followed by cooling at a rate of 2.08°C/min to 1121°C, holding for 15 min, then cooling at 111°C/min to room temperature. Following the solutioning cycle, both M1 and M2 microstructures were subjected to stabilization at 843°C for 4 hours followed by air cooling and aging at 760°C for 8 hours followed by air cooling. Subsequently, the heat treatment blanks were machined (into low cycle fatigue and compact tension specimens specified by URI) and provided to URI. This material is referred to as the “as received material”; M1-ME3 and M2-ME3.

Figure 2-16: Schematic of pancake with specimen cutup plan, where the specimens shown are stacked two layers high and dimensions are in inches. The blanks are for the specimens as follows; 2.1 x 2.1 x 0.675 for compact tension, 4.250 x 0.675 diameter for low cycle fatigue and 2.5 x 0.6 diameter for creep [42].

Figure 2-17: Schematic of vacuum fixture for heat treatment of rod type specimen blanks, with dimensions shown in inches [42].

The as received M1-ME3 material has an average Rockwell C hardness of 46.4 HRC. Its microstructure (Figure 2-18a), etched with Waterless Kallings solution, reveals an equiaxed grain structure with planar grain boundary morphology and an average grain size of 44 μm (ASTM 6). The shape and distribution of the γ' exhibit a bimodal size distribution (Figure 2-18b) where the larger particles, secondary γ' (γ'_s), appear cubical while the smaller ones, tertiary γ' (γ'_t), seem to be spherical (Figure 2-18c). Sizes and volume fractions of the γ'_s are 230 nm and 41%, respectively, while for γ'_t , the measurements are 42 nm and 10.4%, respectively, where the precipitate sizes are reported as the equivalent particle diameter. Their distributions are also shown in Figure 2-19.

The as received M2-ME3 material has an average Rockwell C hardness of 43.8 HRC. The microstructure, shown in Figure 2-20a, reveals serrated equiaxed grains. The shape and distribution of the γ' exhibit a trimodal size distribution (Figure 2-20b).

The secondary γ' (γ'_s), appear to have a dual distribution, where the larger ones have a size and volume fraction of 634 nm and 22.6%, and the smaller ones have a size and volume fraction of 148 nm and 9.1%, respectively. The tertiary γ' (γ'_t), seem to be spherical, with a size and volume fraction of 36 nm and 7.3%, respectively. The size and volume fraction of precipitates in M2 are measured by Ohio State University (M. Mills Research Group).

Figure 2-18: (a) Optical micrograph of the as received M1-ME3 material, etched with Waterless Kallings etchant for 15 sec, showing the typical grain size and planar grain boundary morphology. Secondary electron image of the as received M1-ME3 material, etched with AG-21 for 15 sec, showing (b) γ'_s size and distribution and (c) γ'_t particles within in the matrix.

Figure 2-19: The (a) secondary γ' and (b) tertiary γ' particle distributions in the as received M1-ME3 material.

Figure 2-20: (a) Optical micrograph of the as received M2-ME3 material, etched with Waterless Kallings etchant for 15 sec, showing the typical grain size and serrated grain boundary morphology. (b) Secondary electron image of the as received M2-ME3 material, etched with AG-21 for 15 sec. (c) SEM image using backscattered electron detection showing details of the γ' precipitate size and distributions. Note: figure (c) is an image obtained by Ohio State University (M. Mills Research Group).

Details of the grain boundary serrations in the M2-ME3 material are shown in Figure 2-21. The serrations follow a sinusoidal wave shape which can be characterized by an

amplitude and wavelength, which are measured as 2.3 microns and 15.5 microns; respectively.

Figure 2-21: Details of the serrated grain boundary in the as received M2-ME3 material.

Grain Size Measurements: The grain size was determined by hand, using the mean intercept method, for four randomly selected positions in the material and averaged [41]. This method is done by placing a grid over the micrograph of known magnification, where the grid spacing is along the order of the grain size of the sample. The mean intercept length is defined by:

$$L = \frac{L_T}{N_I M_g} \quad (2-16)$$

where N_I is the total number of intersections of grain boundaries with each vertical and horizontal line, M_g is the magnification and L_T is the total line length and is defined by:

$$L_T = N_V L_V + N_H L_H \quad (2-17)$$

where N_V is the number of vertical lines, L_V is the vertical line length, N_H is the number of horizontal lines and L_H is the horizontal line length. To convert the line length, L , to ASTM grain size number, G , we use the following calculation [43]:

$$G = -6.644(\log_{10} L) - 3.288 \quad (2-18)$$

where L in this equation is in units of mm.

Particle Size Measurements: The particle size is reported as an equivalent particle diameter and is measured using Sigma Scan image analysis software. In this, the SEM image is converted to a binary image (black and white) and an overlay is created over the γ' phase, as shown in Figure 2-22.

Figure 2-22: (a) SEM image of IN100 material showing γ'_p and γ'_s precipitates and (b) a red overlay on the γ'_p precipitates.

The area, A , of each particle is then calculated using the computer software.

The equivalent particle diameter, d , is calculated using the following formula:

$$d = \sqrt{\frac{4A}{\pi}} \quad (2-19)$$

A minimum of 350 particles are measured for a given microstructure and the average particle diameter is reported.

The area fraction (A_f) or volume fraction (V_f) of a given phase is calculated using the following formula:

$$V_f = A_f = \frac{\sum_{i=1}^{N_p} A_i}{A_{Total}} \quad (2-20)$$

where N_p is the total number of particles in the micrograph, A_i is the area of particle i and A_{Total} is the total area of the micrograph. A minimum of 4 micrographs are measured for a given microstructure and the average volume fraction is reported.

In the case of a multi-modal particle distribution, various magnifications are used in which the γ'_p , γ'_s and γ'_t precipitates are revealed as a size which can be measured. It should also be noted that when the smaller precipitates (γ'_s and/or γ'_t) volume fractions are measured, they must be adjusted in order to account for the volume fraction of the larger precipitate phase (γ'_p). The total volume fraction, 1, is the summation of contributions of γ , γ'_p , γ'_s and γ'_t , expressed as:

$$1 = (V_f)_\gamma + (V_f)_{\gamma'_p} + (V_f)_{\gamma'_s} + (V_f)_{\gamma'_t} \quad (2-21)$$

The volume fraction measured by equation (2-20) considers that the total area is equivalent to a volume fraction of 1. Thus, for example, in a tri-modal microstructure, the γ'_p size and volume fraction are calculated using equations (2-19) and (2-20) and the γ'_s and γ'_t volume fractions are calculated as:

$$(V_f)_{\gamma'_s} = (A_f)_{\gamma'_s} = \frac{\sum_{i=1}^{N_p} A_i}{A_{Total}} \left(1 - (V_f)_{\gamma'_p} \right) \quad (2-22)$$

$$(V_f)_{\gamma'_t} = (A_f)_{\gamma'_t} = \frac{\sum_{i=1}^{N_p} A_i}{A_{Total}} \left(1 - (V_f)_{\gamma'_p} - (V_f)_{\gamma'_s} \right) \quad (2-23)$$

The micrograph used to measure the γ'_s does not reveal γ'_p particles. Similarly, the micrograph used to measure the γ'_t does not reveal γ'_s and γ'_p particles.

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CHAPTER 3

DWELL FATIGUE CRACK GROWTH – EXPERIMENTS AND RESULTS

3.1 Introduction

This chapter presents the results of the dwell fatigue crack growth experiments and the correlation between crack growth rate and loading frequency. In this, the relation between frequency and fracture mode is examined. The time-dependent crack growth rate is then discussed and used to calculate the apparent activation energy for the intergranular crack growth process. Analysis of this energy in both air and vacuum showed that intergranular cracking is governed by a mechanism involving grain boundary sliding (in air and vacuum) modified by the diffusion of oxygen (in air). A condition for this mechanism to occur, is that a critical minimum distance exists between slip bands impinging the affected grain boundary. This condition is examined by correlating the slip band spacing and loading frequency using a model based on minimum strain energy accumulation within slip bands and that a unique configuration of number and spacing of bands exists for a given plastic strain. The model outcome, supported by experimental measurements at both high and low loading frequencies, show that a critical spacing exists, coinciding with the transitional loading frequency.

3.2 Experimental Setup

A total of 33 Dwell-fatigue crack growth tests were performed on compact tension (CT) specimens using MTS servohydraulic material testing systems in air and vacuum environments with the crack growth measurements being monitored using the potential drop technique. The set of experiments carried out are summarized in Table 3-1 on the M1-ME3 microstructure and in Table 3-2 for the M2-ME3 microstructure.

Table 3-1: Crack growth experiments carried out at URI for M1-ME3 microstructure (each X represents a test).

Loading Frequency	650°C	704°C	760°C	800°C	650°C	704°C	760°C
s-s-s	Air	Air	Air	Air	Vac	Vac	Vac
0.5-0.5		X					
1.5-1.5	X	X	X				
1.5-30-1.5		X	X				
1.5-100-1.5		XX	X				
1.5-300-1.5	X	X	XX	X		X	X
0.5-600-0.5		X					
1.5-600-1.5	X		XX		X		
1.5-3000-1.5			X				
1.5-7200-1.5		X	X				
1.5-hold		X	X				

Table 3-2: Crack growth experiments carried out at URI for M2-ME3 microstructure (each X represents a test).

Loading Frequency	650°C	704°C	760°C	704°C
s-s-s	Air	Air	Air	Vac
1.5-1.5			X	
1.5-300-1.5	X	X	X	X
1.5-3000-1.5		X	X	

Pre-cracking was first performed at room temperature between 5-10 Hz. The loading cycle during the test consists of 1.5 s loading, 1.5 s unloading and a dwell time of 0 s, 30 s, 100 s, 300 s, 600 s and 7200 s superimposed at the maximum load level with an initial starting ΔK in the range of 28-32 MPa $\sqrt{\text{m}}$. All tests were performed at a stress ratio of 0.1. All specimens were polished to a 1 micron finish prior to testing and in some occasions were scribed on the surface in order to measure grain boundary sliding.

The air tests were performed on CT20 specimens with an initial crack length of 9.04 mm, thickness of 9.88 mm, and width of 39.98 mm (see Appendix A for specimen drawing). Testing in air environments is carried out at high temperature in a resistance heating furnace at 650°C, 704°C, 760°C and 800°C. The high temperature tests were made possible by heating with a two zone ATS furnace, maintaining a temperature of $\pm 1^\circ\text{C}$ throughout the entire test. Temperature is monitored in 5 locations with a $\pm 3^\circ\text{C}$ temperature gradient using Type K thermocouples. The specimens were heated at an average rate of $4^\circ\text{C}/\text{min}$ and once the desired test temperature was reached, the specimen was held at constant temperature for 1 hour

before starting the test. The experimental test setup for air experiments is shown in Figure 3-1a.

The vacuum tests were performed on CT12.5 specimens with an initial crack length of 5.08 mm, thickness of 6.35 mm, and width of 25.40 mm (see Appendix A for specimen drawing). Testing in vacuum environments is carried out at high temperature using an induction heating coil at 704°C and 760°C, maintaining $\pm 1^\circ\text{C}$. The temperature is monitored at 3 locations with a $\pm 10^\circ\text{C}$ temperature gradient using Type K thermocouples. The vacuum pressure is monitored using an ionization gage and is maintained in the range of 10^{-6} - 10^{-7} torr during each test. The experimental test setup for vacuum experiments is shown in Figure 3-1b.

Figure 3-1a

Figure 3-1b

Figure 3-1: Crack growth experimental test setup in an (a) air and (b) vacuum environment in the Mechanics of Materials Research Laboratory at the University of Rhode Island.

Crack growth measurements were monitored using the direct current potential drop technique (see Appendix B for details). The data acquisition during each test is set to collect 20 points during the unloading and loading parts of the cycle and 1 point per second during the hold time. The test results are discussed in the next section.

3.3 Crack Growth Results

3.3.1 a versus N

Typical results for crack length as a function of cycles for 650, 704 and 760°C are shown in Figure 3-2. In all cases, the slope of the a versus N curve increases with N , which is characteristic of the crack growth rate, da/dN . Appendix B details the procedure and programs used to convert the voltage measurements (from the potential drop method) to crack length, as well as the programs to clean the data.

Figure 3-2: Typical crack length, a , versus number of cycles, N for (a) 650°C, (b) 704°C and (c) 760°C.

3.3.2 Crack Growth Rate

Crack growth results of the tests described above are plotted in terms of da/dN versus ΔK and are shown in Figure 3-3 and 3-4 for both M1-ME3 and M2-ME3 microstructures; respectively. These figures show that for all tests on ME3, a steady state crack growth rate was observed in the ΔK range of 31 to 80 MPa $\sqrt{m}$. In addition, the slopes of the steady state parts of the crack growth curves, which indicate the dependency of the crack growth process on ΔK , seems to increase with the increase in temperature. These figures show that the crack growth rate increases with both temperature and hold time duration for both M1-ME3 and M2-ME3 microstructures. In order to view the influence of loading frequency, the graphs are separated by temperature, as shown in Figure 3-5. This figure emphasizes that, for the same temperature, the crack growth rate increases with hold time duration. In addition, in the presence of air the crack growth rate increases when compared with that in vacuum.

Figure 3-3: Crack growth rate in terms of da/dN versus ΔK for M1-ME3 microstructure at 650°C, 704°C, 760°C, and 800°C in both air and vacuum environments at different loading frequencies.

Figure 3-4: Crack growth rate in terms of da/dN versus ΔK for M2-ME3 microstructure at 650°C, 704°C, and 760°C in both air and vacuum environments at different loading frequencies.

Figure 3-5a

Figure 3-5b

Figure 3-5c

Figure 3-5: Crack growth rate in terms of da/dN versus ΔK at (a) 650°C, (b) 704°C and (c) 760°C in air and vacuum environments at different loading frequencies.

3.3.3 Crack Opening Displacement

The crack opening displacement (COD) is measured using a clip gage, as shown in Figure 3-1a (see Appendix B for details). Typical results for COD as a function of cycles for 650, 704 and 760°C are shown in Figure 3-6. Similar to the a versus N curves, the slope of the COD versus N curve increases with N . It is also important to notice that the COD curves, for all temperatures and loading frequencies, are continuous in nature with no jumps due to load reversals. In addition, plotting the COD versus load, as shown in Figure 3-7, shows that there is no evidence of crack closure. During the loading cycle, the COD increases linearly with force, and then increases steadily during the hold at constant force, followed by a linear decrease during the unloading. Upon the next loading segment, this trend repeats, with the COD

increasing faster as N increases, as indicated in Figure 3-7 by the lower slope of the loading-unloading portion at larger values COD. This trend is also shown in Figure 3-6, where the rate or slope of the COD versus N curve increases with N.

Figure 3-6: Typical COD versus number of cycles at maximum force for (a) 650°C (1.5s-1.5s), (b) 704°C (1.5s-7200s-1.5s) and (c) 760°C (1.5s-600s-1.5s) for M1-ME3 microstructure in an air environment. Typical COD versus number of cycles at maximum force for (d) 650°C (1.5s-300s-1.5s), (e) 704°C (1.5s-300s-1.5s) and (f) 760°C (1.5s-1.5s) for M2-ME3 microstructure in an air environment.

Figure 3-7: Typical load versus COD curves for (a) 650°C (1.5s-1.5s), (b) 704°C (1.5s-300s-1.5s) and (c) 760°C (1.5s-600s-1.5s) for M1-ME3 microstructure in an air environment. Typical load versus COD curves for (d) 650°C (1.5s-300s-1.5s), (e) 704°C (1.5s-300s-1.5s) and (f) 760°C (1.5s-1.5s) for M2-ME3 microstructure in an air environment.

3.3.4 Correlation of Crack Growth Rate and Loading Frequency

In order to examine the time dependency of the crack growth curves, the relationship between the crack growth rate and loading frequency is plotted, as shown in Figure 3-8.

Figure 3-8: Crack growth rate, da/dN , versus frequency, f , for various values of ΔK for M1-ME3 microstructure at (a) 650°C, (b) 704°C and (c) 760°C in air environments.

This figure shows that the transition between transgranular and intergranular fracture modes is defined by a transitional frequency, f_t , which is found to be approximately 0.01 Hz for the three test temperatures. These results are categorized into three regions which with the aid of Figure 3-9 and 3-10 showing typical fracture surfaces for various loading frequencies at 650, 704 and 760°C are described as:

- i. Region I (frequencies $< f_t$): a low frequency region, associated with an intergranular fracture mode. The fracture surfaces corresponding to this region are shown in Figure 3-9 (b, c, f, i). In addition, intergranular fracture has been observed in a vacuum environment at low frequency, as shown in Figure 3-10.
- ii. Region II (frequencies $\sim f_t$): an intermediate frequency region near the transitional frequency, associated with a mixed transgranular/intergranular fracture mode. The fracture surfaces corresponding to this region are shown in Figure 3-9 (e, h).
- iii. Region III (frequencies $> f_t$): a high frequency region, associated with a transgranular fracture mode. These loading conditions are cycle-dependent in nature, which is indicated by the lack of dependency between da/dN and f . For high frequency loading, the da/dN is a function of the number of cycles but not the frequency. The fracture surfaces corresponding to this region are shown in Figure 3-9 (a, d, g). These surfaces exhibit typical cleavage fractures and evidence of fatigue striations.

Figure 3-9: Typical fracture surfaces for various loading frequencies for M1-ME3 microstructure in an air environment at 650°C, 704°C and 760°C.

Figure 3-10: Intergranular fracture surface for M1-ME3 microstructure in a vacuum environment at 760°C with a loading frequency of 1.5s-300s-1.5s.

3.3.5 Time Dependent Crack Growth Rate

The da/dN versus ΔK can be plotted in terms of da/dt versus K_{max} for the time-dependent loading frequencies (i.e. frequencies less than the transitional frequency). These plots are shown in Figure 3-11 and 3-12.

The general trends of the curves in Figure 3-11 and 3-12 show that the crack growth speed is temperature dependent, but independent of the hold time duration. The general trend of each temperature curve can be represented by a power law:

$$\frac{da}{dt} = BK_{max}^m \quad (3-1)$$

where B and m are determined through curve fitting and represent material and loading condition constants. The values for these constants for the various temperatures are shown in Table 3-3. This table shows that for M1-ME3 in an air environment, m decreases with the increase in temperature from 650°C to 704°C and increases with an increase in temperature to 760°C. M2 in an air environment shows that m increases with temperature. Additionally, in a vacuum environment, the exponent m is similar for M1-ME3 at 704°C and 760°C and lower for M2-ME3. The lower value m implies a weak dependency on the driving force, K_{max} , and indicates a time dependent crack growth mechanism involving creep effects. On the other hand, the higher values of m are related to conditions of higher dependency on K_{max} involving mechanisms related to environment diffusion and degradation.

Figure 3-11: Crack growth rate in terms of da/dt versus K_{max} for M1-ME3 microstructure at (a) 650°C, 704°C and (b) 760°C in air and vacuum environments for different loading frequencies.

Figure 3-12: Crack growth rate in terms of da/dt versus K_{max} for M2-ME3 microstructure at (a) 650°C, 704°C and (b) 760°C in air and vacuum environments for different loading frequencies.

Table 3-3: Paris law constants, B and m , for experimental da/dt versus K_{max} , for M1-ME3 and M2-ME3 microstructures.

Microstructure	T	Environment	B	m
M1/M2	°C	Air/Vac	m/sec	
M1	650	Air	6.94E-16	4.02
M1	704	Air	7.25E-15	3.72
M1	760	Air	8.895E-18	5.653
M1	704	Vac	2.87E-16	4.14
M1	760	Vac	8.96E-16	4.08
M2	650	Air	5.12E-15	3.6
M2	704	Air	6.16E-17	5.07
M2	760	Air	3.95E-19	6.69
M2	704	Vac	3.37E-15	3.37

It should be mentioned, that in Figures 3-11b and 3-12b, the crack speed at 760°C does not collapse to a single line, but is represented by a region (marked by dashed lines). The rationale of this behavior may be due to the microstructure instability at 760°C which is the aging temperature of the ME3 alloy. It has been experimentally observed that there is evidence of dissolution of the tertiary γ' particles in the immediate crack tip region, see Figure 3-13. The solid line in Figures 3-11b and 3-12b represent an average trend, which will be used in order to interpret the data in relation to the other temperature conditions.

Figure 3-13: Micrograph of M1-ME3 microstructure post testing at 760°C in an air environment revealing dissolution of the tertiary γ' precipitates near the crack tip, where the average size is larger than that of the as received M1-ME3 material as shown by the γ' particle distributions.

3.4 Cracking Kinetics and Operating Mechanisms

3.4.1 Apparent Activation Energy and Operating Mechanisms

An important factor in identifying the cracking mechanisms in time-dependent fracture processes is the knowledge of the related apparent activation energy, Q . This parameter can be determined from the results of the crack growth rates (Figure 3-11). The validity of this approach is based on the observations that crack growth rates da/dt are a function of the test temperature, i.e. are thermally activated. As such, these da/dt curves can be explained by the Arrhenius form:

$$\frac{da}{dt} = A \exp\left(-\frac{Q}{R} \frac{1000}{T}\right) \quad (3-2)$$

where A is the frequency factor, Q is the apparent activation energy and is a function of $K_{\max}$, R is the universal gas constant and T is temperature in Kelvin. Thus, for each of the different temperatures, the crack growth rate is calculated for different $K_{\max}$ and plotted against $1000/T$, as shown in Figure 3-14. These experimental points are fit to equation (3-2), where Q is obtained as a discrete value for different $K_{\max}$ values. These calculated values of apparent activation energy in both air and vacuum environment are plotted versus $K_{\max}$ in Figure 3-15.

Figure 3-14: Crack growth rate, da/dt , versus the inverse of absolute temperature for different $K_{\max}$ values for M1-ME3 microstructure in (a) air and (b) vacuum environments. Note that no crack growth tests were performed in vacuum at 650°C.

Figure 3-15: Apparent activation energy as a function of K_{max} for M1-ME3 microstructure in an air and vacuum environment.

In these figures, the Q versus K_{max} relationship can be fit into a polynomial equation of the form:

$$Q = Q_0 + Q_a (K_{max}) + Q_b (K_{max})^2 \quad (3-3)$$

where the constants Q_0 , Q_a , and Q_b are given in Table 3-4.

Table 3-4: The constants for Q (kJ/mol) given in equation (3-3) for vacuum and air.

Constant	Q_{Air}	Q_{Vac}
Q_0	224.2	144.1
Q_a	-1.6	-0.2
Q_b	7.39E-03	0

The results show that for K_{max} between 30 and 80 MPa√m, Q in vacuum is lower than that in air, with air values ranging from 140-180 kJ/mol, while for vacuum it is in the range of 130-140 kJ/mol. The variations in apparent activation energy have two extreme cases for failure; thermal and athermal failure [1]. The former mode of failure occurs when the thermal energy, in the absence of stress, is sufficient to cause failure. The stress free energy in this case can be calculated by setting K_{max} to zero in the

previous equation, thus yielding $Q = 144$ kJ/mol for an air environment and $Q = 224$ kJ/mol for a vacuum environment. On the other hand, athermal failure occurs when the stress, in the absence of thermal activation, is sufficient to cause failure. This failure mode could occur as a single or a multi mechanism process. An approach to identify the nature of this process is by examining the value of Q corresponding to the asymptote $dQ/dK_{\max} = 0$. For a single athermal process, the $K_{\max}$ approaches infinity or a threshold value ($K_{\max}^{\text{ath}}$) at which $Q(K_{\max}^{\text{ath}}) = dQ/dK_{\max} = 0$. On the other hand, a multiple damage process would correspond to a condition where at $dQ/dK_{\max} = 0$, the value of $Q(K_{\max}^{\text{ath}})$ approaches a finite value [2]. The athermal threshold stress, $K_{\max}^{\text{ath}}$, is determined by equating the derivative of equation (3-3) with respect to $K_{\max}$, to zero and solving for $K_{\max}$, i.e.:

$$K_{\max}^{\text{ath}} = \frac{\frac{dQ}{dK_{\max}} - Q_a}{2Q_b} = -\frac{Q_a}{2Q_b} \quad (3-4)$$

which yields a value of 108 MPa $\sqrt{\text{m}}$ in air at which the activation energy is a finite value of 138 kJ/mol, indicating the presence of multiple damage processes. In vacuum, since Q is idealized as a linear function of $K_{\max}$, $K_{\max}^{\text{ath}}$ is taken to be equal to that at $Q = 0$ which in turn identifies a single fracture process operating at the crack tip. The nature of the operating damage process is determined by comparing the value of the apparent activation energy to those identified in relation known mechanisms by Starink and Reed [3] as shown in Table 3-5.

Table 3-5: Activation energies for various processes contributing to high temperature fatigue crack growth [3].

Process	Activation Energy
<i>Single process</i>	
Oxidation of carbides	~250 kJ/mol
Dynamic embrittlement	~250 kJ/mol
Grain boundary creep	~150 kJ/mol
<i>Mixed process</i>	
Dynamic embrittlement on grain boundaries and creep of ligaments	~250 kJ/mol
Grain boundary creep failure and oxidation dominated fatigue crack growth	~150 to 250 kJ/mol
Grain boundary creep failure and non thermally activated failure	~0 to 150 kJ/mol

Comparing the current results of Q for ME3 with those listed in Table 3-5 shows that the activation energy in vacuum in the range of 130-140 kJ/mol, corresponds to a single damage mechanism involving grain boundary creep (sliding). On the other hand, the values of Q in air in the range of 140-180 kJ/mol, fall in categories identified in the above table as combined damage mechanisms involving grain boundary creep enhanced by oxidation processes. Since the current alloy does not include Fe as an alloying element, supported by the fact that the a versus N curves and COD versus N curves are continuous relationships, one can deduce that the oxidation mechanism is mainly attributed to dynamic embrittlement associated with oxygen diffusion. Furthermore, creep damage could be attributed to cavitation and/or grain boundary sliding. Examination of fracture surfaces have shown no evidence of cavities in this alloy, thus, the sliding mechanism is considered to be dominant.

3.4.2 Experimental Observations of Grain Boundary Sliding and Characteristic Sliding Length

Measurements of Grain Boundary Sliding: Experimental evidence of grain boundary sliding has been observed and reported on by many authors, such as in references [4-6], using scribing or grid deformation methods. The grain boundary sliding in ME3 specimens has been observed using a pre-test scribing method in which the specimens large surfaces have been scribed with lines using 600 grit paper before the start of the crack growth experiment as shown in Figure 3-16. Surface measurements of grain boundary sliding post-testing is taken as the amount of shift in the scribe line in the direction parallel to the grain boundary cracking path, see Figure 3-17. This average measurement is calculated in the range of 1 to 6 microns. Typical micrographs showing the shift in scribe lines, i.e. grain boundary sliding, are shown in Figure 3-18 for tests in both air and vacuum environments.

Figure 3-16: Pre-test line scribing to observe grain boundary sliding during intergranular crack growth testing.

Figure 3-17: Resolution of the displacement shift in the scribe line into normal (U_n) and parallel (U_s) components relative to the grain boundary, where the parallel component is considered as grain boundary sliding displacement.

Figure 3-18: Post-test grain boundary sliding observations in (a) air (760°C, 1.5s-600s-1.5s) and (b) vacuum (760°C, 1.5s-300s-1.5s) environments.

Slip/Grain Boundary Interactions: The sliding rate, which will be discussed in Chapter 4, is a function of the critical sliding length, w_c which is the minimum length

required between two pinning points over which grain boundary sliding occurs. This length has been experimentally determined by analyzing the interactions between lattice dislocations within the slip band and the grain boundary. The macroscopic strain, during cyclic loading, is distributed either homogenously or heterogeneously along a collection of slip bands [7, 8]. The plastic deformation is concentrated locally along these bands, which generally extends between two grain boundaries. The optimum number and spacing of slip bands is based on a minimum energy configuration which indicates the absence of long range stresses in the bulk [9]. A model of slip band spacing as a function of loading parameters is formulated on the basis of a criterion of minimum strain energy accumulation within slip bands and that a unique configuration of number and spacing of bands exists for a given plastic strain [8]. Based on previous work [10, 11], the slip band spacing, w , is formulated as a function of f , loading frequency as:

$$w^2 = \frac{(1-\nu)ah}{2M^2GB} \frac{(\Delta\sigma - 2\sigma_F)}{\varepsilon_p \exp(B'f)} \quad (3-4)$$

where ν is Poisson's ratio, a is the pile-up length, h is the width of a slip band, $\Delta\sigma$ is the remote tensile stress, σ_F is the frictional stress, M is the Taylor factor, G is shear modulus, ε_p is the applied tensile plastic strain, B and B' are scaling parameters. This relationship is detailed further in references [10, 11] and the resulting curve, shown in Figure 3-19, has been optimized by experimental measurements of slip band spacing for a high frequency loading (704°C/0.5-0.5), measured as 0.65 micron, (labeled I in Figure 3-19), and a low frequency loading (760°C/1.5-300-1.5), measured as 3.31 micron (labeled III in Figure 3-19).

Figure 3-19: Slip band spacing as a function of loading frequency at 760°C, with secondary electron micrographs of unetched specimen surfaces, post-testing, showing large and small slip band spacing corresponding to region I and III; respectively.

Figure 3-19 shows three regions which are similar to those described previously. The transition between transgranular and intergranular fracture modes in Figure 3-8 and 3-19 can be interpreted in terms of slip line density or spacing resulting in compatibility or incompatibility along the grain boundary [10]. The incompatibility results in a stress concentration, which is responsible for intergranular fracture. During high loading frequency, there is a small slip band spacing (high slip band density) resulting in compatibility along the grain boundary allowing slip transmission in which there is no stress build up along the grain boundary, therefore failure is by fatigue along the slip plane (transgranular fracture). On the other hand, during low loading frequency, where there is a large slip band spacing (low slip band density), an incompatibility along the grain boundary results in no transmission of dislocations across the grain boundary. As a result, a stress concentration is built up at the grain boundary causing

the lattice dislocation to dissociate into two extrinsic grain boundary dislocations [5, 12-15]; one parallel to the grain boundary plane and the other perpendicular to it. Failure then occurs when the glissile dislocations (mobile dislocations) present in the grain boundary glide [13], i.e. local grain boundary sliding, until reaching an obstruction, resulting in intergranular fracture. With fewer slip bands, the obstruction of sessile dislocations (immobile dislocations) is reduced and the available sliding distance (which is the slip band spacing) is higher.

Characteristic Sliding Length, w_c : In Figure 3-19, the critical slip band spacing, above which grain boundary sliding occurs (intergranular fracture), corresponds to the spacing measured at the transitional frequency, $w(f_t)$, taken as approximately 3 microns. This critical value of slip band spacing, w_c , represents a minimum length for mobility of the gliding grain boundary dislocations in order to create an incompatibility between the two relative grains resulting in grain boundary sliding. The limit of the grain boundary length over which sliding would occur could be as long as the grain size which would provide a crack growth pattern in a discrete step-wise form. However, it is observed from crack opening displacement measurements, that the crack growth is a continuous process. From crack opening displacement (COD) measurements, see Figure 3-20, it is, however observed that the crack growth is a continuous process. Figure 3-20 shows a magnified view corresponding to a ΔK of 40, 50 and 60 MPa $\sqrt{m}$, at which ~ 7.5 , ~ 1.5 and ~ 0.4 cycles, respectively, are required to crack a length equivalent to the grain size (44 microns), indicated by the two vertical lines. The COD during this duration is continuous, indicating that grain

boundary slip occurs over a length smaller than the grain size. This is also supported by the general observation that in low cycle fatigue (LCF) tests, in which surface cracks are propagating by creep mechanisms, the grain boundary openings are limited to lengths smaller than the grain size, Figure 3-21a. Similar observations, indicating that grain boundary sliding occurs along distances smaller than the grain size, are obtained from surfaces of post-test crack growth specimens as shown in Figure 3-21b.

Figure 3-20: COD measurements as a function of number of cycles for ME3 in air at 760°C for a loading frequency of 1.5s-600s-1.5s, indicating a continuous crack growth process, with an enlarged view at a point corresponding to a ΔK of 40 MPa $\sqrt{m}$, 50 MPa $\sqrt{m}$ and 60 MPa $\sqrt{m}$, where the number of cycles between the vertical red lines are ~ 7.5 , ~ 1.5 and ~ 0.4 cycles; respectively.

The significance of these efforts lies in the fact that in the grain boundary cohesive zone model developed in this research program, the parameter w_c represents a reference length equal to the size of the grain boundary element at the crack tip along

which the slip deformation and environmental damage interactions occur. The number of loading cycles required for the crack tip to advance this length would then provide the numerically simulated intergranular crack growth rate.

Figure 3-21: Secondary electron micrograph showing surface grain boundary openings (arrows) during (a) a LCF test (760°C, 1.5% strain range, $1 \times 10^{-4} \text{ sec}^{-1}$) and (b) a dwell-fatigue crack growth test at 760°C in a vacuum environment. Both images indicate that grain boundary sliding is limited to lengths between slip bands.

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CHAPTER 4

DWELL CRACK GROWTH MODEL AND INTERGRANULAR CRACKING MECHANISMS

4.1 Model Overview and Simulation Strategy (Model Components)

A mechanistic based crack growth model has been developed to simulate intergranular cracking in a polycrystalline Nickel-based superalloy at elevated temperatures. The model considers the interactions between creep, fatigue and environment. The basic model components are outlined in the flow chart shown in Figure 4-1. In this, the bulk material is modeled using a coarse crystal plasticity model surrounded by an internal state variable type of model. These two model components will be discussed in Chapter 5. The crack path is described using a cohesive zone approach in which the grain boundary dislocation network is smeared into a Newtonian viscous fluid element. The major grain boundary cracking mechanisms will be reviewed in Section 4.2. As discussed in Chapter 3, the active mechanisms in this work are grain boundary sliding and dynamic embrittlement. The cohesive laws consider deformation and damage through grain boundary sliding and dynamic embrittlement mechanisms as influenced by the grain boundary dislocation mobility and its evolution due to Oxygen diffusion. Each of the model components will be described in the following sections.

Figure 4-1: A flow chart showing basic model components.

4.2 Grain Boundary Deformation and Damage Mechanisms

In general, deformation and damage mechanisms at the crack tip are described by three processes, fatigue, creep and environment. The first (fatigue), is cycle dependent damage, while the two latter (creep and environment) describe time dependent damage processes. Creep-fatigue-environment interactions occur at temperatures and cyclic loading conditions at which thermally activated processes and time-dependent behavior occur [1]. At high frequencies and low temperatures, the

fracture mode is transgranular and the effects of time-dependent processes are eliminated. A general observation is that there is a transitional frequency, above which the fracture mode is transgranular and below which the fracture mode is intergranular. At low frequencies and high temperatures, the time-dependent processes more readily occur. Transition to higher frequencies eliminates the effects of such processes, such as creep and oxidation. Additional time-dependent processes at elevated temperature include dislocation climb and slip, environmental interactions, grain boundary sliding, strain-aging, stress relaxation, creep, cavity nucleation and growth, and microstructural instabilities. Furthermore, strain range has an influence on fatigue life; the shortest life is associated with large strain ranges [1]. The relative creep, fatigue and environment plastic zone sizes as a function loading frequency and hold time are shown in Figure 4-2. Fatigue damage arises from the accumulation of cyclic plastic strain, which is a measure of the degradation of the loaded material [3]. The creep damage is highly dependent on the hold time, as shown in Figure 4-2, as the hold time at maximum load increases, the creep plastic zone size increases. This damage process occurs as a result of grain boundary cavitation or grain boundary sliding. The environmental damage occurs from the diffusion of Oxygen at the crack tip and is a localized phenomena, thus, its plastic zone size is small in relation to the creep and fatigue plastic zones, as shown in Figure 4-2. This type of damage occurs in two ways, two stage oxidation or dynamic embrittlement. The various types of time dependent deformation and damage mechanisms are briefly described next.

Figure 4-2: Schematic of the relation between the creep, fatigue and environment plastic zone sizes as a function loading frequency (or hold time), adapted from reference [2].

4.2.1 Grain Boundary Creep Mechanisms

At high temperature grain boundaries have an important role in flow and fracture of polycrystalline metals, grain boundary phenomena include sliding, migration, diffusion creep and cavitation [4, 5]. One or more of these mechanisms can occur during time-dependent cracking. A phenomena that has a large influence on

cavities is grain boundary sliding. When grain boundary sliding is not accommodated, cracks or voids can form. The following sections will discuss cavitation nucleation, growth and coalescence and grain boundary sliding along with accommodating mechanisms. In order to study these phenomena, it is important to understand the flow and fracture characteristics of different grain size ranges. Langdon defines four grain size ranges when examining the behavior of polycrystalline materials as [5]:

1. Macroscopic grain size ($>1000\text{ }\mu\text{m}$) – (all single crystals, and large grained polycrystals) grain boundaries play no role, flow is controlled by intragranular dislocation slip.
2. Mesoscopic grain size ($10\text{-}1000\text{ }\mu\text{m}$) – grain boundaries make a minor contribution, i.e. grain boundary sliding, includes the formation of subgrains at high temperature, (subgrain size λ is smaller than grain size d) which leads to core/mantle behavior.
3. Microscopic grain size ($0.01\text{-}10\text{ }\mu\text{m}$) – grain boundaries play a dominant role, no subgrains are formed and strain is through mantle behavior.
4. Nanoscopic grain size ($\leq 0.01\text{ }\mu\text{m}$) – it is assumed that boundaries account for most of the deformation due to volume fraction of grain boundaries is $\geq 30\%$.

It is important to note that powder metallurgy Nickel-based superalloys are of the order of $10\text{-}50\text{ }\mu\text{m}$ grain size, thus the material would follow the behavior of the mesoscopic and microscopic grain size regions.

4.2.1.1 Cavitations

In the presence of cavities, decohesion occurs as a result of nucleation, growth and coalescence of cavities or voids along the grain boundary [4, 6, 7]. The time to fracture would represent the entire void history from nucleation to coalescence. Holes reduce the cross section of the material along the grain boundary, acting as a stress concentrator, which in turn causes the holes to grow. The cavities are commonly observed on the fracture surfaces.

High temperature void nucleation is mainly due to the condensation of vacancies, under a high tensile stress, moved by diffusion. Normally, the applied stress is too small to account for cavity nucleation, thus a high concentration of stress is required. Some nucleation sites are interface between inclusion and matrix, triple points, the intersection between a slip band and carbide, oxide or grain boundary [8-10]. In addition to a threshold stress, there is an incubation time during which vacancies cluster to form a cavity of a critical radius [11]. In summary, the factors required for void nucleation are a critical void size, a threshold stress, an incubation time and a nucleation site. A cavity nucleates on a boundary when a void reaches a critical radius, such that the surface tension force trying to close the cavity is balanced by the applied stress.

Following nucleation, the void will grow. For void growth to occur there must be vacancy sources, diffusion of vacancies to cavities, and the grains must be allowed to move apart. For a boundary to supply vacancies there must be a tensile stress applied and the temperature must be high enough to move vacancies. Void growth can

be controlled by three things, boundary diffusion, surface diffusion, or coupling of boundary diffusion and power-law creep. When voids are small they usually grow by diffusion and when they are large power-law creep becomes the dominant mechanism [6]. When the temperature is high enough to permit diffusion and the stress is low, power-law creep can be neglected and voids are assumed to grow by diffusion alone [12]. Growth laws are governed by void volume, surface area, spacing or grain boundary area. These laws are reviewed by many authors, as illustrated in the following references [6, 8, 12-16]. Fracture is assumed to occur when the cavities have reached a critical size or spacing.

4.2.1.2 Grain Boundary Sliding

The grain boundary sliding process is governed by the stress field present at the grain boundary crack tip, which controls the slip/grain boundary interactions. The high stress at the crack tip causes dissociation of lattice dislocations, present in a slip band, into an immobile sessile dislocation and a mobile glissile dislocation [17-24]. The motion of the mobile grain boundary dislocation induces grain boundary sliding. During high frequency loading, when there is a high probability of slip bands in neighboring grains to interact with each other, various scenarios can occur, as illustrated by Figure 4-3. Figure 4-3a shows transmission of a lattice dislocation, Figure 4-3b shows annihilation of the glissile dislocation, and Figure 4-3c shows compatible deformation in which the motion of the glissile dislocations are obstructed. Each of these cases does not result in grain boundary sliding. For this process to take

place, a minimum slip band spacing is required, thus, the motion of the glissile dislocations are not obstructed [19, 25].

Figure 4-3: Two grains exhibiting no grain boundary sliding for various cases depicting (a) transmission of the lattice dislocation, (b) annihilation of the glissile dislocation when the slip bands in the two grains meet at the grain boundary and (c) compatible deformation in which all the glissile dislocations are obstructed [22].

Grain boundary sliding occurs with an accommodating mechanism [26]. When it is not accommodated, sliding results in the growth of voids on the boundaries perpendicular to the stress axis or cracks can form at the triple junctions. An accommodating mechanism will take up incompatibilities that would otherwise appear at the grain surfaces and can fundamentally be divided into three different mechanisms including (1) elastic distortion of grains themselves, (2) diffusion of matter through the grain or around its boundaries, and (3) plastic flow within the grain. Figure 4-4 shows a polycrystalline material with an applied tensile stress and illustrates the relationship between flow problems involving grain boundary sliding.

Figure 4-4: Grain boundary sliding accommodated by elastic distortion (Grains 1), diffusion (Grain 2), and creep (Grain 3). No accommodation for grain boundary sliding results in void growth along the boundary (Grain 5) or cracks at the triple points (Grain 4) [27].

(1) Accommodation by elastic distortion

Sliding can be accommodated by elastic deformation of the surrounding grains. In this case, the deformation is recovered when the stress is removed, and the sliding displacement is small relative to the length of the sliding surface [6, 27, 28]. If sliding displacements are too large to be accommodated elastically, then accommodation is by diffusional or plastic flow [8].

(2) Accommodation by diffusion

Sliding can be accommodated by diffusion through the transport of material along the grain boundaries or through the lattices of grains as seen in Figure 4-5 [6, 8, 27, 28].

Figure 4-5: Grain boundary sliding accommodated by diffusion [8].

Grain boundary sliding may take place as a flow mechanism occurring independently of diffusion creep or as a natural consequence of diffusion creep. Rachinger sliding without diffusion creep may be accompanied by intragranular slip, localized slip adjacent to the boundaries, formation of triple point folds, or opening of wedge cracks at triple points. Rachinger sliding increases strain by attempting to increase the number of grains along the tensile axis. Lifshitz sliding is when the individual grains become elongated through Nabarro-Herring or Coble diffusion creep. In high temperature creep, total strain can be the sum of strain due to intragranular processes occurring within the grain, strain due to grain boundary sliding and any associated accommodation process and strain due to diffusion creep [5]. Langdon's analysis shows that rachinger grain boundary sliding in aluminum is given by:

$$\dot{\epsilon}_{gbs} = \frac{A_{gbs} D G b}{k T} \left(\frac{b}{d} \right)^{0.9} \left(\frac{\sigma}{G} \right)^{3.3} \quad (4-1)$$

where D is the coefficient for lattice self-diffusion, G is the shear modulus, b is the Burgers vector, k is Boltzmann's constant, d is the grain size, A is a dimensionless constant [5].

Wu and Koul have explored grain boundary sliding accommodated by diffusional flow with grain boundary precipitates through their work on IN738LC Ni-base superalloy. They modified Langdon's model by incorporating back stress opposing dislocation glide and climb. They have shown that dislocations moving along a planar boundary under an applied shear stress, τ , with uniformly distributed grain boundary precipitates, will pile up at the precipitates and exert a back stress on moving dislocations at a distance x from the precipitate as illustrated in Figure 4-6 [29].

Figure 4-6: Schemata of dislocation pileups at the edge of a grain boundary precipitate [29].

This back stress is given as:

$$\tau_b(x) = \frac{nGb}{2\pi(1-\nu)x} \quad (4-2)$$

where n is the number of pile up dislocations at a given time, G is the elastic shear modulus, ν is Poisson's ratio, b is the Burgers vector [29]. The average back stress, τ_{ig} , opposing dislocation glide over a distance λ is obtained by:

$$\tau_{ig} = \frac{1}{\lambda} \int_{nb}^{\lambda} \tau_b(x) dx = \frac{1}{\lambda} \int_{nb}^{\lambda} \frac{nGb}{2\pi(1-\nu)x} dx = \frac{nGb}{2\pi(1-\nu)\lambda} \ln \frac{\lambda}{nb} \quad (4-3)$$

The average normal component of back stress, σ_{ic} , opposing dislocation climb, reduces the effective stress, σ , and is expressed as:

$$\sigma_{ic} = \frac{\alpha Gb}{\lambda} \quad (4-4)$$

where α is a factor, less than 1, that is dependent upon the coherency of the precipitate particle. The shear component of back stress, τ_{ic} , opposing dislocation climb, is obtained through the relation:

$$\sigma_{ic} = \sqrt{3\tau_{ic}} \quad (4-5)$$

Xu and Koul assume dislocations are perfect sinks of vacancies and a dislocation climbs a burgers vector when it absorbs a vacancy. They also assume dislocation climb occurs along the noncoherent side of the grain boundary [29]. The minimum energy or work needed to transport a vacancy to a dislocation core on a grain boundary with precipitates is given as:

$$W_{\min} = \frac{(1+\nu)Gb^3}{3\pi(1-\nu)} (1 - \cos \theta_{\min}) = \frac{(1+\nu)Gb^3}{3\pi(1-\nu)} \left(1 - \frac{\lambda}{\sqrt{\lambda^2 + r^2}} \right) \quad (4-6)$$

where λ is the distance between grain boundary precipitates and r is the radius of the grain boundary precipitate, as seen in Figure 4-6 [29]. With the addition of the activation energy for diffusion along clean boundaries, U_d , the total activation energy,

U_c , for dislocation climb can be obtained. The diffusion coefficient can be expressed as a function of the total activation energy as:

$$D = b^2 \nu_0 \exp\left(-\frac{U_c}{kT}\right) = b^2 \nu_0 \exp\left(-\frac{U_d + W_{\min}}{kT}\right) \quad (4-7)$$

where ν_0 is the atomic frequency, k is Boltzmann's constant and T is absolute temperature. With this knowledge, Wu and Koul developed a rate equation for grain boundary sliding of the form:

$$\dot{\gamma}_{gbs} = \frac{A_1 D G b}{kT} \left(\frac{b}{d}\right)^q \left(\frac{\lambda + r}{b}\right)^{q-1} \frac{(\tau - \tau_{ig})(\tau - \tau_{ic})}{G^2} \quad (4-8)$$

where q is the grain size dependent index and takes a value of 1 for a grain boundary without grain boundary precipitates, 2 for a discrete boundary precipitate distribution, and 3 for a grain boundary with a continuous grain boundary precipitate network [29-31].

(3) Accommodation by plastic flow

Sliding can be accommodated by plastic flow involving slip, twinning or creep [6, 27, 28]. Crossman and Ashby explored this mechanism, grain boundary sliding accommodated by plastic flow within the grain. They modeled a polycrystalline material with a shear stress applied in the boundary plane resulting in Newtonian-viscous sliding with a sliding rate given by [26]:

$$\dot{U}_{GB} = \left(\frac{\delta}{\eta_B}\right) \tau \quad (4-9)$$

where δ is the grain boundary thickness, usually twice the atomic size, η_B is the viscosity of the grain boundary and τ is the applied shear stress. The intrinsic viscosity

of a flat boundary, assuming ledges or other irregularities on the boundary surface are not greater than the atomic height is given as:

$$\eta_B^i = \frac{kT}{8bD_B} \quad (4-10)$$

where k is Boltzmann's constant, T is the absolute temperature, b is the atom size and D_B is the grain boundary diffusion coefficient. The viscosity is increased if obstacles are present on the sliding path, such as ledges or particles. Ledges or terraces of height h or boundary curvature of amplitude h , increase the viscosity and can be expressed by:

$$\eta_B = \frac{kTh^2}{8b^3D_B} \quad (4-11)$$

A dispersion of particles with diameter a , and area fraction, V_f , of the particle in the boundary plane, increase the viscosity and is expressed by:

$$\eta_B = \frac{kTV_f a^2}{8b^3D_B} \quad (4-12)$$

Power-law creep is assumed to govern the interior grain deformation and the shear strain rate is given by:

$$\dot{\gamma} = A \left(\frac{\tau}{G} \right)^n \quad (4-13)$$

where τ is the applied shear stress, G is the shear modulus, n is a constant and A is a temperature-dependent constant given as:

$$A = C \frac{D_v G b}{kT} \quad (4-14)$$

where D_v is a lattice diffusion coefficient and b is the magnitude of the burger's vector [26]. Crossman and Ashby use a plane-strain finite element model to simulate the

creep of a polycrystal. At lower strain rates, sliding becomes easier, at very low strain rates, grain boundaries slide freely. When no sliding occurs, the grain shears uniformly and follows the flow law of the interior of a grain, and the average macroscopic shear rate is given as:

$$\bar{\dot{\gamma}}_A = A \left(\frac{\bar{\tau}_A}{G} \right)^n \quad (4-15)$$

For free sliding boundaries, the deformation follows the power law:

$$\bar{\dot{\gamma}}_A = A \left(f \frac{\bar{\tau}_A}{\mu} \right)^n \quad (4-16)$$

where f is a stress enhancement factor describing the acceleration of creep caused by freely sliding boundaries. When lattice diffusion controlled variant of diffusional flow or Nabarro-Herring creep is dominant, the rate is given by:

$$\dot{\gamma}_{NH} = 42 \frac{\Omega D_v \tau}{k T d^2} \quad (4-17)$$

where Ω is the atomic volume and d is the grain size [26]. These variations of the grain boundary sliding rate show that it is a rate-dependent phenomena.

4.2.2 Environmental Degradation Mechanisms

Environmental degradation has been experimentally observed in Nickel-based superalloys [32-36]. Various studies relate this degradation to the loss of ductility in the grain boundary, leading to decohesion of grain boundaries during high temperature loading in an air environment. Several studies have related the reduction of ductility to the decrease of chemical potential as a result of several mechanisms including the

formation of oxides (short range interactions) and dynamic embrittlement (gas phase embrittlement). Ghonem and Zheng [37] have discussed the former mechanism in alloy 718 which includes Iron in its composition and proposed a two-stage crack tip oxidation mechanism in which fracture occurs in a stepwise fashion. In this damage mechanism, the da/dt versus K_{max} curves show dependency on the hold time duration. In the concept of dynamic embrittlement, on the other hand, see the work of Krupp et al [38], an initial time is needed to establish the Oxygen concentration profile along the grain boundary path at the crack tip. It is assumed that in a pure environmental damage process, fracture occurs once the accumulated Oxygen at the crack tip is sufficient to result in grain boundary decohesion. This level of critical concentration Oxygen, C_{crit} , which is stress dependent, is reached accumulatively in the crack tip region. As such, the time required to attain C_{crit} at points ahead of the crack tip decreases as the crack length increases. This ensures that the crack advances almost continuously which is clearly demonstrated in the current work on ME3, which, as previously indicated, does not include Fe as a composition element. This absence of Iron, coupled with the activation energy analysis detailed in the previous chapter are taken as basis for the approach that the environmental damage mechanism in intergranular cracking of ME3 is driven by dynamic embrittlement of the crack tip region rather than by oxide formation. An important parameter in the dynamic embrittlement mechanism analysis is the concentration, C , of Oxygen at a distance x in front of the crack tip.

4.2.2.1 Two Stage Oxidation

During two stage oxidation, the Oxygen interacts with the surface forming a porous oxide layer, such as Iron Oxide (Figure 4-7). This reduces the partial pressure of Oxygen at the surface and a dense layer of Cr_2O_3 will form second. The time required from complete formation of Cr_2O_3 is the Oxygen passivation time, t_p , and no further penetration of Oxygen will take place [35]. At this time, a saturation in grain boundary ductility level is established.

Figure 4-7: Schematic of two stage oxidation mechanism showing the oxide layers which are built up as a function of time [35].

Grain boundary decohesion will occur when the strain produced by an external load becomes greater than the amplitude of ductility, i.e. the oxidation time $\geq t_p$ [39]. This ductility, ε_d , is expressed as a function of the oxidation parameter, β , which is the ratio between the weight of Cr_2O_3 and to the total weight of oxides, these functions are expressed as:

$$\varepsilon_d = a_1 \arctg(-a_2 \beta^2 + a_3) + a_4 \quad (4-18)$$

$$\beta = m_{\text{Cr}_2\text{O}_3} / m_{\text{ox}} \quad (4-19)$$

where a_1 , a_2 , a_3 and a_4 are material, temperature and loading condition dependent constants. The time required for formation of the Cr_2O_3 will result in a discrete or step wise cracking process. This is commonly observed in the crack length or crack

opening displacement as jumps or discrete steps in length as a function of number of cycles.

4.2.2.2 Dynamic Embrittlement

During dynamic embrittlement, the Oxygen atom will diffuse preferentially along the grain boundary in the crack tip region (Figure 4-8). Under an applied stress acting normal to a grain boundary, Oxygen atoms will have a tendency to enter the grain boundary as interstitial atoms. These atoms can reduce the cohesive strength of the grain boundary, i.e. embrittling the boundary [40].

Figure 4-8: Schematic of dynamic embrittlement mechanism showing the diffusion of Oxygen along the grain boundary resulting in grain boundary decohesion [40].

During dynamic embrittlement, crack growth will occur when the Oxygen concentration reaches a critical limit. This is viewed as a continuous process as the diffusion of Oxygen is a localized phenomenon. The governing equations can be derived from Fick's first and second laws, which are given as:

$$J = -D \frac{\partial C(x,t)}{\partial x} \quad (4-20)$$

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2} \quad (4-21)$$

where J is the particle flux, which describes the transfer of solute atoms per unit area, D is diffusivity, x is the distance from the surface, t is time and C is the concentration of the diffusing species. Using Boltzmann's approach, as detailed in reference [41], the concentration of the diffusing species, in this case Oxygen, can be derived. In this approach we let:

$$\lambda = x/\sqrt{t} \quad (4-22)$$

By substituting equation (4-22) into (4-21), Fick's second law can be rewritten as:

$$-\frac{\partial C}{\partial \lambda} \frac{\lambda}{2D} = \frac{\partial^2 C}{\partial \lambda^2} \quad (4-23)$$

Letting

$$\frac{\partial C}{\partial \lambda} = A e^{-a\lambda^n} \quad (4-24)$$

Equation (4-23) reduces to:

$$\lambda^{n-2} = an2D \quad (4-25)$$

which is satisfied for $n = 2$ and $a = 1/(4D)$, thus, equation (4-24) becomes:

$$\frac{\partial C}{\partial \lambda} = A e^{-\frac{\lambda^2}{4D}} \quad (4-26)$$

Letting

$$\xi = \frac{\lambda}{2\sqrt{D}} \quad (4-27)$$

and applying the boundary conditions for a solid of unit length as $C = C_s$ for $x = 0, 1$, and at $t = 0$, $C = C_0$ for $0 < x < 1$, equation (4-26) can be solved and the concentration of Oxygen as a function of time and position can be written as:

$$C = C_0 + (C_s - C_0) \left\{ 1 - \operatorname{erf} \left(\frac{x}{2\sqrt{tD}} \right) \right\} \quad (4-28)$$

where C_0 is the initial concentration of Oxygen in the bulk material and C_s is the concentration of Oxygen at the surface (i.e. the concentration of Oxygen in air). The diffusivity is written using an Arrhenius law as:

$$D = D_0 e^{-\frac{Q_{app}}{RT}} \quad (4-29)$$

where D_0 is the frequency factor, R is the universal gas constant, T is absolute temperature and Q_{app} is the experimental apparent activation energy for diffusion of Oxygen in the grain boundary, which is a stress dependent term. In a fully environmental controlled cracking process, Q_{app} is estimated as 250 kJ/mol [42], in ME3, however, where the crack tip fracture is controlled by multiple damage mechanisms, Q_{app} , as discussed earlier, is a function of K_{max} . D_0 is a frequency factor, the magnitude of which is not available for the ME3 alloy. Observed values of D_0 for Oxygen in Nickel vary from 10^{-5} to 10^4 m²/s [43]. These wide variations are related to the high solubility of Nickel for other elements and, as such, the influence of the large number of different alloying elements might be reflected in the extent of the variation of D_0 . In the absence of an exact value of D_0 for ME3, an attempt is made to determine an approximate limit using available crack growth experimental results. This is achieved by first utilizing an arbitrary value of D_0 to calculate the depth of Oxygen diffusion as a function of temperature and K_{max} . An example using $D_0 = 10^{-4}$

m^2/s , at the three test temperatures, for $K_{\max} = 50 \text{ MPa}\sqrt{\text{m}}$ is shown in Figure 4-9. A critical Oxygen concentration estimated as 12% [41], was used to determine the crack growth extension at different exposure times. The depth of penetration and corresponding time provide the crack growth rate due to pure environmental damage, as shown in Figure 4-10 as a function of $K_{\max}$. Figure 4-10, shows that the experimental crack growth rate coincides with the pure environmental simulation data generated with $D_0 = 10^{-3} \text{ m}^2/\text{s}$. This value, since the crack growth process in ME3 is a combined grain boundary sliding and environmental damage, would represent an upper limit in the cohesive zone simulation procedure. The fundamental role of Oxygen in a dynamic embrittlement process is to alter the grain boundary sliding in a manner that reduces the time required for the grain boundary slip process to reach its critical limit. This type of interaction will be detailed in Section 4.3.

Figure 4-9: Oxygen concentration profiles as a function of exposure time at a $K_{\max}$ of $50 \text{ MPa}\sqrt{\text{m}}$ at (a) 650°C , (b) 704°C and (c) 760°C .

Figure 4-10: Crack growth rate in terms of da/dt versus K_{max} for M1-ME3 microstructure at (a) 650°C and (b) 704°C in an air environment and those generated for fully environmental damage using various values of D_0 .

4.3 Grain Boundary Cohesive Zone Interface Model

The grain boundary interface is modeled using a cohesive zone approach in which the grain boundary dislocation network is smeared into a Newtonian fluid element. The interface is considered to be a node to node contact and is described by traction displacement laws. This law follows a viscoelastic relationship in which the tangential elastic opening displacement is the difference between the total tangential displacement and grain boundary sliding displacement. Each of these components along with the fracture criterion will be described in the following sections.

Traction-Displacement Laws

The viscoelastic traction-displacement law acting on the grain boundary phase can be described by the linear form:

$$T_i = k_i u_i^e \quad (4-30)$$

where i is the normal or tangential direction, T_i is traction, k_i is cohesive stiffness, and u_i^e is the elastic opening displacement. This displacement is the difference between the total and inelastic displacements as:

$$u_i^e = u_i^{tot} - u_i^{in} \quad (4-31)$$

The inelastic component in the normal direction is due to cavities and in the tangential direction is due to grain boundary sliding. Thus the traction-displacement laws can be written as:

$$T_n = k_n (u_n - u_c) \quad (4-32)$$

$$T_t = k_t (u_t - u_s) \quad (4-33)$$

where $T_{n,t}$ is traction, $k_{n,t}$ is cohesive stiffness, $u_{n,t}$ is the total opening displacement, u_c is the normal opening displacement due to cavities and u_s is the tangential opening displacement due to grain boundary sliding. In the current analysis, $u_c = 0$, due to the absence of cavity related deformation mechanisms.

Grain Boundary Sliding

The grain boundary dislocation network is smeared into a Newtonian viscous fluid element. If we consider the grain boundary to consist of two layers moving at different velocities, the viscosity arises from the shear stress between the layers. The viscosity describes the resistance of a material under an applied shear stress. A higher viscosity means a higher resistance to motion. The relationship between the shear

stress and the velocity gradient can be obtained by considering two planes closely spaced at a distance y , separated by a grain boundary material. This can be schematically represented by considering the lower plane to be fixed and the upper plane is moving under an applied shear stress, as shown in Figure 4-11.

Figure 4-11: Schematic of shear stress and velocity gradient between two boundaries.

The applied shear stress, τ , is proportional to the velocity and inversely proportional to the separation distance as:

$$\tau = \eta \frac{\partial u}{\partial y} = \frac{\eta}{\delta} \dot{u}_s \quad (4-34)$$

where η is the proportionality factor called viscosity, ∂u or $\dot{u}_s$ is the velocity of grain boundary sliding and ∂y or δ is the grain boundary thickness. Rearranging this equation yields the relative velocity of two sliding boundaries, written as a viscous flow law [26]:

$$\dot{u}_s = (\delta/\eta) \tau \quad (4-35)$$

Rearranging this equation and considering the shear stress equivalent to the tangential stress, T_t , the viscosity can be written as:

$$\eta = (\delta/\dot{u}_s)T_t \quad (4-36)$$

The viscosity can be derived following the approach of Kê [44], where the rate of relative displacements of the sliding surfaces can be described by the distance slipped along the grain boundary during the relaxation time, t_c . This approach yields the expression:

$$\eta = \frac{\delta E_{GB}}{w_c} t_c \quad (4-37)$$

where E_{GB} is the unrelaxed shear modulus and w_c is the characteristic length over which grain boundary sliding occurs or the length between pinning points. As stated by Cosandey, since the grain boundaries are composed of discrete dislocations, grain boundary sliding can be described by the climb process of these dislocations [45]. In this case, the relaxation time or characteristic time, t_c , is the time a dislocation can climb a distance λ and is written as:

$$t_c = \lambda/v_c \quad (4-38)$$

The climb velocity or dislocation velocity, v_c , is given by the Einstein mobility relation [45-47]:

$$v_c = M_d F = M_d T_t b \quad (4-39)$$

where M_d is the mobility of grain boundary dislocations and F is the force per unit length acting on the dislocation and b is the Burger's vector. The intrinsic mobility term is a function of the grain boundary diffusivity, D_{gb} , Boltzmann's constant, k , and temperature, T , and is expressed as:

$$M_d = \frac{D_{gb}}{kT} \quad (4-40)$$

The above analysis yields the relaxation time, viscosity and sliding rate in terms of dislocation mobility as:

$$t_c = \frac{\lambda}{M_d T_t b} \quad (4-41)$$

$$\eta = \frac{\delta E_{GB} \lambda}{w_c M_d T_t b} \quad (4-42)$$

$$\dot{u}_s = w_c \frac{M_d b}{E_{GB} \lambda} T_t^2 \quad (4-43)$$

By dividing equation (4-43) by the length w_c , the grain boundary sliding strain rate is obtained as:

$$\dot{\epsilon}_s = \frac{\dot{u}_s}{w_c} = \frac{M_d b}{E_{GB} \lambda} T_t^2 \quad (4-44)$$

This can be written in a generalized form, as a power law equation given as:

$$\dot{\epsilon}_s = \frac{M_d b E_{GB}}{\lambda} \left(\frac{|T_t|}{E_{GB}} \right)^p \quad (4-45)$$

It has also been suggested by many authors [46, 48, 49] that a threshold stress, T_{th} , exists, below which grain boundary sliding will not occur, thus equation (4-45) is modified to the form:

$$\dot{\epsilon}_s = \begin{cases} 0 & |T_t| \leq T_{th} \\ \frac{M_d b E_{GB}}{\lambda} \left(\frac{|T_t - T_{th}|}{E_{GB}} \right)^p & |T_t| > T_{th} \end{cases} \quad (4-46)$$

This strain rate is calculated for the grain boundary sliding process occurring in vacuum conditions. In order to consider the role of Oxygen diffusion on the embrittlement mechanism of the grain boundary cracking process in air, the intrinsic dislocation mobility is hindered by the solute atom, Oxygen, see Dahal et al [17, 18].

This mobility term, M_s , is related to the velocity of the solute atom and is assumed to move with the dislocation. Thus, the force exerted by a single dislocation, F_s , on a solute atom must satisfy the Einstein relationship for the velocity of a solute atom [46, 50, 51]:

$$v_c = M_s F_s = M_d F \quad (4-47)$$

where M_s is the solute mobility given as:

$$M_s = \frac{D_s}{kT} \quad (4-48)$$

M_d and F are the dislocation mobility and force exerted by each solute atom on the dislocation respectively. This force is equivalent to the product, $n_s F_s$ [46], where n_s is the number of solute atoms per unit length and the mobility M is the quantity M_s/n_s . The value n_s is described as a function of the local solute concentration (atomic fraction), C_s , as [46]:

$$n_s = \frac{b^2}{\Omega} C_s \quad (4-49)$$

where b is the Burger's vector and Ω is the atomic volume. The dislocation mobility in the presence of impurity atoms decreases due to the drag caused by segregated impurities and is described by references [45, 46]. Substituting equation (4-49) in equation (4-47), the dislocation mobility can be expressed as:

$$M_d = \frac{M_s}{n_s} = \frac{D_s \Omega}{kT b^2 C_s} \quad (4-50)$$

This equation shows that dislocation mobility increases with purity, with an upper limit ($C_s = 0$), when no impurities are present, set by the intrinsic mobility given in equation (4-95).

In order to relate the mobility term to Oxygen diffusion along the grain boundary interface, the diffusivity term, D_s , is equated with the diffusivity of Oxygen in the grain boundary, D_{O_2} , which is described by an Arrhenius type of law as:

$$D_{O_2} = D_0 e^{-\frac{Q_{O_2}}{RT}} \quad (4-51)$$

where D_0 is the frequency factor, Q_{O_2} is the stress dependent activation energy for diffusion of Oxygen in the grain boundary, R is the universal gas constant and T is absolute temperature. The Oxygen concentration in the bulk material, C_{O_2} , can be derived from Fick's first and second law and is expressed as:

$$C_{O_2} = C_0 + (C_s - C_0) \left[1 - \operatorname{erf} \left(\frac{r}{2\sqrt{D_{O_2}t}} \right) \right] \quad (4-52)$$

where C_0 is the initial concentration of Oxygen in the bulk material, C_s is the concentration of Oxygen in air, r is the distance from the crack tip and t is time. Equations (4-51) and (4-52) are substituted into equation (4-50) to provide a full definition for the mobility term in the presence of Oxygen, M_{O_2} . This term, as detailed in the next chapter, will alter the fracture criterion, measured as the critical sliding strain by evoking the following relationship [17]:

$$\varepsilon_{crit} = \varepsilon_{crit0} - z/M_{O_2} \quad (4-53)$$

where ε_{crit0} is the critical strain for grain boundary sliding in vacuum obtained as a function of temperature and z is an Oxygen diffusion related constant. This factor z/M_{O_2} addresses the complex stress-environment interactions stemming from the fact that for a hold time condition, the crack tip stress relaxation is accompanied with both a decrease of the diffusivity of Oxygen in the grain boundary, D_{O_2} , and an increase in the concentration of Oxygen, C_{O_2} . This combination of effects will result in a decrease

in mobility leading to accelerated failure of the cohesive zone element. This concept is analyzed in the next chapter.

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CHAPTER 5

CONTINUUM MATERIAL DEFORMATION MODELS

5.1 Introduction

The deformation behavior of Nickel-based superalloys can be described by numerous length scales from atomic to macroscopic or component length scales. The continuum deformation model in this chapter is developed at two length scales, a macroscopic formulation described by an internal state variable type model (ISV) and a smaller scale, described by a coarse crystal plasticity type model (XP). These two scales are a necessity for use in Chapter 6, in the cohesive zone simulation of intergranular cracking. The ISV model will be used to describe the far field material region, approximately 10 grains removed from the crack tip. The ISV model has fewer variables as compared to the XP model, thus, it improves computational efficiency. In addition, this scale is used as a means to determine the material constants necessary for implementation directly from experimental low cycle fatigue tests. Then, through a series of calculations these constants are used to determine the material parameters of the XP model. The XP model is used to describe the deformation of the near crack tip region (10x10 region of grains at the crack tip). This scale is necessary, since it considers orientation effects. The orientation allows the displacement and tractions across the grain boundary (along the crack path) to be dissociated into both normal and tangential directions. As discussed in Chapter 3, the dominant intergranular

cracking mechanism is grain boundary sliding, controlled by the tangential tractions. Without the XP model, the ISV model is insufficient to describe the tangential grain boundary traction and displacement components and would be dominated only by normal components. The low cycle fatigue experiments and model parameter determination are also outlined in Section 5.3. These model parameters are then used in one and two dimensional uniaxial simulations using the ISV model for various strain controlled loading scenarios and various temperatures. Additionally, the XP model parameters are used to simulate a two dimensional array of 100 grains under similar loading and temperature conditions as used for the ISV model.

5.2 Continuum Material Deformation Models – Theory

In order to describe the stress field surrounding the crack path, multi-scale continuum models are developed as shown in Figure 5-1. The immediate crack tip region is described by a coarse scale crystal plasticity model (XP), capable of providing both normal and tangential stress components along the grain boundary path. This is surrounded by a macroscopic scale internal state variable model (ISV) located several grains removed from the crack tip. The macroscopic model is also used as a step to determine material parameters from experimental data, which can then be converted for use at the coarse crystal plasticity scale. In addition, multi-scale modeling improves the computational efficiency. These continuum models are first used to simulate uniaxial loading scenarios and later combined with the grain boundary interface model to simulate the intergranular crack growth process.

Figure 5-1: Schematic illustrating the multi-scale model components.

5.2.1 ISV Model Formulations

The viscoplastic constitutive equations developed by Chaboche and Rousselier [1, 2] are utilized to describe the continuum cyclic and monotonic behavior of ME3 alloy. The model is formulated on the assumption that a viscoplastic potential, Ω , exists in the stress space and is a function of the viscous stress which is represented by:

$$\sigma_v = J(\sigma - X) - R - k \quad (5-1)$$

where $J(\sigma - X)$ is Von Mises second invariant, σ is the applied stress tensor and the scalar k is a temperature dependent material constant representing the initial size of the

elastic domain. R is a scalar variable corresponding to isotropic stress or drag stress and X represents the kinematic stress or back stress tensor.

The introduction of viscous stress allows a saturation of plastic strain rate above a certain strain rate. Furthermore, the relation between plastic flow and the viscoplastic potential is determined by means of the normality rule and flow rules, resulting in the plastic strain rate:

$$\dot{\epsilon}_p = \frac{3}{2} \dot{p} \frac{\sigma' - X'}{J(\sigma - X)} \quad (5-2)$$

where σ' and X' are the deviatoric parts of σ and X respectively and its modulus or accumulated strain rate can be described as:

$$\dot{p} = \left\langle \frac{\sigma_v}{K} \right\rangle^n \exp \left(\alpha \left\langle \frac{\sigma_v}{K} \right\rangle^{n+1} \right) \quad (5-3)$$

The exponential term describes the saturation of viscosity. When $\alpha = 0$, the equation is reduced to the classical power law form.

The Kinematic hardening, X is presented as the sum of several time-dependent terms [3, 4]:

$$X = \sum_i X_i \quad (5-4)$$

In order to consider the time recovery behavior of a material, an additional term is added in the back stress expression as a dependent function of the accumulated plastic strain. A general form is described by [5]:

$$\dot{X}_i = C_i \left(\frac{2}{3} a_i \dot{\epsilon}_p - X_i \dot{p} \right) - \beta_i |X_{iII}|^{r_i-1} X_i \quad (5-5)$$

where C , a , β , and r are material-dependent parameters, and X_{iII} is the second invariant of X . The first part describes the strain hardening as a function of X , plastic strain rate

and accumulated plastic strain rate. The second part, which is strongly temperature-dependent, describes the time-recovery or static-recovery as a function of X .

Two terms ($i = 1, 2$), X_1 and X_2 , of back stress are considered in this analysis. The first term, X_1 , which is a fast saturated variable, expresses the short range hardening effects. The second term, X_2 , is a quasi-linear variable describing the long range hardening effect. These two terms are written as [1, 2, 5]:

$$X = X_1 + X_2 \quad (5-6)$$

$$\dot{X}_1 = C_1 \left(a_1 \dot{\epsilon}_p - X_1 \left| \dot{\epsilon}_p \right| \right) - \beta_1 \left| X_{1II} \right|^{r_1-1} X_1 \quad (5-7)$$

$$\dot{X}_2 = C_2 \left(a_2 \dot{\epsilon}_p - X_2 \left| \dot{\epsilon}_p \right| \right) - \beta_2 \left| X_{2II} \right|^{r_2-1} X_2 \quad (5-8)$$

The constants, C_1 and C_2 are material-dependent parameters associated with the hardening rate, while a_1 and a_2 are the limiting values of the kinematic hardening variables. For very small values of C_i , X_i is nearly equivalent to a linear kinematic hardening form. For large values of C_i , X_i is of the nonlinear form and rapidly approaches the saturation value of a_i .

An introduction of an isotropic hardening part into the kinematic variable is accomplished through the function of $\phi(p)$, which describes the changes in the plastic tangent modulus as function of cyclic isotropic hardening [6]. This allows the back stress to approach an increasing value as a function of accumulated plastic strain less than the limiting saturation value of a_i with each cycle. When $\phi_s = 1$, X_i reduces to the classical Armstrong-Frederick form [7]. The function $\phi(p)$ is expressed as:

$$\phi(p) = \phi_s + (1 - \phi_s) e^{-\gamma p} \quad (5-9)$$

The back stress form including strain hardening, static recovery and the isotropic hardening term described above can then be written as:

$$\dot{X}_i = C_i \left(a_i \dot{\varepsilon}_p - \phi(p) X_i \left| \dot{\varepsilon}_p \right| \right) - \beta_i \left| X_{ill} \right|^{n_i-1} X_i \quad (5-10)$$

where $i = 1, 2$. In order to describe the evolution of back stress during each cyclic transient the material parameter ϕ_s must be determined. The back stress of the saturated cycle is expressed in the form:

$$X = \frac{a_1}{\phi_s} \tanh\left(C_1 \phi_s \frac{\Delta \varepsilon_p}{2}\right) + \frac{a_2}{\phi_s} \tanh\left(C_2 \phi_s \frac{\Delta \varepsilon_p}{2}\right) \quad (5-11)$$

During the saturated cycle, X approaches a_i/ϕ_s at a rate of $C_i \phi_s$. In this case, consider $\phi_s = 1$, since the tangent modulus does not significantly vary cycle to cycle.

The slow evolution of microstructure associated with cyclic hardening or softening of the material can be described by the isotropic hardening variable, R . This is measured as the difference in the saturation position after a loading cycle and that corresponding to the monotonic loading for the same plastic strain and is governed by the following equations:

$$\dot{R} = b(Q - R)\dot{p} \quad (5-12)$$

$$Q = Q_{\max} \left(1 - e^{-\mu q} \right) \quad (5-13)$$

$$q = \max\left(\left| \varepsilon_p \right|, q\right) \quad (5-14)$$

where Q is the saturation limit of R and b is a temperature and material-dependent parameter describing how fast R reaches Q . $Q_{\max}$ is the final value of Q , where a positive value indicates cyclic hardening and a negative value indicates cyclic

softening. The constant μ describes the rate at which Q reaches Q_{max} and q is a memory term [8] to account for the maximum strain achieved during loading.

Based on the formulations described above, the equations for the internal state variable model can be summarized as follows:

$$\begin{aligned}
\sigma_v &= |\sigma - X| - R - k \\
\dot{\varepsilon}_p &= \left\langle \frac{\sigma_v}{K} \right\rangle^n \exp \left(\alpha \left\langle \frac{\sigma_v}{K} \right\rangle^{n+1} \right) \text{sign}(\sigma - X) \\
X &= X_1 + X_2 \\
\dot{X}_1 &= C_1 (a_1 \dot{\varepsilon}_p - X_1 |\dot{\varepsilon}_p|) - \beta_1 |X_1|^{r_1-1} X_1 \\
\dot{X}_2 &= C_2 (a_2 \dot{\varepsilon}_p - X_2 |\dot{\varepsilon}_p|) - \beta_2 |X_2|^{r_2-1} X_2 \\
q &= \max(|\varepsilon_p|, q) \\
Q &= Q_{max} (1 - e^{-\mu q}) \\
\dot{R} &= b(Q - R) |\dot{\varepsilon}_p| \\
\dot{\sigma} &= E(\dot{\varepsilon}_t - \dot{\varepsilon}_p)
\end{aligned}$$

where $\langle h \rangle$ is the Heaviside function and $\text{sign}(m)$ is the sign function defined as:

$$\langle h \rangle = \begin{cases} h & \text{if } h > 0 \\ 0 & \text{if } h \leq 0 \end{cases} \text{ and } \text{sign}(m) = \begin{cases} 1 & \text{if } m > 0 \\ 0 & \text{if } m = 0 \\ -1 & \text{if } m < 0 \end{cases}$$

and 16 material constants are required for implementation in either one or two dimensional simulation. These will be experimentally determined.

5.2.2 XP Model Formulations

Crystal plasticity is based on the finite deformation theory, as outlined in references [9, 10]. In this, the current and initial coordinates of a material particle are related as:

$$dx = FdX \quad (5-15)$$

where x and X are the current and initial coordinates and F is the deformation gradient. This total deformation is considered to take place in two steps; first, from initial to intermediate and second, from intermediate to current configurations, as illustrated in Figure 5-2.

Figure 5-2: Schematic showing the initial, intermediate and current configurations of an element of material undergoing deformation [9].

During the first step, the crystal undergoes plastic deformation due to dislocation movements in which the intermediate and initial coordinates of a material particle are related as:

$$dp = F^p dX \quad (5-16)$$

where p is the intermediate coordinate and F^p is the plastic part of the deformation gradient. In this step, the crystal lattice retains its shape and orientation. During the second step, the crystal undergoes elastic deformation where no dislocation movement

is involved. This deformation consists of lattice rotation and distortion in which the current and intermediate coordinates of a material particle are related as:

$$dx = F^e dp \quad (5-17)$$

where F^e is the elastic part of the deformation gradient. Combining equations (5-15) to (5-17), the total deformation gradient can be expressed as:

$$F = F^e F^p \quad (5-18)$$

The deformation rate of the current configuration is then described by the velocity gradient, L , as:

$$L = \dot{F}F^{-1} \quad (5-19)$$

This gradient can also be decomposed into the deformation rate and spin tensor as:

$$L = D + \Omega \quad (5-20)$$

where D is the symmetric rate of stretching tensor and Ω is the antisymmetric spin tensor. These tensors can be decomposed into lattice or elastic parts (D^e , Ω^e) and plastic (D^p , Ω^p) parts as:

$$D = D^e + D^p \quad (5-21)$$

$$\Omega = \Omega^e + \Omega^p \quad (5-22)$$

where the elastic and plastic velocity gradients are defined as:

$$L^e = \dot{F}^e (F^e)^{-1} = D^e + \Omega^e \quad (5-23)$$

$$L^p = \dot{F}^p (F^p)^{-1} = D^p + \Omega^p \quad (5-24)$$

The plastic deformation caused by dislocation slip is related to the shearing strain rate of the α th slip system as [11]:

$$L^p = \sum_{\alpha=1}^{\alpha_t} \dot{\gamma}^{(\alpha)} s^{(\alpha)} m^{(\alpha)} \quad (5-25)$$

where α_t is the total number of active slip systems. Slip in the FCC crystal occurs on four unique $\{111\}$ planes and within each plane there are three independent $\langle 110 \rangle$ directions, thus 12 slip systems are considered in this work. The unit vectors $s^{(\alpha)}$ and $m^{(\alpha)}$ are the slip direction and normal to slip plane in the initial configuration, respectively. These deform and rotate as a function of the elastic deformation gradient as:

$$s^{*(\alpha)} = F^e s^{(\alpha)}; \quad m^{*(\alpha)} = m^{(\alpha)} (F^e)^{-1} \quad (5-26)$$

where $s^{*(\alpha)}$ is the vector lying along the slip direction of the α th slip system and $m^{*(\alpha)}$ is the vector normal to the slip plane in the deformed configuration. By considering the Schmid factor, $\mu_{ij}^{(\alpha)}$ and tensor, $\omega_{ij}^{(\alpha)}$, the plastic parts in the velocity gradient are related to the slip rate, $\dot{\gamma}^{(\alpha)}$, as:

$$D^p = \sum_{\alpha=1}^{\alpha_t} \mu_{ij}^{(\alpha)} \dot{\gamma}^{(\alpha)} \quad (5-27)$$

$$\Omega^p = \sum_{\alpha=1}^{\alpha_t} \omega_{ij}^{(\alpha)} \dot{\gamma}^{(\alpha)} \quad (5-28)$$

$$\mu_{ij}^{(\alpha)} = \frac{1}{2} \left[s_i^{*(\alpha)} m_j^{*(\alpha)} + s_j^{*(\alpha)} m_i^{*(\alpha)} \right] \quad (5-29)$$

$$\omega_{ij}^{(\alpha)} = \frac{1}{2} \left[s_i^{*(\alpha)} m_j^{*(\alpha)} - s_j^{*(\alpha)} m_i^{*(\alpha)} \right] \quad (5-30)$$

The resolved shearing stress on the α th slip system can be written as a function of the Schmid factor as:

$$\tau^{(\alpha)} = \mu_{ij}^{(\alpha)} \sigma_{ij} \quad (5-31)$$

where σ is the Cauchy stress tensor, which is equivalent to the 2nd Piola-Kirchhoff tensor for infinitesimal deformation. This shear stress is related to slipping rate or shear strain rate, $\dot{\gamma}^{(\alpha)}$, through the flow law. The flow rule is given as [12]:

$$\dot{\gamma}^{(\alpha)} = \left\langle \frac{|\tau^{(\alpha)} - x^{(\alpha)}| - g^{(\alpha)} - g_0^{(\alpha)}}{K^*} \right\rangle^n \text{sign}(\tau^{(\alpha)} - x^{(\alpha)}) \quad (5-32)$$

where K^* and n are material constants, $g_0^{(\alpha)}$ is the initial strength of the slip plane, $g^{(\alpha)}$ is the isotropic hardening parameter or current slip hardness of slip system α and $x^{(\alpha)}$ is the kinematic hardening parameter or back stress.

The kinematic hardening stress, $x^{(\alpha)}$, is formulated in a manner similar to the macroscopic level, where it is a two term stress including dynamic and static recovery as:

$$x^{(\alpha)} = x_1^{(\alpha)} + x_2^{(\alpha)} \quad (5-33)$$

$$\dot{x}_1^{(\alpha)} = C_1^* \left(a_1^* \dot{\gamma}^{(\alpha)} - |\dot{\gamma}^{(\alpha)}| x_1^{(\alpha)} \right) - \beta_1^* |x_1^{(\alpha)}|^{r_1^* - 1} \quad (5-34)$$

$$\dot{x}_2^{(\alpha)} = C_2^* \left(a_2^* \dot{\gamma}^{(\alpha)} - |\dot{\gamma}^{(\alpha)}| x_2^{(\alpha)} \right) - \beta_2^* |x_2^{(\alpha)}|^{r_2^* - 1} \quad (5-35)$$

where C_1^* , C_2^* , a_1^* , a_2^* , β_1^* , β_2^* , r_1^* and r_2^* are material dependent parameters, which are considered to be the same for each slip system α .

Similarly, the isotropic stress, $g^{(\alpha)}$, is formulated as:

$$\dot{g}^{(\alpha)} = b^* \left(Q^{(\alpha)} - g^{(\alpha)} \right) \dot{\gamma}^{(\alpha)} \quad (5-36)$$

$$Q^{(\alpha)} = Q_{\max}^* \left(1 - e^{-\mu^* q^{(\alpha)}} \right) \quad (5-37)$$

$$q^{(\alpha)} = \max(|\dot{\gamma}^{(\alpha)}|, q^{(\alpha)}) \quad (5-38)$$

where b^* , Q_{max}^* and μ^* are material constants, $Q^{(\alpha)}$ represents the saturation limit of $g^{(\alpha)}$ and $q^{(\alpha)}$ is a memory term. The isotropic hardening parameter can also consider latent hardening and formulated as [13]:

$$\dot{g}^{(\alpha)} = \sum_{\beta=1}^{\alpha_l} h_{\alpha\beta} \dot{\gamma}^{(\beta)} \quad (5-39)$$

where $h_{\alpha\beta}$ is the hardening modulus, allowing for coupling between hardening of slip system α due to shearing on slip system β . When $\alpha = \beta$, the hardening modulus, $h_{\alpha\alpha}$, represents self hardening and when $\alpha \neq \beta$, the hardening modulus, $h_{\alpha\beta}$, represents latent hardening. Stouffer and Dame [12] state that the hardening created by the γ' phase is larger than that created by the dislocation network in γ - γ' alloys, thus this effect can be neglected in the current study. In addition, this coarse scale model does not consider Non-Schmid phenomena such as diffusion, twinning or cross-slip, thus equations (5-36) to (5-38) are sufficient to describe the isotropic hardening component.

Based on the formulations described above, the main equations describing the flow law and hardening parameters for the coarse crystal plasticity model can be summarized as follows:

$$\begin{aligned} x^{(\alpha)} &= x_1^{(\alpha)} + x_2^{(\alpha)} \\ \dot{\gamma}^{(\alpha)} &= \left\langle \frac{|\tau^{(\alpha)} - x^{(\alpha)}| - g^{(\alpha)} - g_0^{(\alpha)}}{K^*} \right\rangle^n \text{sign}(\tau^{(\alpha)} - x^{(\alpha)}) \\ \dot{x}_1^{(\alpha)} &= C_1^* \left(a_1^* \dot{\gamma}^{(\alpha)} - |\dot{\gamma}^{(\alpha)}| x_1^{(\alpha)} \right) - \beta_1^* |x_1^{(\alpha)}|^{r_1-1} \\ \dot{x}_2^{(\alpha)} &= C_2^* \left(a_2^* \dot{\gamma}^{(\alpha)} - |\dot{\gamma}^{(\alpha)}| x_2^{(\alpha)} \right) - \beta_2^* |x_2^{(\alpha)}|^{r_2-1} \\ \dot{g}^{(\alpha)} &= b^* \left(Q^{(\alpha)} - g^{(\alpha)} \right) \dot{\gamma}^{(\alpha)} \\ Q^{(\alpha)} &= Q_{\max}^* \left(1 - e^{-\mu^* q^{(\alpha)}} \right); \quad q^{(\alpha)} = \max(|\dot{\gamma}^{(\alpha)}|, q^{(\alpha)}) \end{aligned}$$

The equations described above are implemented into a UMAT subroutine following the work of Huang [14]. In this, 19 material constants describing the hardening laws are required, which will be experimentally determined in Section 5.3. These constants are assumed to be the same for each set of slip systems. The model also requires initial orientations of each grain in order to define the orientations of the 12 slip systems of the FCC crystal. In total, there are 198 material constants and 161 material variables in the subroutine. The next section will describe the parameter determination for the XP model based on the ISV material parameters.

5.3 Material Parameters Determination - Experiments and Analysis

The set of parameters required to implement the ISV and XP material models, previously described, are determined here using the results of low cycle fatigue tests carried out at different loading rates and strain ranges for each temperature under consideration. These tests, analysis of their output and their manipulation to generate model parameters are described in this section.

5.3.1 Low Cycle Fatigue Tests and Results

The mechanical testing was carried out on smooth cylindrical specimens using a MTS servohydraulic test machine. The gage sections of the specimens were 0.25 in diameter and 0.8 in length and were polished to a 1 micron finish prior to testing (see Appendix A for specimen drawing). The high temperature tests were made possible by

heating with a two zone ATS furnace and measuring the strain with an MTS quartz rod extensometer. The temperature of the top, center, and bottom of the specimen were measured with type K thermocouples, maintaining a temperature of $\pm 1^\circ\text{C}$ throughout the entire test. The experimental test setup is shown below in Figure 5-3.

Figure 5-3: Low cycle fatigue experimental setup in the Mechanics of Materials Research Laboratory at the University of Rhode Island.

The specimens were heated at an average rate of $4^\circ\text{C}/\text{min}$ under force control, at zero force, to allow for thermal expansion. Once the desired test temperature of 650°C , 704°C or 760°C , was reached, the specimen was held at constant temperature for 1 hour before starting the test. A series of tests were carried out on the M1 material at three temperatures, 650, 704 and 760°C , under strain controlled loading conditions. These tests include fully reversed cyclic loading, monotonic and stress relaxation (hold at constant strain) testing. The set of experiments carried out are shown in Table 5-1. In this table, the strain rate and strain range are indicated for cyclic tests, strain

rate and total strain value are indicated for monotonic tests, and the strain value and hold time are indicated for stress relaxation tests. Results of these low cycle fatigue tests for M1 microstructure in the form of stress versus strain relationships are shown in Figures 5-4 to 5-6 for monotonic and cyclic loading at different strain ranges.

Table 5-1: Strain controlled low cycle fatigue experiments carried out at URI for M1 microstructure.

Temperature	Type of Test	Test Specifications
650°C	Cyclic	$10^{-4} \text{ s}^{-1} \pm 0.5, \pm 0.6, \pm 0.7\%$,
	Monotonic	10^{-4} s^{-1} to 0.8%,
	Stress Relaxation	Hold at 0.8% (~2 hrs),
	Monotonic	10^{-4} s^{-1} to 1%
	Stress Relaxation	Hold at 1% (~2.1hrs),
	Monotonic	10^{-4} s^{-1} to 1.4%,
	Stress Relaxation	Hold at 1.4% (~2.6hrs),
	Monotonic	10^{-4} s^{-1} to 2%
650°C	Cyclic	$10^{-4} \text{ s}^{-1} \pm 0.8, \pm 0.9\%$
704°C	Monotonic	10^{-6} s^{-1} to 1%, 10^{-5} s^{-1} to 2%, 10^{-4} s^{-1} to 3%
704°C	Cyclic	$10^{-4} \text{ s}^{-1} \pm 0.5, \pm 0.7, \pm 1\%$,
	Stress Relaxation	Hold at 1 % (~2.5hrs),
	Monotonic	10^{-3} s^{-1} to 2%
704°C	Cyclic	$10^{-4} \text{ s}^{-1} \pm 0.6, \pm 0.8, \pm 1, \pm 1.2, \pm 1.4\%$,
	Stress Relaxation	Hold at 1.4% (~14hrs),
	Cyclic	$10^{-4} \text{ s}^{-1} \pm 1.4\%$,
	Stress Relaxation	Hold at 1.4% (~4.25hrs),
	Cyclic	$10^{-4} \text{ s}^{-1} \pm 1.5\%$,
	Stress Relaxation	Hold at 1.5% (~2.5hrs)
760°C	Monotonic	10^{-4} s^{-1} to 0.8%,
	Stress Relaxation	Hold at 0.8% (~2hrs),
	Monotonic	10^{-4} s^{-1} to 1.2%,
	Stress Relaxation	Hold at 1.2% (~2hrs),
	Monotonic	10^{-4} s^{-1} to 1.6%,
	Stress Relaxation	Hold at 1.6% (~2.1hrs),
	Monotonic	10^{-4} s^{-1} to 2%, 10^{-5} s^{-1} to 3%, 10^{-6} s^{-1} to 4%
760°C	Cyclic	$10^{-4} \text{ s}^{-1} \pm 0.6, \pm 0.8, \pm 0.9, \pm 1, \pm 0.012, \pm 0.014, \pm 0.015\%$

Figure 5-4: Experimental stress versus strain results at 650°C for M1 microstructure for cyclic loading at a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ at strain ranges (a) $\pm 0.8\%$ and (b) $\pm 0.5\%$, $\pm 0.6\%$, and $\pm 0.7\%$. (c) Monotonic loading at a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ with hold in strain control at 0.8%, 1% and 1.4%.

Figure 5-5: Experimental stress versus strain results at 704°C for M1 microstructure for (a) monotonic loading at a strain rate of $1 \times 10^{-6} \text{ sec}^{-1}$ to 1% strain, $1 \times 10^{-5} \text{ sec}^{-1}$ to 2% strain and $1 \times 10^{-4} \text{ sec}^{-1}$ to 4% strain. (b) Cyclic loading at a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ at strain ranges $\pm 0.5\%$, $\pm 0.7\%$ and $\pm 1\%$, with a hold in strain control at 0.5% and 0.7%. (c) Cyclic loading at a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ at strain ranges $\pm 0.6\%$, $\pm 0.8\%$ and $\pm 1\%$.

Figure 5-6: Experimental stress versus strain results at 760°C for M1 microstructure for (a) monotonic loading at a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ with hold in strain control at 0.8%, 1.2% and 1.6% followed by monotonic loading at a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ to 2% strain, $1 \times 10^{-5} \text{ sec}^{-1}$ to 3% strain and $1 \times 10^{-6} \text{ sec}^{-1}$ to 4% strain. (b) Cyclic loading at a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ at strain ranges $\pm 0.6\%$, $\pm 0.8\%$, $\pm 0.9\%$, $\pm 1\%$, $\pm 1.2\%$, $\pm 1.4\%$ and $\pm 1.5\%$.

Results of the stress relaxation tests as a function of time for the three temperature conditions are shown in Figure 5-7. It is clear that the rate of relaxation, over the same time duration, increases with temperature. To illustrate this pattern, in 8000 seconds the stress level reaches the magnitudes of 800 MPa, 700 MPa and 600 MPa at 650, 704 and 760°C; respectively. These trends are significant in relation to crack tip stress relaxation conditions and the spread of the viscoplastic affected zone.

Figure 5-7: Experimental stress relaxation versus time profiles for M1 microstructure at (a) 650°C for a hold in strain control at 0.8%, 1% and 1.4%, (b) 704°C for a hold in strain control at 0.7%, 1% and 1.5% and (c) 760°C for a hold in strain control at 0.8%, 1.2% and 1.6%.

General Observations

The relationship between the 0.2% offset yield stress and temperature is shown in Figure 5-8. This inverse relationship is similar to that obtained independently by Pratt and Whitney by testing the same material at 704°C and 760°C as indicated in the same figure. These results do not show anomalous yielding effects as observed in

other Nickel-based superalloys [15], where the increase in yield with temperature is caused by the formation of Kear-Wilsdorf locks. This increase is typically seen up to temperatures between 700°C and 800°C. It has been shown for a Ni-Cr-Al alloy that an alloy consisting of 100% γ' has a strong positive dependence of yield with temperature as compared to an alloy consisting of 100% γ . ME3 alloy consists of approximately 50% γ' which can shift the peak of the temperature dependence to a lower value.

The cyclic loading curves in Figures 5-4 to 5-6 are characterized by cyclic hardening and softening. The hardening behavior is described by the evolution of isotropic and kinematic hardening parameters as a function of plastic strain. The isotropic hardening describes how the peak stress evolves from cycle to cycle as a function of accumulated plastic strain. The isotropic stress is calculated as the difference between the initial monotonic and saturated cyclic stress strain curves, as shown in Figure 5-9 for the three temperatures.

Figure 5-8: Yield stress (0.2% offset) as a function of temperature for M1 microstructure.

Figure 5-9: Isotropic stress as a function of accumulated plastic strain for M1 microstructure at 650°C, 704°C and 760°C.

Figure 5-9 shows that for temperatures 650 and 704°C, cyclic hardening is observed where the isotropic stress increases with plastic strain, while at 760°C, cyclic softening is observed. In addition to isotropic hardening, the cyclic stress strain loops show evidence of the Bauschinger effect. The kinematic stress is measured and is plotted as a function of plastic strain in Figure 5-10. Figure 5-10a shows that the two temperatures 704 and 760°C have a similar kinematic stress and lower values at 650°C. Similarly, Figure 5-10b and 5-10c show that the components X_1 and X_2 are similar at 704 and 760°C. At 650°C, X_1 is lower and X_2 is higher, then X_1 and X_2 at 704 and 760°C. Since X_2 reflects the influence of γ' precipitates, Figure 5-10c implies that they are more influential at 650°C to the total back stress.

Figure 5-10: (a) Total kinematic stress, X , (b) term 1, X_1 , and (c) term 2, X_2 , as a function of plastic strain for M1 microstructure at 650°C, 704°C and 760°C.

The effect of temperature on stress relaxation has also been observed in terms of the viscous stress component. Figure 5-11 shows a schematic of the viscous stress component ($\Delta\sigma$) measured as the difference in stress at 0 sec hold time and an arbitrary hold time of 1000 sec and 6000 sec from the stress relaxation curve.

Figure 5-11: Schematic of viscous stress component, $\Delta\sigma$.

This component has been measured for M1 microstructure at the three temperatures, 650, 704 and 760°C for stress relaxation curves at multiple constant strain values from Figure 5-7 and is shown in Figure 5-12.

Figure 5-12: Viscous stress component, $\Delta\sigma$, as a function of strain for M1 microstructure at 650°C, 704°C and 760°C.

Figure 5-12 shows that for the same total strain, stress relaxation is independent of temperature, indicating similar strain rate sensitivity as a function of temperature. The viscous stress component measured at a short hold time (1000 sec) is lower than that measured at 6000 sec. Each component (at 1000 sec and 6000 sec) is directly proportional to strain. Furthermore, the large value of the viscous component indicates that time dependency plays an important role in the deformation behavior of ME3.

Shifting attention to the general observations of the surfaces of low cycle fatigue specimens, indicate the presence of slip band traces, the spacing of which seems to correlate inversely with the loading rate. For a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ and a maximum strain range of 1.5% at 760°C, the slip line spacing is in the range of 2.4 to 6.1 microns. Typical surface slip lines are shown in Figure 5-13. In addition, extensive cracking has been observed on the specimen surfaces, see Figures 5-14 and 5-15.

These cracks are located primarily along boundaries that are normal to the loading direction and at the intersection between a slip band and grain boundary, as shown in Figure 5-16. Furthermore, fracture surface features of broken low cycle fatigue specimens have shown to be dominated by intergranular facets, see Figure 5-17.

Figure 5-13: Slip band spacing for M1 microstructure at 760°C which has been fatigued at $1 \times 10^{-4} \text{ sec}^{-1}$ at a maximum strain range of 1.5%.

Figure 5-14: Grain boundary fracture along the gage section of an LCF specimen for M1 microstructure at 760°C which has been fatigued at $1 \times 10^{-4} \text{ sec}^{-1}$ at a maximum strain range of 1.5%.

Figure 5-15: Grain boundary fracture for M1 microstructure at 760°C which has been fatigued at $1 \times 10^{-4} \text{ sec}^{-1}$ at a maximum strain range of 1.5%.

Figure 5-16: Grain boundary fracture at the intersection of a slip line and grain boundary for M1 microstructure at 760°C which has been fatigued at $1 \times 10^{-4} \text{ sec}^{-1}$ at a maximum strain range of 1.5%.

Figure 5-17: Typical intergranular fracture surface for M1 microstructure at 760°C which has been fatigued at $1 \times 10^{-4} \text{ sec}^{-1}$ at a maximum strain range of 1.5%.

5.3.2 ISV Model Parameter Determination

A series of isothermal strain-controlled tests including monotonic, relaxation, cyclic and strain rate sensitivity, are carried out on smooth specimens at high temperature, to determine the various material parameters described above to fully identify the ISV model. These identification procedures are detailed in references [8, 16]. A monotonic test is carried out in order to determine the modulus, E , and yield stress, k , of the material. Furthermore, results of the cyclic stress-strain tests, described above, are used to determine the hardening parameters as follows.

Kinematic Hardening Parameters

For each strain range, the hardening of the material in the first cycle of the cyclic stress-strain loops is assumed to be related to the kinematic hardening only and the accumulated plastic strain p is equation to the plastic strain ε_p . The kinematic stress corresponds to the center of the linear part of the first reversible cyclic loop at each strain range, as schematically shown in Figure 5-18. From this figure, X is determined as:

$$X = \sigma_{\max} - \frac{\sigma_{\max} + |\sigma_{\min}|}{2} = \sigma_{\max} - \sigma_y^{\text{tensile}} \quad (5-40)$$

Neglecting the recovery term, X can be represented as the sum of two terms ($i = 1, 2$):

$$X = \sum_i a_i (1 - e^{-C_i \varepsilon_p}) = a_1 (1 - e^{-C_1 \varepsilon_p}) + a_2 (1 - e^{-C_2 \varepsilon_p}) \quad (5-41)$$

where a_i and C_i are obtained from curve fitting of experimental X versus ε_p data as shown in Figure 5-19.

Figure 5-18: Schematic illustration of the kinematic hardening stress X .

The time-dependent recovery parameters, β_1, β_2 , are determined from the stress time curve shown in Figure 5-7. The time dependent back stress can be expressed by rearranging equation (5-1) to the form:

$$X(t) = \sigma(t) - (R + k + \sigma_v) \quad (5-42)$$

As time approaches infinity, stress is assumed to be linearly dependent on time and the latter terms in equation (5-42) are constant. Using a linear recovery term, $r_i = 1$, as time approaches infinity, X can also be expressed as:

$$X(t) = \frac{C_1 a_1 \dot{p}}{C_1 \dot{p} + \beta_1} + \frac{C_2 a_2 \dot{p}}{C_2 \dot{p} + \beta_2} \quad (5-43)$$

Equation (5-42) can be plotted as function of time and fit to equation (5-43). The parameter, β_1 , can be approximated as 0 since X_1 saturates quickly, and β_2 , is approximated by equation (5-43).

Figure 5-19: Kinematic stress versus plastic strain for M1 microstructure at (a) 650°C, (b) 704°C and (c) 760°C.

Isotropic Hardening Parameters

The slow internal changes, described by isotropic stress, occur over a large number of cycles which is dependent on accumulated plastic strain. The isotropic stress, R , corresponds to the difference in the maximum stress of the current cycle and the maximum stress of the first loop associated with the monotonic stress-strain curve, as shown in Figure 5-20.

Figure 5-20: Schematic illustration of the isotropic hardening stress R .

Assuming the increase in maximum stress occurs only due to isotropic hardening, R , the following relationship can be determined through integration of equation (5-12), where the ratio R/Q experimentally is given as:

$$\frac{R}{Q} = \frac{\sigma_M - \sigma_{M0}}{\sigma_{MS} - \sigma_{M0}} = (1 - e^{-bp}) \quad (5-44)$$

where σ_M , σ_{MS} , σ_{M0} correspond to the maximum stress of cycle M , the maximum stress of the saturated cyclic loop and the first loop respectively. The accumulated plastic strain, p , is defined by $p = |\varepsilon_p| = 2N\Delta\varepsilon_p$. N is the number of cycles and ε_p is the sum of plastic strain in tension and compression. The material parameter, b , is determined by fitting of the above equation to the experimental R/Q versus p data as shown in Figure 5-21. Q_{max} and μ are calculated by fitting of equation (5-13), to the experimental Q versus q data as shown in Figure 5-22, where $Q = \sigma_{MS} - \sigma_{M0}$ and q is the maximum plastic strain achieved during loading, in this case $q = \Delta\varepsilon_p/2$. These values of Q and q are taken for different strain ranges.

Figure 5-21: R/Q versus log of accumulated plastic strain for M1 microstructure at (a) 650°C, (b) 704°C and (c) 760°C.

Figure 5-22: Q versus q for M1 microstructure at (a) 650°C, (b) 704°C and (c) 760°C.

Viscous Stress Parameters

To describe the time-dependent viscous stress term, n , K and α , strain-rate sensitive parameters must be determined. Thus, relaxation tests or strain-controlled monotonic or cyclic stress-strain tests are performed with points at which strain is held constant for periods ranging from 11.5 to 840 minutes. The relaxation stress versus hold time at various total strain values are shown in Figure 5-7 for M1 microstructure at 650, 704 and 760°C. Under constant strain conditions during hold time, the plastic strain rate can be expressed in terms of stress rate and modulus as:

$$\dot{\varepsilon}_p = -\dot{\sigma}/E \quad (5-45)$$

In order to define the plastic strain rate as shown above, stress and stress rate are determined as a function of hold time. The plastic strain rate can also be expressed by:

$$\dot{\epsilon}_p = \langle \sigma_v / K \rangle^n \quad (5-46)$$

where the viscous stress can be written as the sum of two terms:

$$\sigma_v = \sigma - \sigma_i = \sigma - X - R - k \quad (5-47)$$

For different values of σ_i , the viscous stress versus plastic strain rate can be plotted, as shown in Figure 5-23. The parameters n and K are determined from fitting the linear part of the log-log curve to equation (5-46), an average of n and K are taken for each total strain value (from the hold at constant strain from Figure 5-7). The material constant, α , is taken to be the saturation limit of viscous stress for high plastic strain rates. This value is determined at large plastic strain rates.

All material parameters obtained from the experimental results in the manner described above have been optimized using a nonlinear parameter identification technique employing the commercial program iSIGHT [17]. Figure 5-24 shows a flow chart of the optimization scheme. The optimized material parameters, obtained from the scheme shown in Figure 5-24, are shown in Table 5-2 below.

Figure 5-23: Typical viscous stress versus plastic strain rate for M1 microstructure at (a) 650°C at a total strain of 1%, (b) 704°C at a total strain of 1% and (c) 760°C at a total strain of 1.2%. The solid black line is a fit to equation (5-46).

Figure 5-24: Optimization flow chart using MATLAB and iSIGHT.

Table 5-2: Optimized 1-D ISV material constants for ME3 (M1 As Received) at 650°C, 704°C and 760°C. Note this table lists the temperature dependent constants, the temperature independent constants are: $C_2 = 1$, $b = 27$, $\alpha = 0$, $r_2 = 1$.

T (°C)	E (MPa)	k (MPa)	a_1 (MPa)	C_1	a_2 (MPa)	Q_{max} (MPa)	μ	n	K (MPa)	β_1 (Pa ^{r₁})	β_2 (Pa ^{r₁})	r_1
650	192140	450	232	675	31877	94	1192	5.7	2719	0	0	1
704	182140	350	312	690	21877	20	555	6.1	2184	2.00E-09	0.02	1.1
760	165939	240	312	690	21877	-170	167	6.6	1800	2.00E-09	0.02	1.2

Two Dimensional ISV material parameters

The material parameters for the 2-D ISV model have been determined from the 1-D ISV material parameters and are given in Table 5-3.

Table 5-3: The constants for the 2-D ISV model for ME3 (M1 As Received) at 650°C, 704°C and 760°C. Note this table lists the temperature dependent constants, the temperature independent constants are: $C_2 = 1$, $b = 27$, $\alpha = 0$, $r_2 = 1$.

T (°C)	E (MPa)	k (MPa)	a_1 (MPa)	C_1	a_2 (MPa)	Q_{max} (MPa)	μ	n	K (MPa)	β_1 (Pa ^{r₁})	β_2 (Pa ^{r₁})	r_1
650	192140	450	162.4	675	22313.9	94	1192	5.7	2719	0	0	1
704	182140	350	218.4	690	15313.9	20	555	6.1	2184	2.00E-09	0.02	1.1
760	165939	240	218.4	690	15313.9	-170	167	6.6	1800	2.00E-09	0.02	1.2

5.3.3 XP Model Parameter Determination

The material parameters for the crystal plasticity model can be approximated from the 2-D ISV model using the Taylor factor, M . The Taylor factor [18] is defined as:

$$M = \sigma/\tau \quad (5-48)$$

where σ is the external stress and τ is the shear stress. In order to define the shear strain in terms of external strain we need to define the internal incremental work or volume expended by slip in a given grain as:

$$dw_{\text{int}} = \sum \tau^i d\gamma^i \quad (5-49)$$

Where τ^i is the shear stress required for slip on the i th slip system and $d\gamma^i$ is the shear strain on that slip system. It is also necessary to define the external incremental work as a function of external stress and strain as:

$$dw_{\text{ext}} = \sigma d\varepsilon \quad (5-50)$$

Assuming each grain of a polycrystal experiences the same volume change as the entire polycrystal, we can equate the internal work to the external work as:

$$dw_{\text{int}} = dw_{\text{ext}} \quad (5-51)$$

Letting $\tau d\gamma = \sum \tau^i d\gamma^i$ and substituting equation (5-49) and (5-50) into (5-51) yields:

$$\tau d\gamma = \sigma d\varepsilon \quad (5-52)$$

Equation (5-52) can be rearranged and equated with equation (5-48) to give:

$$\frac{d\gamma}{d\varepsilon} = \frac{\sigma}{\tau} = M \quad (5-53)$$

Thus, the external stress and strain, in terms of shear stress and shear strain, are defined as:

$$\varepsilon = \gamma / M \quad (5-54)$$

$$\sigma = M \tau \quad (5-55)$$

Figure 5-25 below shows an example of implementing the above equations on a stress-strain curve. These relationships have been used to convert the global isotropic and kinematic stress and strain variables into local variables. In addition, the stiffness

matrix for an FCC cubic material can be calculated using Young's modulus, E , and Poisson's ratio, ν , [19] as:

$$C_{11} = \frac{E(1-\nu)}{(1-\nu-2\nu^2)}, \quad C_{12} = \frac{E\nu}{(1-\nu-2\nu^2)}, \quad C_{44} = G = \frac{E}{2(1+\nu)} \quad (5-56)$$

A summary of the material constants corresponding to the local variables for the XP model are shown in Table 5-4.

Figure 5-25: Comparison of stress-strain curves for single and polycrystalline aluminum. The polycrystalline curve was constructed from the single-crystal curve using a Taylor factor of 3.06 [18].

Table 5-4: 2-D XP material constants for ME3 (M1 As Received) at 650°C, 704°C and 760°C. Note this table lists the temperature dependent constants, the temperature independent constants are: $\nu = 0.3$, $C_2^* = 0.3$, $b^* = 8.8$, $r_2 = 1$. Furthermore, the static recovery terms (r_1 , r_2 , β_1 , β_2) in the XP model are considered to be the same as those listed in Table 5-3.

T (°C)	C_{11} (MPa)	C_{12} (MPa)	C_{44} (MPa)	k^* (MPa)	a_1^* (MPa)	C_1^*	a_2^* (MPa)	Q_{max}^* (MPa)	μ^*	n	K^* (MPa)
650	258650	110850	73900	147.1	53.1	220.6	7292.1	30.7	389.5	5.7	888.6
704	245188.5	105080.8	70053.8	114.4	71.4	225.5	5004.5	6.5	181.4	6.1	713.7
760	223379.4	95734	63822.7	78.4	71.4	225.5	5004.5	-55.6	54.6	6.6	588.2

5.4 Uniaxial Model Simulations

One-Dimensional Simulation of ISV Model using Matlab

Based on the non-linear kinematic hardening model the equations used to simulate one-dimensional monotonic and cyclic behavior are summarized as follows:

$$\begin{aligned}
 X(t) &= X_1(t) + X_2(t) \\
 \sigma_v &= |\sigma - X| - R - k \\
 \dot{\varepsilon}_p &= \left\langle \frac{\sigma_v}{K} \right\rangle^n \exp \left(\alpha \left\langle \frac{\sigma_v}{K} \right\rangle^{n+1} \right) \text{sign}(\sigma - X) \\
 \dot{X}_1 &= C_1 (a_1 \dot{\varepsilon}_p - X_1 |\dot{\varepsilon}_p|) - \beta_1 |X_1|^{\eta-1} X_1 \\
 \dot{X}_2 &= C_2 (a_2 \dot{\varepsilon}_p - X_2 |\dot{\varepsilon}_p|) - \beta_2 |X_2|^{\eta_2-1} X_2 \\
 q &= \max(|\varepsilon_p|, q) \\
 Q &= Q_{\max} (1 - e^{-\mu q}) \\
 \dot{R} &= b(Q - R) |\dot{\varepsilon}_p| \\
 \dot{\sigma} &= E(\dot{\varepsilon}_t - \dot{\varepsilon}_p)
 \end{aligned}$$

Modeling was carried out for 1-D simulation to check applicability of model. For this, a MATLAB [20] program was implemented for uniaxial loading (see Appendix C for description of codes). The set of viscoplastic constitutive equations described above, are integrated simultaneously using the MATLAB ordinary differential equation solver. A flow chart of the 1-D simulation is given in Figure 5-26.

Figure 5-26: Flow chart of the ISV Model using Matlab in strain-controlled loading condition.

Using the list of material parameters generated above, the following case studies under strain-controlled conditions were simulated using the ISV material model:

- Set 1: Cyclic loading at 650°C
 - (1-1) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 0.8\%$
- Set 2: Cyclic loading at 650°C
 - (2-1) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 0.5\%$

- (2-2) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 0.6\%$
- (2-3) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 0.7\%$
- Set 3: Monotonic and relaxation loading at 650°C
 - (3-1) Monotonic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, 0% to 0.8%
 - (3-2) Hold in strain control at 0.8%
 - (3-3) Monotonic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, 0.8% to 1%
 - (3-4) Hold in strain control at 1%
 - (3-5) Monotonic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, 1% to 1.4%
 - (3-6) Hold in strain control at 1.4%
 - (3-7) Monotonic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, 1.4% to 2%
- Set 4: Monotonic loading with variable strain rates at 704°C
 - (4-1) Monotonic loading with a strain rate of $1 \times 10^{-6} \text{ sec}^{-1}$, 0% to 1%
 - (4-2) Monotonic loading with a strain rate of $1 \times 10^{-5} \text{ sec}^{-1}$, 1% to 2%
 - (4-3) Monotonic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, 2% to 4%
- Set 5: Cyclic and relaxation loading at 704°C
 - (5-1) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 0.5\%$
 - (5-2) Hold in strain control at 0.5%
 - (5-3) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 0.7\%$
 - (5-4) Hold in strain control at 0.7%
 - (5-5) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 1\%$
- Set 6: Cyclic loading at 704°C
 - (6-1) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 0.6\%$
 - (6-2) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 0.8\%$
 - (6-3) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 1\%$
- Set 7: Monotonic and relaxation loading at 760°C
 - (7-1) Monotonic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, 0% to 0.8%
 - (7-2) Hold in strain control at 0.8%
 - (7-3) Monotonic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, 0.8% to 1.2%
 - (7-4) Hold in strain control at 1.2%
 - (7-5) Monotonic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, 1.2% to 1.6%
 - (7-6) Hold in strain control at 1.6%
 - (7-7) Monotonic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, 1.6% to 2%
 - (7-8) Monotonic loading with a strain rate of $1 \times 10^{-5} \text{ sec}^{-1}$, 2% to 3%
 - (7-9) Monotonic loading with a strain rate of $1 \times 10^{-6} \text{ sec}^{-1}$, 3% to 4%
- Set 8: Cyclic loading at 760°C
 - (8-1) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 0.6\%$
 - (8-2) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 0.8\%$
 - (8-3) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 0.9\%$
 - (8-4) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 1\%$
 - (8-5) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 1.2\%$
 - (8-6) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 1.4\%$
 - (8-7) Cyclic loading with a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$, $\pm 1.5\%$

Results of the case studies mentioned previously are shown in Figures 5-27 to 5-29. These results are compared with those obtained experimentally under similar loading conditions.

Figure 5-27: Experimental versus analytical stress-strain curves for M1 at 650°C for (a) set 1, (b) set 2 and (c) set 3, where the analytical curves are produced using the one-dimensional ISV model.

Figure 5-28: Experimental versus analytical stress-strain curves for M1 at 704°C for (a) set 4, (b) set 5 and (c) set 6, where the analytical curves are produced using the one-dimensional ISV model.

Figure 5-29: Experimental versus analytical stress-strain curves for M1 at 760°C for (a) set 7 and (c) set 8, where the analytical curves are produced using the one-dimensional ISV model.

Two-Dimensional Simulation of ISV Model using Abaqus

The non-linear kinematic hardening model equations used to simulate one-dimensional monotonic and cyclic behavior are extended to their tensor forms in order to simulate two-dimensional behavior; these equations are summarized as follows:

$$\begin{aligned}
X_{ij}(t) &= X_{ij}^1(t) + X_{ij}^2(t) \\
\sigma_{ij}^v &= J_2(\sigma_{ij} - X_{ij}) - R - k \\
\dot{p} &= \left\langle \frac{\sigma_{ij}^v}{K} \right\rangle^n \exp \left(\alpha \left\langle \frac{\sigma_{ij}^v}{K} \right\rangle^{n+1} \right) \\
\dot{X}_{ij}^1 &= C_1(a_1 \dot{\varepsilon}_{ij}^p - X_{ij}^1 \dot{p}) - \beta_1 (X_{1II})^{r_1-1} X_{ij}^1 \\
\dot{X}_{ij}^2 &= C_2(a_2 \dot{\varepsilon}_{ij}^p - X_{ij}^2 \dot{p}) - \beta_2 (X_{2II})^{r_2-1} X_{ij}^2 \\
q &= \max(|\varepsilon_{ij}^p|, q) \\
Q &= Q_{\max} (1 - e^{-\mu q}) \\
\dot{R} &= b(Q - R) \dot{p} \\
\sigma_{ij} &= \lambda \varepsilon_{kk}^e \delta_{ij} + 2\mu \varepsilon_{ij}^e
\end{aligned}$$

where the equivalent plastic strain is calculated as:

$$|\varepsilon_{ij}^p| = \sqrt{\frac{2}{3} \varepsilon_{ij}^p \varepsilon_{ij}^p} \quad (5-57)$$

The material model describing the non-linear kinematic hardening model was implemented through a UMAT subroutine (see Appendix C for description of codes). The set of viscoplastic constitutive equations described above, are integrated simultaneously using the 4th order Runge-Kutta method. In order to carry out the 2-D simulation, a finite element model was built. This model consists of a single axisymmetric element. A 4-node bilinear axisymmetric quadrilateral element, type C4X4, is used in Abaqus [21]. The length and width of this element is 1 mm. The left edge is fixed in the x direction, free to move in the y direction and rotation is not allowed in the z direction. The bottom edge is fixed in the y direction, free to move in the x direction and rotation is not allowed in the z direction. The top edge has a displacement amplitude applied in the y direction. Since this is a unit element (1 mm x 1 mm), the displacement applied to the top edge is equivalent to the strain. Thus,

applying a displacement as a function of time is the same as simulating a strain controlled condition. The same case studies performed in the 1-D ISV simulations are performed using the 2-D ISV model. The results are shown in Figures 5-30 to 5-32.

Figure 5-30: Experimental versus analytical stress-strain curves for M1 at 650°C for (a) set 1, (b) set 2 and (c) set 3, where the analytical curves are produced using the two-dimensional ISV model.

Figure 5-31: Experimental versus analytical stress-strain curves for M1 at 704°C for (a) set 4, (b) set 5 and (c) set 6, where the analytical curves are produced using the two-dimensional ISV model.

Figure 5-32: Experimental versus analytical stress-strain curves for M1 at 760°C for (a) set 7 and (c) set 8, where the analytical curves are produced using the two-dimensional ISV model.

Two-Dimensional Simulation of XP Model using Abaqus

The main non-linear kinematic hardening equations used to simulate a single crystal can be summarized as follows:

$$\begin{aligned}
x^{(\alpha)} &= x_1^{(\alpha)} + x_2^{(\alpha)} \\
\dot{\gamma}^{(\alpha)} &= \left\langle \frac{|\tau^{(\alpha)} - x^{(\alpha)}| - g^{(\alpha)} - g_0^{(\alpha)}}{K^*} \right\rangle^n \text{sign}(\tau^{(\alpha)} - x^{(\alpha)}) \\
\dot{x}_1^{(\alpha)} &= C_1^* \left(a_1^* \dot{\gamma}^{(\alpha)} - |\dot{\gamma}^{(\alpha)}| x_1^{(\alpha)} \right) - \beta_1^* |x_1^{(\alpha)}|^{r_1-1} \\
\dot{x}_2^{(\alpha)} &= C_2^* \left(a_2^* \dot{\gamma}^{(\alpha)} - |\dot{\gamma}^{(\alpha)}| x_2^{(\alpha)} \right) - \beta_2^* |x_2^{(\alpha)}|^{r_2-1} \\
\dot{g}^{(\alpha)} &= b^* \left(Q^{(\alpha)} - g^{(\alpha)} \right) \dot{\gamma}^{(\alpha)} \\
Q^{(\alpha)} &= Q_{\max}^* \left(1 - e^{-\mu^* q^{(\alpha)}} \right) \\
q^{(\alpha)} &= \max(|\gamma^{(\alpha)}|, q^{(\alpha)})
\end{aligned}$$

The material model describing the non-linear kinematic hardening model was implemented through a UMAT subroutine (see Appendix C for description of codes). In order to carry out the 2-D XP simulation, a finite element model in Abaqus [21] was created. This model consists of an assembly of hexagonal grains of grain size, d . An array of 10 x 10 grains are assembled into a rectangular polycrystal. Each grain is assigned an orientation within $\pm 15^\circ$ of its neighboring grains using a MATLAB [20] program (see Appendix C for description of codes). The rectangular polycrystal assembly of length, l , and height, h , is shown in Figure 5-33a. The left edge is fixed in the x direction, free to move in the y direction and rotation is not allowed in the z direction. The bottom edge is fixed in the y direction, free to move in the x direction and rotation is not allowed in the z direction. A displacement-time profile is applied in the y direction along the top edge, where displacement is related to strain by the following relationship:

$$d_y = h \varepsilon_y \quad (5-58)$$

The grains are meshed using a 4-node bilinear plane strain quadrilateral element, type CPE4, as shown in Figure 5-33b. The same case studies performed in the 1-D and 2-D

ISV simulations are performed using the 2-D XP model. The results are shown in Figures 5-34 to 5-36.

Figure 5-33: Polycrystal model consisting of 100 grains, used for 2-D uniaxial tension-compression simulations with the XP material model showing (a) loading and boundary conditions and (b) meshing.

Figure 5-34: Experimental versus analytical stress-strain curves for M1 at 650°C for (a) set 1, (b) set 2 and (c) set 3, where the analytical curves are produced using the two-dimensional crystal plasticity model consisting of 100 grains.

Figure 5-35: Experimental versus analytical stress-strain curves for M1 at 704°C for (a) set 4, (b) set 5 and (c) set 6, where the analytical curves are produced using the two-dimensional crystal plasticity model consisting of 100 grains.

Figure 5-36: Experimental versus analytical stress-strain curves for M1 at 760°C for (a) set 7 and (c) set 8, where the analytical curves are produced using the two-dimensional crystal plasticity model consisting of 100 grains.

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CHAPTER 6

DWELL CRACK GROWTH SIMULATION

6.1 Introduction

The previous chapters provided the basic models for continuum and grain boundary interface time dependent deformation response of ME3 alloy. These models are used here in a finite element approach to simulate the dwell crack growth, as illustrated previously in Figure 5-1. A comparison of simulation results with those obtained experimentally defines the validity of the developed cohesive zone model and provides a basis to examine the dwell crack growth rate in this alloy under different material and environment conditions that have not been used in the modeling efforts. In addition, case studies varying Oxygen partial pressure and grain boundary diffusivity have been performed in order to study the model sensitivity.

6.2 Finite Element Model

The crack growth simulations were carried out on a half model of CT12.5 specimen geometry. The specimen dimensions are shown in Figure 6-1a. The specimen thickness is 6.35 mm. The small rectangular zone in Figure 6-1a, which is modeled by the crystal plasticity model, is an array of 100 grains; each grain has a size of 44 microns. In this zone, as described in Chapter 5, each grain is assigned an orientation within $\pm 15^\circ$ of its neighboring grains. This zone is surrounded by the ISV

material. The finite element model, with its boundary conditions, is shown in Figure 6-1b.

Figure 6-1: (a) Schematic of specimen dimensions for a CT12.5 half specimen, where the dimensions are in mm. (b) Crack growth model consisting of a half CT12.5 specimen linked to a rigid plane.

The bottom left corner of both the CT specimen and rigid plane are fixed in the x and y directions and rotation is not allowed in the z direction. The rigid plane is fixed in the x and y directions and rotation is not allowed in the z direction. The grain boundary crack path is modeled as a straight slab as the interface between the CT specimen and the rigid plane. This interface is modeled as a node to node contact using the UINTER subroutine (see Appendix C for description of codes) to describe the interface material. Force controlled loading is applied on the pin hole of the specimen at the desired ramp time and hold time condition. This force is applied using the UTRACLOAD subroutine (see Appendix C for description of codes). The maximum and minimum force applied is chosen such that a certain ΔK or $K_{\max}$ is achieved. The crack tip is located 2 grains within the crystal plasticity zone. The meshing details are shown in Figure 6-2. There are a total of 3601 plane strain elements. These are four and three node bilinear type elements (CPE4 and CPE3). The element size is approximately 5 microns to 1.5 mm in length away from the crack path. Along the grain boundary crack path in the grains immediately surrounding the crack tip, the element length is 2 microns in length.

Figure 6-2: Details of the meshing for the crack growth simulation.

The model formulations described in the previous two chapters are implemented into a cohesive zone model using an Abaqus finite element platform [1]. A summary of the equations used to model the grain boundary interface are given below:

$T_n = k_n (u_n)$; $T_t = k_t (u_t - u_s)$; traction-displacement relationships

$\dot{u}_s = w_c \frac{D_{gb} b E_{GB}}{\lambda k T} \left\langle \frac{|T_t - T_{th}|}{E_{GB}} \right\rangle^p$; grain boundary sliding displacement rate

$\varepsilon_{crit} = \varepsilon_{crit0} - z/M_{O_2}$; grain boundary critical strain as affected by Oxygen diffusion

$M_{O_2} = \frac{D_{O_2} \Omega}{k T b^2 C_{O_2}}$; mobility in presence of Oxygen

$D_{O_2} = D_0 e^{\frac{Q}{RT}}$; grain boundary Oxygen diffusivity

$C_{O_2} = C_0 + (C_s - C_0) \left[1 - \text{erf} \left(\frac{r}{2\sqrt{D_{O_2} t}} \right) \right]$; Oxygen concentration

The material constants for the grain boundary interface model are given in Table 6-1.

Table 6-1: Grain boundary interface material constants for M1-ME3 at 650°C, 704°C and 760°C.

Parameter	650°C	704°C	760°C	Units
k_n	2.32E+07	2.20E+07	2.00E+07	MPa/mm
k_t	9.25E+06	8.77E+06	8.00E+06	MPa/mm
D_{gb}	9.86E-08	4.71E-07	2.00E-06	mm ² /sec
E_{gb}	2.07E+12	1.97E+12	1.80E+12	MPa
T_{th}	0	0	0	MPa/mm
Ω	3.27E-20	3.27E-20	3.27E-20	mm ³
w_c	2.00E-03	2.00E-03	2.00E-03	mm
k	1.38E-20	1.38E-20	1.38E-20	mm ³
p	2	2	2	
λ	3.00E-07	3.00E-07	3.00E-07	mm
b	4.52E-07	4.52E-07	4.52E-07	mm
D_0	2.00E+01	2.00E+01	2.00E+01	mm ² /sec
R	8.31E-03	8.31E-03	8.31E-03	kJ/mol·K
C_0	0	0	0	%
C_s	21	21	21	%

The additional parameters required for the fracture criterion, ϵ_{crit} , will be discussed in detail in the next section. The basic interactions of the model components are outlined in the flow chart in Figure 6-3. In this, the total displacements across the grain boundary are dictated by the stress field surrounding the crack tip. This field is described by the multi-scale continuum model through a UMAT subroutine. The total displacements across the grain boundary are used to calculate the grain boundary tractions in the normal and tangential directions (UINTER subroutine). The tangential traction is then used to calculate the grain boundary sliding rate. The resultant traction is used to update the fracture criterion as a function of temperature and diffusion of Oxygen. Then the sliding strain is compared to the critical strain to check for failure in order to advance the crack tip. This sequence is repeated until fracture occurs.

Figure 6-3: Flow chart for the crack growth simulation using a cohesive zone model.

6.3 Grain Boundary Sliding Fracture Criterion

In order to predict the crack growth rate as a function of $K_{\max}$, a fracture criterion must be established. As previously described, in equation (4-53), fracture is assumed to occur when the grain boundary sliding displacement measured over a characteristic length, i.e. grain boundary sliding strain, reaches a critical limit and is considered to be a function of both temperature and environment. The constants

necessary to fully describe this criterion are determined through a series of simulation cases and experimental results.

Firstly, the critical displacement for grain boundary sliding in vacuum, u_{crit0} , is obtained as a function of temperature. The approach to determine this critical value is described here with reference to Figure 6-4 [2]. Here, it is assumed that at time $t = 0$, an element of material at a distance w_c ahead of the crack tip begins to experience grain boundary sliding. This length is equivalent to the critical slip band spacing estimated to be equal or larger than 3 microns which is required for the occurrence of grain boundary sliding in ME3 alloy. After time t , this distance reduces to $r = w_c - vt$, where v is the velocity of crack growth.

Figure 6-4: Cohesive zone crack tip at two different time positions as affected by the crack growth velocity, v .

During this time, the grain boundary sliding displacement (u_s) due to strain accumulation along the boundary is assumed to increase to:

$$u_s = \varepsilon_s w_c \quad (6-1)$$

where ε_s is the strain due to grain boundary sliding which could also be calculated by the integration of the grain boundary sliding strain rate as:

$$\varepsilon_s = \int_0^{w_c/v} \dot{\varepsilon}_s(w_c - vt) dt = \frac{1}{v} \int_0^{w_c} \dot{\varepsilon}_s(r) dr \quad (6-2)$$

Assuming that grain boundary fracture occurs when the grain boundary sliding displacement, u_s , becomes equal to a critical distance, u_{crit0} . This distance is obtained by combining the two above equations as:

$$u_{crit0} = \varepsilon_s w_c = \frac{w_c}{v} \int_0^{w_c} \dot{\varepsilon}_s(r) dr \quad (6-3)$$

This can also be written as:

$$u_{crit0} = \frac{w_c}{v} (\bar{\varepsilon}_s w_c) \quad (6-4)$$

where $\bar{\varepsilon}_s$ is the average grain boundary sliding strain over the distance w_c . Equation (6-4) can be rearranged to provide the time corresponding to the critical strain or displacement in vacuum environment, written as:

$$t_{break} = w_c / v \quad (6-5)$$

where w_c is the element length at the crack tip, in this case 2 μm and v is experimentally obtained the velocity of crack growth process at a given K_{max} and temperature condition. (Note this w_c is equivalent to w_c calculated in Chapter 3. In this chapter it is calculated as 3 μm , but here it is implemented in the simulation as 2 μm in order to increase the speed of the simulation). The above equation could then be estimated experimentally. For a similar K_{max} and temperature condition, a simulation is carried out, and the sliding displacement, u_s , at time t_{break} corresponds to the critical sliding displacement, u_{crit0} , at which fracture occurs. This operation is carried out for

three different $K_{\max}$ values at the two temperatures 704 and 760°C and the corresponding u_{crit0} values are shown in Figure 6-5.

Figure 6-5: Evolution of grain boundary sliding displacement as a function of time with corresponding critical displacement values at failure for (a) 704°C and (b) 760°C under creep crack growth conditions in a vacuum environment.

By invoking the assumption that critical sliding displacement is independent of $K_{\max}$, an average value of u_{crit0} is estimated (represented by the horizontal lines in Figure 6-5) for each temperature. These average values normalized by element size ($u_{crit0}/2\mu m$) are plotted in Figure 6-6 as a function of temperature and are fit into a closed form solution for the critical grain boundary sliding strain in vacuum, which is given as:

$$\varepsilon_{crit0} = \frac{u_{crit0}}{w_c} = A' e^{b'T} \quad (6-6)$$

where the constants A' and b' are 9.25e-5 and 7.7e-3 respectively, and T is the absolute temperature. This analysis has led to the determination of the critical grain boundary sliding strain for ME3 in vacuum environment.

Figure 6-6: Critical grain boundary sliding strain in vacuum, ϵ_{crit0} , as a function of temperature as described by equation (6-6).

As discussed previously, the influence of environment on the grain boundary sliding limit in an air environment is introduced by considering the mobility of Oxygen, M_{O2} , in the grain boundary. This type of interaction is described by equation (4-53). The factor z/M_{O2} addresses the complex stress-environment interactions stemming from the fact that for a hold time condition, the crack tip stress relaxation is accompanied with both a decrease of the diffusivity of Oxygen in the grain boundary, D_{gbO2} , and an increase in the concentration of Oxygen, C_{O2} . This combination of effects will result in a decrease in the mobility leading to accelerated failure of the cohesive zone element.

The temperature dependent parameter z can then be determined by rearranging equation (4-53) to the form:

$$z = (\epsilon_{crit0} - \epsilon_{critA}) M_{O2} \quad (6-7)$$

where ϵ_{critA} and M_{O2} are the critical sliding strain and dislocation mobility in the presence of Oxygen (in an air environment) at time t_{break} , respectively. The time t_{break} is calculated by equation (6-5) using the experimental crack growth rate in air. Since

M_{O2} is stress dependent, an average value of z is approximated for the different K_{max} simulations. These average values of z are plotted in Figure 6-7 as a function of temperature and are fit to a closed form solution:

$$z = z_1 + z_2 e^{z_3 T} \quad (6-8)$$

where $z_1 = -0.112$, $z_2 = 2.8e-6$, and $z_3 = 0.012$ and T is temperature in Kelvin.

Figure 6-7: Material parameter, z , as a function of temperature as described by equation (6-8).

Equations (6-6) and (6-8) are combined with equation (4-53) to fully define the critical sliding strain as a function of both temperature and environment. This limit is written as:

$$\epsilon_{crit} = \epsilon_{crit0} - \frac{z}{M_{O2}} = \left(A' e^{b'T} \right) - \frac{\left(z_1 + z_2 e^{z_3 T} \right)}{M_{O2}} \quad (6-9)$$

Integrating the above described criterion into the grain deformation model provides a tool to determine the time for the crack tip to advance the characteristic length (w_c , the cohesive element size) which, in turn, provides the crack growth rate under the prescribed loading and environment conditions.

extension, U_2 . In addition, environmental variables such as Q_{O_2} , D_{O_2} and C_{O_2} are resultants of the grain boundary tractions and displacements, thus act on the grain boundary interface. These various components will be presented and discussed in the following sections.

Case studies performed at 760°C with a loading frequency of 1.5s-30s-1.5s, at different K_{max} conditions, are presented in this section. The general trends of traction and displacement components are also discussed. During the loading-unloading portions of the cycle, the global force increases as a function of time, reaching a maximum load, P_{max} , followed by a decrease in load to a minimum force, P_{min} . During the hold at P_{max} , the local stress field at the crack tip relaxes. These events directly affect the normal and tangential displacements across the grain boundary interface, as shown in Figure 6-9. Figure 6-9a shows the normal displacement as a function of time is highest when the global force is at P_{max} , with an overall decreasing trend. Figure 6-9b shows that the tangential displacement as a function of time has a general increasing trend with drops corresponding to the load reversals. Furthermore, the normal and tangential displacements are larger with higher K_{max} values.

Figure 6-9: (a) Normal and (b) tangential total displacements across the grain boundary as a function of time for M1-ME3 at different K_{max} values with a loading frequency of 1.5s-30s-1.5s.

Since the normal traction-displacement relationship is linear in nature, the normal traction as a function of time has a similar trend to Figure 6-9a, as shown in Figure 6-10a. The tangential traction as a function of time is shown in Figure 6-10b. Although u_t increases with time, this figure shows that T_t decreases with time. Contrary to the normal traction-displacement relationship, the tangential traction-displacement law is non-linear. The tangential traction is linear with the elastic displacement, where the elastic displacement is the difference between the total and inelastic displacement components. The inelastic displacement, described by the grain boundary sliding displacement, u_s , has an increasing trend with time and $K_{\max}$, as shown in Figure 6-11. The increase in grain boundary sliding enhances the relaxation effect in the tangential direction. This is why the rate of relaxation in the tangential traction is higher than that in the normal direction.

Figure 6-10: Grain boundary (a) normal and (b) tangential traction as a function of time for M1-ME3 for different $K_{\max}$ values at a loading frequency of 1.5s-30s-1.5s (the time starts after the loading portion of the cycle, at 2 seconds).

Figure 6-11: Grain boundary sliding displacement as a function of time for M1-ME3 at different $K_{\max}$ values with a loading frequency of 1.5s-30s-1.5s.

Similarly, case studies performed at temperatures of 650, 704 and 760°C under creep crack growth conditions (i.e. a loading frequency of 1.5s-hold), at different $K_{\max}$ values, are presented in this section. The general trends of traction and displacement components are discussed, as well as, the influence of temperature.

The u_n , u_t and u_s , as a function of time for the three temperatures studied, at a constant $K_{\max}$, are shown in Figure 6-12. This figure shows results similar to the 1.5s-30s-1.5s loading presented above, where u_n decreases, while u_t and u_s increase with the increase in time and temperature. Figure 6-12a shows that the normal displacement is approximately the same at 704 and 760°C and larger at 650°C. This trend controls the relaxation of the normal traction, as shown in Figure 6-13a. In this, the 704 and 760°C T_n values differ since the stiffness, k_n , is lower with the increase in temperature. Again, the normal traction - displacement relationship has a purely elastic relation (i.e. no inelastic cavitation component). Thus, the relaxation in the normal stress direction is due to the relaxation of the surrounding continuum as a function of hold time, this is more apparent at higher temperature where the plastic zone size is

larger. The plastic zone size is proportional to $K_{\max}$ and inversely proportional to stress. For a constant $K_{\max}$, at the peak of the loading cycle, the stress at the crack tip is proportional to the yield strength. Since, yield decreases with temperature, the plastic zone size increases with temperature, as shown in Figure 6-14 at a time of 1.6 sec. With the addition of time or creep, the stress at the crack tip relaxes and the plastic zone size increases as a function of time. Figure 6-14 shows that for a constant temperature, the zone of the stress field increases as a function of time during creep.

Figures 6-12b and 6-12c show that the total (u_t) and inelastic (u_s) tangential displacement components are of similar magnitude. Each as a function of time show a rapid increase followed by saturation. Figure 6-12c emphasizes the fact that the sliding displacement increases rapidly with the increase in temperature. At 1000 seconds, the sliding displacement at 704 and 760°C are 2 and 3.5 times higher than that at 650°C. This behavior can be attributed to the rapid relaxation in tangential traction with the increase in temperature, as seen in Figure 6-13b. Initially the tangential traction is higher at 760°C, but the rapid decline in traction is enhanced by the large sliding displacement. As the sliding displacement becomes comparable in value to the total tangential displacement, the elastic component vanishes and the tangential traction approaches zero as time increases.

In addition to the interface stresses and displacements, the element components, S_{22} and U_2 , were examined at 760°C under creep crack growth conditions. These are shown in Figure 6-15 as a function of time. Figure 6-15a shows that the stress in the continuum decreases with time, similar to the trend of T_n versus time in Figure 6-13a. This is accompanied by an increase in plastic zone size, strain

and element extension of the continuum material surrounding the crack tip. The element extension in the normal direction as a function of time is shown in Figure 6-13b.

Figure 6-12: (a) Normal and (b) tangential total displacements, and (c) sliding displacement across the grain boundary as a function of time and temperature for M1-ME3 at a K_{max} of 55 MPa $\sqrt{m}$ under creep crack growth conditions (the time starts after the loading portion of the cycle, at 2 seconds).

Figure 6-13: Grain boundary (a) normal and (b) tangential traction as a function of time and temperature for M1-ME3 under creep crack growth conditions at a K_{max} of 55 MPa $\sqrt{m}$ (the time starts after the loading portion of the cycle, at 2 seconds).

Figure 6-14: Normal stress (S_{22}) field as a function of time and temperature for M1-ME3 under creep crack growth conditions at a $K_{\max}$ of $55 \text{ MPa}\sqrt{\text{m}}$.

Figure 6-15: (a) Stress and (b) element extension in the normal direction as a function of time at 760°C for M1-ME3 under creep crack growth conditions.

Crack Growth Rate

Results obtained from the simulation of crack growth without environment effects are validated by comparing with the crack growth experiments performed in vacuum. The effect of environment is nullified by putting the concentration of Oxygen

at the surface (C_s) equal to zero and the fracture limit is defined solely by ϵ_{crit0} . Figure 6-16a (704°C Vac) and Figure 6-16b (760°C Vac) show the simulated curves compared with crack growth experiments in vacuum. The numerical crack growth curves for 704°C and 760°C in vacuum are shown to follow a trend similar to that of the experimental data. The slopes of the simulated set expressing a higher power. The simulated data show under prediction of the crack growth rates at lower K_{max} values, while the opposite is observed at high K_{max} . This is possibly due to the stress sensitivity of the grain boundary sliding rate to the stress exponent which is used in the calculation of grain boundary sliding displacement.

Figure 6-16: Experimental and numerical crack growth rate as a function of K_{max} at (a) 650°C and 704°C and (b) 760°C and 800°C, in both air and vacuum environments. Note that the symbols are the experimental data points and the lines represent the numerically obtained crack growth rate from the cohesive zone simulation.

In addition to the 650-760°C temperature range, the crack growth process was also examined at 800°C in an air environment. The experimental crack growth rate at this temperature is similar to that at 760°C. The crack growth rates for temperatures ranging from 650 to 800°C in air environment have been predicted and the results are shown in Figure 6-16. The numerical results of the four temperatures studied are in agreement with the range and trends of the experimental crack growth data. Similar to the vacuum results, the numerically simulated crack growth rate curves in air can be represented by a power law form. However, in contrast to vacuum results, air simulated curves show a slightly lower slope than that obtained in experimental curves. This is due to the difficulty in determining the frequency term of the Oxygen diffusivity function. The low $K_{\max}$ region can be identified as a more temperature dependent region, with an increased effect of environment. The impact of both temperature and environment reduces in the upper crack growth region.

In summary, from the above discussion, the crack growth rate depends upon interplay of three external parameters which are later correlated in order to define the material intrinsic properties. The three parameters are stress, temperature and environment. The stress and temperature control the creep deformation rate, thermal and athermal energy contributions. In lower stress regions, the creep deformation is highly dependent on temperature, i.e. in sense of energy balance; fracture energy is mostly provided by the input thermal energy to the system. At the higher stress region, the creep damage is more stress dependent and the time to fail becomes almost the same for all three temperatures. The presence of creep deformation in the crack tip has two opposite effects; damage leading to failure as a detrimental effect and stress

relaxation in the crack tip which has a beneficial effect by reducing the crack tip driving force which would ultimately result in reduction of crack growth rate. This beneficial effect is not present in the ME3 alloy as a reduction of crack growth rate and is not observed at higher temperature.

The role of environment increases the damage processes by embrittling the grain boundary, which is seen to be more active at the lower stress region. The environmental rate is characterized by the effect of concentration of Oxygen, which is related to the diffusion process along the grain boundary, being a dependent function of stress, temperature and time. The complex interplay of these parameters, in addition to the roles of apparent energy of activation, diffusivity and Oxygen concentration are discussed in the following section.

Activation Energy for Oxygen Diffusion

As discussed previously, the role of environment is viewed as a dynamic embrittlement process. This can be described by the diffusivity of Oxygen in the grain boundary and the concentration of Oxygen in the grain boundary. In this study, the $K_{\max}$ dependency of activation energy was first obtained from the experimental crack growth data in air as described in Chapter 2 as:

$$Q_{O_2} = 224.2 - 1.6(K_{\max}) + 7.39E-03(K_{\max})^2 \quad (6-10)$$

This form is similar to that developed by Zheng [3] in which the stress dependent term is the inelastic strain energy density. In order to incorporate Q_{O_2} with cohesive zone parameters, equation (6-10) has been written in terms of equivalent traction, T_{tot} , across the grain boundary as:

$$Q_{O_2} = 226.2 - 0.05(T_{tot}) - 1.26E - 06(T_{tot})^2 \quad (6-11)$$

$$T_{tot} = \sqrt{\frac{1}{2}T_n^2 + \frac{1}{2}T_t^2} \quad (6-12)$$

During time dependent crack growth, the stress increases during the loading portion and relaxes during the hold time. Thus, during the hold time, stress decreases and Q_{O_2} increases, as shown in Figure 6-17. This figure shows that at the start of the hold time, Q_{O_2} is less than the stress free activation energy, indicating that during the loading portion of the cycle, the Q_{O_2} decreases. Once the maximum traction is achieved (i.e. at the start of the hold), as the stress field relaxes at the crack tip, the activation energy increases. More energy is required to move Oxygen atoms into the grain boundary when a lower stress or lower driving force is present; hence activation energy is lower with an increase in stress intensity. In addition, the activation energy increases with temperature. To further examine this fact, the activation energy at 2 seconds and 300 seconds for K_{max} of 40 and 60 MPa $\sqrt{m}$ for the three temperature conditions are summarized in Figure 6-18.

Figure 6-17: Evolution of grain boundary activation energy due to Oxygen as a function of time for (a) 650°C, (b) 704°C and (c) 760°C for M1-ME3 under creep crack growth conditions (the time starts after the loading portion of the cycle, at 2 seconds).

Figure 6-18: Bar chart showing grain boundary activation energy due to Oxygen at two time and K_{max} conditions for 650°C, 704°C and 760°C.

Figure 6-18 shows that the variation in energy from time 2 sec to 300 sec is enhanced with temperature and higher K_{max} . This is due to that fact that the traction across the grain boundary is higher with lower temperature, thus the diffusion of Oxygen has a

higher stress assistance requiring lower activation energy. At lower $K_{\max}$, the amount of relaxation is less than that at higher $K_{\max}$, this explains the fact that there is a smaller variation in Q_{O_2} as a function of time for at 40 MPa $\sqrt{m}$.

Diffusivity of Oxygen in the Grain Boundary

The diffusivity of Oxygen in the grain boundary is a function of the frequency factor, temperature and activation energy. The diffusivity increases with a decrease in activation energy (i.e. increase in stress) or increase in temperature. Since Q_{O_2} increases during the hold time, D_{O_2} decreases, as shown in Figure 6-19 as a function of time for the three different temperatures. Similarly, the trends for Q_{O_2} explain the variations in D_{O_2} as a function of time, with the knowledge that D_{O_2} increases with a decrease in Q_{O_2} and increase in temperature.

Figure 6-19: Evolution of grain boundary diffusivity due to Oxygen as a function of time for (a) 650°C, (b) 704°C and (c) 760°C for M1-ME3 under creep crack growth conditions (the time starts after the loading portion of the cycle, at 2 seconds).

Evolution of Crack Tip Oxygen Concentration

The Oxygen concentration in the bulk material, C_{O_2} , increases with an increase in time and diffusivity, D_{O_2} . Since D_{O_2} and the rate of D_{O_2} decrease with time, the concentration will increase more rapidly at lower times. The concentration as a function of time, at a distance of 2 μm from the crack tip, is shown in Figure 6-20, which indicates that the concentration of Oxygen as a function of time is higher with K_{max} and temperature. The concentration of oxygen at 300 seconds for K_{max} of 40 and 60 $\text{MPa}\sqrt{\text{m}}$ for the three temperature conditions are summarized in Figure 6-21. This figure give the enhanced evolution patterns of Oxygen concentration as a function of temperature and K_{max} .

Figure 6-20: Evolution of Oxygen concentration in the grain boundary as a function of time for (a) 650°C, (b) 704°C and (c) 760°C for M1-ME3 under creep crack growth conditions.

Figure 6-21: Bar chart showing the concentration of Oxygen in the grain boundary at 300 sec at two $K_{\max}$ conditions for 650°C, 704°C and 760°C.

Figure 6-21 shows that the variation in concentration with temperature is less significant at higher $K_{\max}$. At 40 MPa√m, the concentration at 704 and 760°C are 2.2 and 3.4 times higher than that at 650°C. At 60 MPa√m, the concentration at 704 and 760°C are 1.1 and 1.5 times higher than that at 650°C. This implies that environment plays a larger role at lower stress, thus higher stress may be dominated more by the grain boundary sliding process. As previously discussed, the diffusion of Oxygen in the grain boundary is assumed to affect the fracture limit in an air environment.

6.4.2 Model Parameter Analysis – Oxygen Partial Pressure

The partial pressure of Oxygen has been studied by varying the surface concentration of oxygen, C_s , from vacuum conditions ($C_s = 0\%$) to that of air environment ($C_s = 21\%$). The difference between the surface concentration of Oxygen and the concentration of Oxygen in the bulk material, $C_s - C_0$, controls the Oxygen flux and its reduction decreases the diffusion of Oxygen in the grain boundary. The

Oxygen concentration at a depth of 2 μm , i.e. the size of the cohesive element, as a function of time for a C_s of 0, 5, 10, 15 and 21% has been calculated and is shown in Figure 6-22 for the three temperatures. This shows that the concentration of Oxygen in the grain boundary increases rapidly in the early part of the diffusion process followed by a trend towards saturation. Temperature is also an influential parameter on Oxygen concentration, its role can be illustrated by observing, for example, that for $C_s=10\%$, the time to reach a concentration of 4% is 665s, 335s, 155s at 650, 704 and 760°C; respectively. The concentration of Oxygen in the grain boundary controls the critical sliding strain which is calculated as a function of the grain boundary mobility, M_{O_2} , which is proportional to T and C_{O_2} . The effect of C_s on the critical sliding strain at a K_{max} of 55 $\text{MPa}\sqrt{\text{m}}$ is shown in Figure 6-23. This figure illustrates the time to failure as the intersection of the critical sliding strain with the developed grain boundary sliding strain at each of the temperature conditions. Results of this figure are summarized in Figure 6-24 which shows that the time to fail increases with the reduction of both temperature and surface concentration of Oxygen, i.e. reducing the partial pressure of oxygen from air to vacuum.

Figure 6-22: Evolution of Oxygen concentration in the grain boundary as a function of time for (a) 650°C, (b) 704°C and (c) 760°C for M1-ME3 under creep crack growth conditions at a K_{max} of 55 MPa√m and environments varying from air ($C_s = 21\%$) to vacuum ($C_s = 0\%$). Note the legend indicates the value of C_s .

Figure 6-23: Grain boundary sliding strain as a function of time for (a) 650°C, (b) 704°C and (c) 760°C for M1-ME3 under creep crack growth conditions at a K_{max} of 55 MPa√m. The dotted lines indicate the critical sliding strain as a function of time for environments varying from air ($C_s = 21\%$) to vacuum ($C_s = 0\%$).

Figure 6-24: Time to fail as a function of surface concentration of Oxygen for M1-ME3 at 650°C, 704°C and 760°C at a $K_{\max}$ of 55 MPa $\sqrt{\text{m}}$.

Relative Significance of Temperature and Environment

The influence of temperature alone on the critical sliding displacement is shown in Figure 6-6, illustrating that ε_{crit0} increases with temperature. Thus, the grain boundary can accommodate more sliding at higher temperatures. The addition of environment degrades this value, as shown in Figure 6-23. The relative influence of both temperature and environment can be examined by plotting the ε_{crit} as a function of C_s , as shown in Figure 6-25.

Figure 6-25: Critical grain boundary sliding strain, ε_{crit} , as a function of Oxygen partial pressure in terms of the parameter C_s for 650°C, 704°C and 760°C at a $K_{\max}$ of 55 MPa $\sqrt{\text{m}}$.

This figure shows that the percent of degradation of the critical grain boundary sliding strain in air compared to that in vacuum is 50%, 40% and 28% for the temperatures 650, 704 and 760°C respectively. It is interesting, however, to observe that the influence of C_s on the critical sliding strain is relatively higher at 650°C than the other two higher temperatures. The da/dt at 650°C is lower than da/dt at 704 or 760°C, thus, for the same K_{max} and the same crack length increment, it will take a longer time to reach failure at 650°C. This additional time will allow the diffusion of oxygen to play a more significant role on the ϵ_{crit} . Similarly, environment will have a more dominant role at lower K_{max} values as opposed to higher K_{max} values, where the time to failure is longer. These results should be considered in attempts to engineer the grain boundary in a manner to increase resistance to environment.

6.4.3 Model Parameter Analysis – Effects of Grain Boundary Diffusivity and Modulus

The role of grain boundary viscosity, η , is presented here by modifying either the grain boundary diffusivity, D_{gb} , or grain boundary modulus, i.e. the unrelaxed shear modulus, E_{gb} . By decreasing D_{gb} , the intrinsic dislocation mobility, described by equation (4-40) is lowered. In turn, this would increase the η and increase the resistance of the grain boundary to sliding. Increasing the stiffness of the grain boundary, described by E_{gb} , would also increase η . By decreasing the D_{gb} by 50% or

increasing the E_{gb} , by a factor of 2, the grain boundary viscosity is changed by a factor of 2, from η_1 to $2\eta_1$. For these two cases, the grain boundary viscosities are:

$$\eta_1 = \frac{E_{GB}}{D_{gb}} \frac{\delta \lambda k T}{w_c T_t b}; \quad \eta_2 = \frac{E_{GB}}{0.5 D_{gb}} \frac{\delta \lambda k T}{w_c T_t b} = 2 \frac{E_{GB}}{D_{gb}} \frac{\delta \lambda k T}{w_c T_t b} = 2\eta_1 \quad (6-13)$$

This increase in η , is shown in Figure 6-26 to reduce the sliding rate and sliding displacement. This reduction would result in a decrease in crack growth rate.

Figure 6-26: Grain boundary sliding displacement as a function of time and K_{max} for two grain boundary viscosities, η_1 (dashed line) and $2\eta_1$ (solid line), at (a) 650°C, (b) 704°C and (c) 760°C for M1-ME3 under sustained load crack growth conditions [4].

The decrease of u_s is high at higher loading conditions compared to the observed decrease at lower loading conditions for all temperatures. This non linear stress dependency of sliding causes a higher crack growth rate decrease at higher

stresses, i.e. the difference in crack growth rate from 35 to 45 MPa√m is lower than that from 65 to 75 MPa√m.

References

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CHAPTER 7

INFLUENCE OF MICROSTRUCTURE ON INTERGRANULAR CRACKING

7.1 Introduction

The last part of this study has performed a sensitivity analysis of the cohesive zone model. In this, the grain boundary sliding is controlled through modification of the continuum material properties or the intrinsic grain boundary viscosity, as illustrated in Figure 7-1.

Figure 7-1: Schematic of the cohesive zone model components and the influence of γ' precipitates and grain boundary serrations.

Modification of the continuum material is examined through incorporating the γ' particle size and volume fraction explicitly in the hardening laws of the ISV and XP material models, this is addressed in Section 7.2 of this chapter. The stress field surrounding the crack tip is modified through control of the γ' size and/or volume fraction. This stress field will dictate the total normal and tangential displacement components across the grain boundary and in turn alter the traction and grain boundary sliding.

Secondly, Section 7.3 addresses the effect of grain boundary morphology on grain boundary sliding is examined by incorporating grain boundary serrations. The grain boundary serration height and wavelength are integrated by modifying the grain boundary viscosity. The grain boundary viscosity is directly related to the intergranular cracking resistance.

7.2 Effect of Precipitate Size and Volume Fraction

The continuum deformation behavior of Nickel-based superalloys is complex and is dependent on a number of factors. Some of these factors include microstructure (γ' size, shape, volume fraction, channel width, distribution), orientation relative to the loading direction, stress and temperature levels. These factors will determine the active deformation mechanisms under the current conditions. The basic strengthening mechanisms in Nickel-based superalloys, which will be discussed in Section 7.2.1, describe the interactions between dislocations and various obstacles. These obstacles include other dislocations, solute atoms, grain boundaries and precipitates. In addition,

the active deformation modes in IN100 and ME3 alloys will be discussed. Following this, Section 7.2.2 will describe how the deformation mechanisms are incorporated into a microstructure explicit continuum deformation model, incorporating γ' size and volume fraction, are described and implemented.

7.2.1 Strengthening Mechanisms in Nickel-Based Superalloys

There are many factors that act as strengthening mechanisms in Nickel-based superalloys. Strengthening arises from the interaction of a dislocation with an obstacle. Major obstacles and their strengthening effects, which are discussed in the remainder of this section, can be summarized as follows;

- (1) *Work hardening*: Plastic deformation will cause an increase in dislocation density, thus, promoting the interaction of dislocations with other dislocations.
- (2) *Solid solution strengthening*: When a dislocation travels through a matrix phase, its motion will be disrupted by the concentration of solute atoms residing in the matrix material. The solute atom will have a corresponding stress field associated with its atomic size and strength mismatch relative to the matrix material.
- (3) *Grain boundary strengthening*: A stress build-up is required for a dislocation traveling in one grain to cross a grain boundary. In addition, the neighboring grains have a misorientation, thus, the dislocation must also change direction.
- (4) *Precipitation hardening*: The interaction between a dislocation and particle is dependent on many factors. The two basic mechanisms are shearing, in which

the dislocation passes through the particle and Orowan by-passing, in which the dislocation loops the particle.

7.2.1.1 Work Hardening

Work hardening or strain hardening describes the interactions between dislocations. As plastic deformation occurs, plastic strain will build up and dislocation density will increase, this will increase the probability of interactions of dislocations with other dislocations and as a result an increase in flow stress. Assuming that the average distance between dislocations is proportional to $\rho^{-1/2}$ and the stress field associated with the interaction between two obstacles is proportional to 1/distance between them, then the increase in flow stress can be described by the Taylor equation [1, 2]:

$$\tau = \alpha G b \rho^{1/2} \quad (7-1)$$

where α is a material constant, G is shear modulus, b is Burgers vector and ρ is dislocation density. The hardening is controlled by the competition between storage and annihilation of dislocations. The basic law which describes the evolution of dislocation density is given as [3, 4]:

$$\dot{\rho} = (k_1 \rho^{1/2} - k_2 \rho + k_3) \dot{\gamma} \quad (7-2)$$

where k_1 , k_2 and k_3 are material constants and γ is plastic shear strain. The three terms in equation (7-2) describe the generation of statistically stored dislocations, dynamic recovery (annihilation and rearrangement) of statistically stored dislocations and production of geometrically necessary dislocations; respectively. The constant k_1

relates to the distribution of forest dislocations [5]. Feaugas and Haddou express the constant k_2 as the product of annihilation distance and probability of the annihilation event [5]. Cheng et al describe this constant as the product of two competing processes, k_s and F [3]; (1) k_s is proportional to the solute content, thus, would decrease when precipitates are formed. (2) F is a factor which increases when precipitates are formed, thus, k_2 would increase when precipitates (shearable or nonshearable) are formed and the rate of dynamic recovery would decrease. Cheng et al make an assumption that the effect of depletion of solid solution and increase in precipitate fraction would cancel each other and product $k_s F$ is constant. The constant k_3 defines a length path. This constant is inversely proportional to grain size or mean free path between (nonshearable) particles [3-5]. This constant is not effected by shearable precipitates [3]. Internal stresses arise from this increase in dislocation density, thus, an increase in work hardening.

7.2.1.2 Solid Solution Strengthening

The interaction between dislocations and solute atoms, distributed randomly in a matrix material, will give rise to solid solution strengthening. Solute atoms reside in the matrix either as interstitial or substitutional atoms. The strengthening contribution of a solute is dependent on its concentration, atomic size and strength relative to the matrix atoms [6]. This mismatch in geometry and/or properties will modify the mobility of dislocations moving through the matrix lattice.

Based on an approach given by Gypen and Deruyttere [6, 7], the hardening stress increases as a function of solute concentration and follows the form [9, 10]:

$$\tau_{ss} = \frac{1}{M} \sum_i \frac{d\sigma}{d\sqrt{C_i}} \sqrt{C_i} \quad (7-3)$$

where M is the Taylor factor, C is concentration of solute i and $d\sigma/d\sqrt{C_i}$ is the strengthening coefficient of solute i . Roth et al [10] have determined the strengthening coefficients for various elements based on the work of Mishima et al [11] in which they studied binary Nickel alloys with varying atomic percents of solutes. Table 7-1 shows the strengthening coefficients for the elements which contribute to strengthening of the gamma matrix, along with the chemical composition of the gamma matrix for IN100 alloy.

Table 7-1: Strengthening coefficient [10] and chemical composition [9] of the gamma matrix in IN100.

		Ni	Al	Ti	Co	Cr	Mo	V
Strengthening Coefficient	MPa $\sqrt{\text{frac}}$		225	775	39	337	1015	408
Chemical Composition of γ	wt %	Bal	2.25	0.93	27.8	24.5	5.73	0.05

This value was measured at 0.2% flow stress at 77K by Mishima and coworkers [11]. This coefficient describes the strain induced in the lattice due to adding the impurity.

This strengthening effect can occur by a number of mechanisms including elastic, modulus, stacking-fault, electrical, short-range order and/or long-range order interactions [12]. The strengthening due to elastic interactions is directly proportional to the misfit of the solute atom. Strengthening which is due to modulus interactions

will modify the modulus of the material, which occurs if the solute atom has a different shear modulus than the matrix atom. Stacking-fault interactions occur when the solute atom preferentially segregates to stacking-faults. This increase in concentration of solute atoms will lower the stacking-fault energy, thus modifying the mobility of dislocations through the matrix. Electrical interactions arise if the solute atom has a charge associated with it, which would interact with electrical dislocation dipoles. Short-range ordered regions are clusters of solute atoms, which have a lower degree of order. These types of regions will increase the stress required for a dislocation to pass through the region. Long-range ordered regions are a long-range periodic arrangement of dissimilar atoms or a superlattice. When a dislocation moves through a superlattice, the dislocation will dissociate into two ordinary dislocations separated by an anti-phase boundary. This will alter the motion of a dislocation through the region. One or more of these mechanisms can contribute to the magnitude of the strengthening coefficient in equation (7-3), thus, altering solid solution strengthening ability of a particular solute atom.

7.2.1.3 Grain Boundary Strengthening

Grain boundaries act as obstacles to slip, thus, grain size plays an important role in defining the yield strength of a material. Dislocations will pile-up at grain boundaries until the stress is large enough to break through the boundary. The initial critical resolved shear strength for bulk yielding of the material is related to grain size by the classical Hall-Petch relationship [13, 14]:

$$\tau_{CRSS} = \tau_0 + k_{HP} d_G^{-1/2} \quad (7-4)$$

where τ_0 is the frictional stress (i.e. Peierls stress), k_y is a material constant (i.e. Hall-Petch coefficient) and d_G is the grain size. This relationship shows that yield strength will increase with a decrease in grain size. The grain boundaries act as dislocation barriers for two main reasons [13]; (1) as a dislocation passes from one grain to another across a grain boundary, the two grains are of different crystallographic orientations and the dislocation will have to change its direction of motion. The higher the degree of misorientation between two grains, the more difficult it is for a dislocation to cross the grain boundary. (2) The atomic disorder along the grain boundary defines a discontinuity of slip planes between two grains, thus, making it difficult for a dislocation to pass.

7.2.1.4 Precipitation Hardening Mechanisms

Precipitation hardening mechanisms can occur as single or multiple mechanisms. The interactions between dislocations and precipitates are complex and are dependent on many material factors such as particle size, volume fraction, coherency, lattice misfit, matrix/precipitate modulus, stacking-fault energy or surface energy. The major hardening mechanisms are (1) chemical strengthening, (2) stacking-fault strengthening, (3) modulus misfit hardening, (4) coherency strengthening and (5) order strengthening. These mechanisms control the critical resolved shear stress required to shear a particle. When a particle cannot be sheared, i.e. the size is larger than a critical size or the particle is incoherent, the critical

resolved shear stress corresponds to that required to by-pass or loop a particle, thus, the hardening mechanism is (6) Orowan by-passing. A review of these mechanisms are given in references [6, 15-17] and they are summarized in the following sections.

Shearing Mechanisms

Chemical Strengthening: The creation of an additional surface area, as shown in Figure 7-2, when a particle is sheared by a dislocation leads to chemical or surface strengthening.

Figure 7-2: New surface (hatched area), of area $2\pi rb$, created when edge dislocation shears a particle [17].

Associated with this new interface is a surface energy γ_s , where the total increase in energy is the product of this energy with the increase in surface area, $2\pi rb\gamma_s$, where r is particle radius and b is the Burgers vector. The force required to move a dislocation through a particle is given by [16]:

$$F_{\max} = \frac{\pi rb\gamma_s}{b} = \pi r\gamma_s \quad (7-5)$$

This will result in an increase in yield stress, where a simplistic form is given as [16]:

$$\tau_{chem} = \frac{F_{\max}}{bL'} = \frac{\pi r\gamma_s}{bL'} = \frac{\pi f\gamma_s}{2b} \quad (7-6)$$

where L' is the effective obstacle spacing and is approximated by $2r/f$, assuming the dislocation segment remains straight. A more complex form is given by Reppich as [17]:

$$\tau_{chem} = 0.94 \left(\frac{3}{2} \right) \frac{f\gamma_s}{r} \left[\frac{2}{3} \left(\frac{6\gamma_s b}{\pi f T_L} \right)^{1/2} + 1 \right] \quad (7-7)$$

where T_L is the dislocation line tension in the matrix, approximated by $(Gb^2)/2$ [17]. According to this equation, for a given volume fraction, the critical resolved shear stress decreases with increasing particle size. Thus, maximum strength occurs when particles are extremely small, i.e. of atomic size [17]. In the case of the ME3 or IN100 alloys studied in this work, the smallest particle is greater than 1 nm in size, thus, chemical strengthening is minimal and is not considered in this work.

Stacking-Fault Strengthening: If the stacking-fault energies between the matrix and particle are very different, there is stacking-fault strengthening. The stacking-fault energy of a material describes the ability of a dislocation to glide [17]. The force required to move a dislocation is based on the particle size (d or $2r$), ribbon width of the split dislocation in the matrix (s_m) and particle (s_p) and stacking-fault energies in the matrix (γ_{sfm}) and particle (γ_{sfp}). If the stacking-fault energy in the matrix is greater than that in the particle, i.e. $\gamma_{sfm} > \gamma_{sfp}$, then the maximum force required to move the split dislocation is given as [15, 17]:

$$F_{max} = |\gamma_{sfm} - \gamma_{sfp}| l \quad (7-8)$$

where l is the length of the chord inside the particle at the breaking condition. Two conditions, as illustrated in Figure 7-3, will be considered, (1) when d is comparable to or less than s_m and (2) when d is greater than s_m .

Figure 7-3: (a) The split dislocation / particle configuration when d is comparable to or less than s_m and when d is greater than s_m . (b) The corresponding interaction force profiles over the interaction length γ_{SL} [17].

For condition (1), the chord length is equal to d , thus, the maximum force becomes:

$$F_{\max} = |\gamma_{sfm} - \gamma_{sfp}| d \quad (7-9)$$

In this case, the critical resolved shear stress increases with particle size and volume fraction and is given by the form [15, 17]:

$$\tau_{SF} = |\gamma_{sfm} - \gamma_{sfp}|^{3/2} \left(\frac{3\pi^2 f r}{32 T_L b^2} \right)^{1/2} \quad (7-10)$$

In this case, for a given volume fraction, the critical resolved shear stress increases with particle size. On the other hand, for condition (2), the chord length is less than d

and the maximum force has a magnitude less than that given in equation (7-9). The critical resolved shear stress will then take the form [17]:

$$\tau_{SF} = \left(\frac{2Kf}{\gamma_{sfm} + \gamma_{sfp}} \right)^{1/2} \frac{|\gamma_{sfm} - \gamma_{sfp}|}{br^{1/2}} \quad (7-11)$$

where K is a constant. For a given volume fraction, the critical resolved shear stress decreases with the increase in particle size.

Modulus Misfit Hardening: When the modulus of the matrix and precipitate differ, the energy of a dislocation segment will change when it enters a particle. The force required to move a dislocation through a particle is given by [16]:

$$F_{\max} = br(G_p - G_m) = G_m br \varepsilon_{\text{mod}} \quad (7-12)$$

where G_p and G_m are the particle and matrix shear modulus respectively. The modulus parameter is given by:

$$\varepsilon_{\text{mod}} = \frac{G_p - G_m}{G_m} \quad (7-13)$$

This will result in an increase in yield stress, where a simplistic form is given as [16]:

$$\tau_{\text{mod}} = \frac{F_{\max}}{bL'} = \frac{G_m br \varepsilon_{\text{mod}}}{bL'} = \frac{1}{2} G_m f \varepsilon_{\text{mod}} \quad (7-14)$$

A more complex form is given by Ardell as [15]:

$$\tau_{\text{mod}} = 0.9(rf)^{1/2} \frac{T_L}{b} (\varepsilon_{\text{mod}})^{3/2} \left[2b \ln(2r/f^{1/2}b) \right]^{-3/2} \quad (7-15)$$

The modulus mismatch in γ/γ' alloys is not extensive and in general is not considered as a hardening mechanism in these alloys [17].

Coherency Strengthening: Lattice misfit between a matrix and particle creates a stress field at the particle-matrix interface. This is known as coherency or size misfit hardening. The coherency misfit parameter is given by:

$$\varepsilon_{coh} = \frac{a_p - a_m}{a_m} \quad (7-16)$$

where a_p and a_m are the particle and matrix lattice parameters respectively. When the lattice parameter of the matrix is greater than that of the particle, the coherency strain is positive and the particle is in a state of tension, while for the opposite case, the particle is in a stage of compression. These states will vary the stress field surrounding the particle, which will lead to an attraction or repulsion of dislocations. Either case will increase the yield strength of the material, in addition, the particle size or volume fraction will increase the influence of the coherency strain hardening. This increase in yield is given as [16, 17]:

$$\tau_{coh} = \alpha |\varepsilon_{coh}|^{3/2} G \left(\frac{rf}{b} \right)^{1/2} \quad (7-17)$$

where α is a material constant varying between 3 and 4.1 [17].

Order Strengthening: The energy required to create an anti-phase boundary results in order strengthening. When a single dislocation shears a particle it creates an antiphase boundary (APB), as illustrated in Figure 7-4, which disrupts the A-B stacking sequence.

Figure 7-4: (a) An edge dislocation approaching a particle with A-B bonds, (b) partially through the particle and (c) completely shearing the particle. This process is accompanied by the formation of an antiphase boundary in which A-A and B-B bonds form across the slip plane [16].

The A-A and B-B bonds, which constitute the antiphase boundary, have a higher energy state than the original A-B bonds called antiphase boundary energy (γ_{APB}). This stacking sequence is unstable, thus, dislocations travel in pairs so that the second dislocation can pass behind the first to restore the original A-B bonds. The separation distance of the two dislocations depends on the ratio between the force that binds the dislocations (γ_{APB}) and the force that separates the dislocations (Gb), where G is the shear modulus and b is the Burgers vector [16]. When this ratio (γ_{APB} / Gb) is low, the dislocations travel separately or are widely spread, thus, they are weakly coupled dislocations and when this ratio is high, the dislocations travel closely, thus, they are strongly coupled dislocations. In general, this ratio is smaller for a smaller particle size. When the dislocation spacing is large in comparison to the particle size (weakly coupled pair), the second dislocation is far behind the leading dislocation leaving faulted particles between the two dislocations, as shown in Figure 7-5a. When the spacing between dislocation pairs is on the order of the particle size (strongly coupled pair), the second dislocation enters the particle before the first exits, as shown in Figure 7-5b.

Figure 7-5: Particles being sheared by (a) a weakly coupled dislocation pair and (b) strongly coupled dislocation pair. Where (1) is the leading dislocation, (2) is the trailing dislocation, τ is the applied shear stress, r_i is particle radius, λ_i is the distance between pinning points and i corresponds to 1 and 2 (the leading and trailing dislocation) [18].

The stress required to shear by weakly coupled dislocations is proportional to volume fraction, f and particle size, r . A simplistic form is given as [16]:

$$\tau_{weak} = 0.7G \left(\frac{\gamma_{APB}}{Gb} \right)^{3/2} \left(\frac{rf}{b} \right)^{1/2} \quad (7-18)$$

A more complex form is given as [19]:

$$\tau_{weak} = \frac{\gamma_{APB}}{2b} \left[A_1 \left(\frac{\gamma_{APB}rf}{T_L} \right)^{1/2} + A_2 f \right] (1 + 0.5f^{1/3}) \quad (7-19)$$

where A_1 and A_2 are material constants, $A_1 - A_2 = 1$, and the dislocation line tension, T_L , can be approximated by $Gb^2/2$ [20].

For particles with radius greater than the critical size, shearing takes place by strongly coupled dislocation pairs. This shear stress is proportional to f and inversely proportional to r as given by [19]:

$$\tau_{strong} = 0.39 \frac{T_L w f^{1/2}}{b r} \left[\frac{\pi^2}{4} \left(\frac{\gamma_{APB} r}{w T_L} \right) - 1 \right]^{1/2} (1 + 0.5 f^{1/2}) \quad (7-20)$$

where w is an adjustable parameter, accounting for elastic repulsion of the dislocation within the precipitate.

Orowan by-passing Mechanism

When a particle size is above a critical radius or the particle is incoherent with the γ matrix, the favorable mechanism is dislocation looping, i.e. Orowan by-passing. In this process, a dislocation will not shear the particle; instead it will bow around a particle. This is shown in 5 steps [12], as illustrated in Figure 7-6; (1) a straight dislocation approaches two particles of radius r and effective particle spacing $L-2r$. (2) The dislocation begins to bend and (3) it reaches a critical value of curvature. (4) Once the dislocation has reached its critical value of curvature, it will move forward and the segments of the dislocation on either side of the particle will meet. These segments are opposite in sign and will annihilate over a portion length and leave behind a dislocation loop. (5) Then the original dislocation is free from the particles and can continue to move through the material. Once the bypass mechanism occurs, a dislocation loop of radius r^* is left around the particles and reduces the effective particle spacing to $L-2r^*$ [16]. This introduces a back stress which must be overcome for further slip to occur.

Figure 7-6: A view from the slip plane of a dislocation approaching particles of radius r and effective particle spacing $L-2r$, where $r > r_c$. The dislocation bows around the particle and the bypass mechanism occurs leaving a dislocation loop of radius r^* around the particles and reducing the effective particle spacing to $L-2r^*$ [12].

The yield stress due to dislocation looping is expressed as a function inversely proportional to the channel width, l , which can then be formulated in the following manner [16]:

$$\tau_{loop} = \frac{Gb}{L-2r} = \frac{Gb}{l} \quad (7-21)$$

where L is the mean spacing between particles (measured from center to center), and l is the channel size. The measured mean values of l are found to be both a function of r and f which can be fit into an expression written as: $l = \alpha r(1-f)/f$, where α is a fitting parameter. The fitting parameter is determined by comparing the theoretical value of l with experimental measurements. This expression is similar to several formulae of l reported in literature [21]. Substituting this expression into (7-21) we obtain:

$$\tau_{loop} = \alpha Gb \frac{f}{r(1-f)} \quad (7-22)$$

For a constant volume fraction, the shear stress required for Orowan by-passing will decrease as particle size increases.

Shearing / Looping Transition

For a constant volume fraction of particles, a peak stress exists at a critical radius, r_c , where the shear stress due to shearing is equal to the shear stress due to looping. The critical radius, as shown in Figure 7-7, defines the particle/dislocation interactions in the following manner:

- i) $r < r_c$; $\tau_{shear} < \tau_{loop}$, particle shearing occurs
- ii) $r = r_c$; $\tau_{shear} = \tau_{loop}$, transition between particle shearing and looping
- iii) $r > r_c$; $\tau_{shear} > \tau_{loop}$, particle looping occurs
- iv) r_c increases with volume fraction

Figure 7-7: Schematic of critical resolved shear stress contribution from precipitation hardening (τ_{ppt}) as a function of particle size (in terms of radius, r) and volume fraction (f), where $f_1 < f_2 < f_3$. For a constant volume fraction, the ascending and descending curves correspond to particle shearing and looping; respectively. The intersection of these two curves, for a given f , defines the critical particle size, r_c .

In general, γ/γ' alloys follows a bi- or trimodal γ' distribution. The contribution of each particle population to the total critical resolved shear stress is assumed to follow a rule of mixtures law as [4, 17, 22]:

$$\tau_{CRSS} = \sum_{i=1}^{N_p} \tau_i \quad (7-23)$$

where τ_{CRSS} is the summation of contributions from N_p particle populations ($N_p = 2$ for a bimodal particle distribution). Similarly, if the critical shearing/looping particle size falls within a given particle distribution, the rule mixtures applies as:

$$\tau_i = \tau_{shear} + \tau_{loop} \quad (7-24)$$

where τ_{shear} is for the particles with $d < d_c$ and τ_{loop} is for the particles with $d > d_c$. This critical size will determine the average particle size and volume fraction contribution to both shearing and looping as defined by f^{shear} , r^{shear} , f^{loop} and r^{loop} . Where f^{shear} and r^{shear} are the volume fraction and average size of particles which are cut by dislocations and the latter two terms are those which are looped by dislocations [23, 24]. These sizes and volume fractions can be determined assuming that the particle size follows the classical LSW type distribution given as [25]:

$$P(u) = \begin{cases} \frac{4u^2}{9} \left(\frac{3}{3+u} \right)^{7/3} \left(\frac{-3/2}{u-3/2} \right)^{11/3} \exp\left(\frac{u}{u-3/2} \right) & \text{for } u < 3/2 \\ 0 & \text{for } u \geq 3/2 \end{cases} \quad (7-25)$$

where u is the normalized particle size, r/r_m , r is particle radius and r_m is the mean particle radius. If $u_c = 3/2$, where $u_c = u|_{r=r_c} = r_c/r_m$, then the critical shearing/looping particle size falls within the particle distribution. The population of particles relative to a critical size is shown in Figure 7-8.

Figure 7-8: Illustration of LSW distribution, P versus u , with a critical particle size and corresponding ratios of particles less then and greater then the critical value.

The fraction of particles for $u = 0$ to u_c defines f^{shear} , while those from $u = u_c$ to $3/2$ defines f^{loop} . These are defined as:

$$f^{shear}(r, r_m) = \frac{\int_0^{r_c} r^3 P(r, r_m) dr}{\int_0^{(3/2)r_m} r^3 P(r, r_m) dr}; \quad f^{loop}(r, r_m) = \frac{\int_{r_c}^{(3/2)r_m} r^3 P(r, r_m) dr}{\int_0^{(3/2)r_m} r^3 P(r, r_m) dr} \quad (7-26)$$

The average size corresponding to the above volume fractions are expressed as:

$$r^{shear}(r, r_m) = \frac{\int_0^{r_c} r P(r, r_m) dr}{\int_0^{r_c} P(r, r_m) dr}; \quad r^{loop}(r, r_m) = \frac{\int_{r_c}^{(3/2)r_m} r P(r, r_m) dr}{\int_{r_c}^{(3/2)r_m} P(r, r_m) dr} \quad (7-27)$$

The volume fractions and sizes expressed in equations (7-26) and (7-27) would be substituted into the classical equations given in previous sections to define τ_{shear} and τ_{loop} for implementation in equation (7-24) to define the total shear stress due to a given particle population.

Precipitation Hardening Mechanisms in ME3 and IN100 Alloys

Changes in deformation behavior from γ' shearing to looping has been observed in IN100 alloy at 650°C associated with a variation in secondary γ' size from small to large. Figure 7-9 shows the typical deformation microstructures obtained after low cycle fatigue (LCF) tests in two different microstructures of IN100 at 650°C [26].

Figure 7-9: Transmission electron micrograph of LCF specimen, with γ'_s volume fraction and particle radius of (a) 26.7% and 72.1 nm, showing activation of γ' Orowan by-passing and shearing mechanisms, and (b) 26.7% and 37.5 nm, where shearing of γ' is the prevailing mechanism [26].

In Figure 7-9a, the dislocation loops blocked at the γ/γ' interfaces indicate the evidence of the Orowan by-passing mechanism activation. Other mechanisms may occur due to high local stresses or γ' distribution heterogeneity like γ' shearing by partial dislocations or superdislocations. On the contrary, as seen in Figure 7-9b, the prevailing mechanism is the γ' shearing regardless of the γ' particle type. The shear concerns both γ'_s and γ'_t and occurs very often with partial dislocations leaving the default contrast.

A review of deformation mechanisms in ME3 alloy under various stress and temperature conditions is given in Figure 7-10.

Figure 7-10: Dislocation mechanisms identified by TEM characterization by M. Mill's research group at OSU of deformed creep samples as a function of applied stress and temperature [27].

The mechanisms identified in Figure 7-10 are done by M. Mill's research group at Ohio State University and are documented as part of the MAI report in reference [27]. Creep testing at lower stresses in the range of 95 to 105 ksi, at 1300°F has shown evidence of microtwinning, isolated faulting, and paired and unpaired dislocation activity. Microtwinning is a very thin twin, on the order of 4-50 atomic planes, defined by the number of adjacent $\{111\}$ planes which have been sheared by partial dislocations [28]. Unpaired dislocations can shear γ' by climbing over the particles, while paired APB shearing occurs by reordering. At higher stresses in the range of 110 to 115 ksi, at 1300°F, creep testing has shown evidence of microtwinning, paired dislocation activity and wavy slip. In addition, constant strain rate tests showed

planar (stacking faults) and non-planar (wavy slip with cube shearing) deformation, as well as, dislocation activity in the γ channels.

7.2.2 Microstructure Explicit ISV and XP Models

A major factor influencing time dependent material behavior of superalloys is the microstructure including grain size, grain boundary morphology and precipitate statistics. At elevated temperatures, microstructure effects such as coarsening, solubility and diffusion become critical factors in the deformation and damage mechanisms of these materials. These microstructure changes are established through variations in the heat treatment cycle including solutioning, stabilization and aging. In this section, a model based on the explicit role of precipitate statistics on high temperature hardening parameters is developed. This work is carried out on P/M Nickel-based superalloy, IN100. Parameters verifications of the model are carried out by subjecting IN100 to various heat treatments to control aspects of the precipitate statistics. The model formulations and model implementation is described in the following paper:

**ISOTROPIC AND KINEMATIC HARDENING AS FUNCTIONS OF GAMMA
PRIME PRECIPITATES IN A NICKEL-BASED SUPERALLOY**

by

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Abstract

A microstructure-explicit constitutive model has been developed for the P/M nickel-based alloy, IN100. In this model, the isotropic and kinematic stress components have been derived as a function of the size and volume fraction of γ' precipitates. The hardening contributions of these precipitates consider dislocation/precipitate interactions which have been identified in terms of particle shearing and/or Orowan by-passing mechanisms. The critical shearing/looping particle size has been determined from the knowledge of the yield stress of heat treated specimens having different γ' sizes. This critical size is used to calculate the relative contribution of particles in a distribution based on their size being cut or looped by mobile dislocations. The back stress has been examined through two terms, the first being a short range variable arising from solid solution and the second is a long range variable related to particle by-passing. The material parameters necessary to fully define the isotropic and kinematic hardening components are determined through a series of strain controlled low cycle fatigue tests carried out at 923K (650°C) on smooth specimens of the IN100 alloy varying in their secondary and tertiary γ' precipitate sizes while maintaining constant volume fractions. Numerical simulations of the fully reversed strain-controlled loading, at a strain rate of 10^{-5} sec^{-1} , and strain ranges of ± 0.006 , ± 0.007 and $\pm 0.008 \text{ mm/mm}$, were carried out and the stress versus strain, as well as, the isotropic, kinematic and viscous stress components are compared with the corresponding experimental results.

1. Introduction

Advances in gas turbine technologies drive toward higher temperature and longer flight capabilities. As operating temperatures and hold times at maximum stress of gas turbine discs increases, time dependent material behavior becomes increasingly important. A major factor, influencing both temperature and time dependent material behavior, is the different constituents of the microstructure. During the operation of a disc at elevated temperatures microstructure effects such as coarsening, solubility and diffusion become critical factors in the deformation and damage mechanisms of the disc material. Damage at elevated temperatures, particularly the crack growth process, has been shown to be sensitive to microstructure interactions in the crack tip zone [29-32]. In precipitate hardened superalloys, dislocation/particle interactions have been viewed by several authors [29, 30, 33-38] to be the damage controlling mechanism. Microstructure details of the grain size, grain boundary morphology and precipitates statistics are established by the heat treatment cycle including solutioning, stabilization and aging.

It is well established that precipitate size and volume fraction affect many mechanical properties such as the yield stress, back stress, stress relaxation and creep. Gabb et al [39] have shown that yield strength of LSHR alloy increased with decreasing γ'_s size, and was enhanced with high volume fraction of γ'_s and for combinations of low γ'_s and γ'_t size. Sinha's work [40] on IN-738LC, shows that during long creep tests, γ' precipitates ripen and coarsen, become irregularly shaped and develop rafted microstructures of plate or rod morphology. Fitting of experimental data to a Norton power law form, has a strong dependence of the exponent on stress

and microstructure. Sinha suggests that the change in value of this exponent is from a transition in which dislocations climb over gamma prime particles to that in which they cut the particles. Conversely, Shenoy's work [4] on IN100 implies that all γ' particle/dislocation interactions are shearing. The authors described the isotropic hardening in terms of a threshold stress, using a Taylor type relationship, in which the threshold stress is directly proportional to the square root of dislocation density, which in turn, is inversely proportional to the effective particle spacing. The threshold stress described above is added to the initial critical resolved shear stress (CRSS) to give the total isotropic stress. This CRSS is inversely proportional to the square root of grain size, proportional to volume fraction and inversely proportional to size of γ'_s and γ'_p , and proportional to volume fraction and size of γ'_t particles. These patterns of gamma prime particle/dislocation interactions are consistent with how Milligan et al [22] incorporates γ'_s and γ'_t into the yield stress. However, their work shows no influence of the γ'_p , the size and volume fraction of which did not show a strong correlation with Ramberg-Osgood constants. This is supported by nano-indentation studies, which show that the γ'_p particles are of comparable hardness to the matrix, thus comparable yield strengths. In contrast, work by Heilmer et al [19] on IN100 and Reppich et al [41] on Nimonic 105 incorporate shearing by weakly and strongly coupled dislocations, as well as, looping. The studies mentioned here all agree that precipitate size and volume fraction will influence the CRSS regardless of which mechanisms are operating, γ' shearing and/or Orowan by-passing.

Precipitates, in addition to their contribution to the isotropic hardening, play a role in the kinematic hardening. In this regard, two principal Bauschinger effect

theories, back stress and Orowan theory are generally considered. Back stress is based on dislocation dynamics theory. An increase in dislocation density increases the number of dislocation-dislocation interaction sites and thus increases back stress. In general, particles act as an interaction size, thus a higher volume fraction of precipitates increases the number of interactions between dislocations and particles [42]. Several authors [4, 43, 44] have based their back stress formulations on the increase in dislocation density, where the steady state value of back stress is proportional to the square root of dislocation density. In this type of theory, the microstructure can be incorporated in the dislocation density formulation through the production of GNDs (geometrically necessary dislocations) where it is dependent on the mean free path of dislocations which, in the matrix channels, is dictated by the obstacle spacing in a glide plane. This spacing could be the channel width between γ' precipitates and/or grain size [4, 43]. Grain to grain plastic strain incompatibilities arise from variations in crystallographic orientations and slip activities, thus grain size becomes a factor in the amount of internal stress present [5]. Internal stress arises from these plastic strain incompatibilities in order to maintain strain continuity across the grain boundary and within the grain. This type of theory can also be dealt with in a classical type of model, where explicit formulations of dislocation density are not considered. For example, Chaboche and Rousselier [45, 46], and Nouailhas [47] use a two term back stress formulation. The first term being a fast saturated term accounting for dislocation-dislocation interactions (short range interactions) and the second being a slow saturated term accounting for dislocation-particle or dislocation-grain boundary

interactions (long range interactions). These terms are a function of plastic strain accounting for both dynamic and static recovery.

Dislocation-particle interactions are known as Orowan theory and are based on the assumption that not all particles contribute equally to back stress. Tinga have suggested that back stress originates from spatial distribution of dislocations and thus only relates to GNDs. Whereas SSDs (statistically stored dislocations) usually have random orientation and the back stress contribution is negligible [48]. GNDs result from geometric incompatibilities and they form to account for the strain gradient. Martin has stated that GNDs are produced when a particle is looped while SSDs accumulate during plastic deformation of single-phase materials and also those containing particles which are sheared by dislocations [21]. When particles are coherent and below the critical size, thus the cutting mechanism is operating, the dislocation retardation force depends only on particle chemistry and particle composition [42]. In Orowan theory shearable particles are considered to be part of the solid solution contribution to back stress. When particles are coherent and above the critical size or incoherent, the Orowan by-passing mechanism is operating. In this case, the back stress is dependent on interparticle spacing as discussed previously. The work of Del Valle et al [23] is based on an Orowan theory type of treatment, where they have shown that the hardening behavior of the Inconel X-750 superalloy mainly arises from order hardening from the gamma prime precipitates. The authors carried out heat treatments on the material resulting in gamma prime precipitate sizes ranging from 10 to 53 nm, with a constant volume fraction of 15%. They also carried out tests on a solution heat treated and quenched condition and showed that the back stress is a

summation of two independent contributions, one from the solid solution and the other is from the unshearable gamma prime precipitates. Furthermore, the back stress of the solid solution is due to two factors, heterogeneity in the forest dislocation substructure and the influence of the carbides, of which volume fraction is not negligible. The back stress arising from the gamma prime precipitates takes a form given by Brown and Stobbs [49, 50], where it is dependent on particle volume fraction and size as well as plastic prestrain. Del Valle has used the back stress and yield stress as an indication describing the transition between mechanisms of cutting to looping of gamma prime precipitates. His work has shown that a critical particle size lies around 15 nm, at which this transition takes place.

In line with the studies examined above, which all agree on the significant role of precipitates, particularly those relate to tertiary γ' in the deformation processes, the goal of this paper is to examine the influence of the secondary γ' statistics on the hardening behavior in the P/M alloy, IN100. The first section of this paper examines heat treatments aiming at producing microstructures with variations in their precipitate sizes. Strain controlled fully reversed cyclic loading tests were then carried out on specimens having as received, as well as modified microstructures, at 923K (650°C) in order to develop a constitutive model, which explicitly considers particle size and volume fraction in the isotropic and kinematic hardening variables. The experimental tests will then be utilized to determine the necessary model parameters. The yield stress provides, for each volume fraction, means of determining the critical particle radius for the transition between particle cutting and looping. This critical size has been utilized to determine the relative contributions of particle sizes and volume

fractions to shearing and looping mechanisms. With this knowledge, the back stress has been formulated as an explicit function considering the contribution of these two different mechanisms. The validity of these formulations is examined by a comparison with back stress measurements as obtained from experimental results. Finally, a series of monotonic case studies, performed on a unimodal particle distribution, will examine the model capabilities over a wide range of particle sizes and volume fractions.

2. Material

2.1 Microstructure control: Heat treatment and quantitative analysis

The role of γ' particle size and volume fraction parameters on hardening behavior is established here by a test procedure that requires specimens of the same material having distinct variations in these parameters. This has been achieved through the use of different heat treatment cycles. The model material in this work is Inconel 100 (IN100) with the chemical composition (in weight percent): 4.85 Al, 4.24 Ti, 18.23 Co, 12.13 Cr, 3.22 Mo, 0.71 V, 0.071 Zr, 0.02 B, 0.072 C, and balance Ni [51]. This is a powder metallurgy processed material with a grain size of approximately 5 μm . Since grain size is known to influence the mechanical properties of an alloy, it is kept constant here by performing solutioning heat treatment cycles at subsolvus temperatures. The subsolvus solutioning maintains also the control of γ'_p volume fraction and size (equivalent particle diameter), 24.2% and 1.8 μm respectively. All specimens have seen the typical three stage heat treatment sequence including solutioning, stabilization and aging, termed as received in Table 1. This heat treatment results in a trimodal particle distribution of γ'_p , γ'_s and γ'_t . The intragranular γ' particles, including both γ'_s and γ'_t precipitates, are the main focus of this paper. Two

heat treatments aiming at generating maximum variations in size of the intragranular γ' , are performed on smooth specimens of the as received material (conditions labeled 1 and 2 in Table 1).

Table 1: Heat treatment conditions of as received, overaged and water quenched IN100 materials and corresponding γ'_t and γ'_s particle sizes (mean equivalent particle radius), respectively.

HT Condition	Solutioning	Stabilization	Aging	γ'_t Size (nm)	γ'_s Size (nm)
As received	1422K (1149°C)/ 2hrs/oil quench	1255K (982°C)/ 1hr/fan cool	1005K (732°C)/ 8hrs/air cool	4.7	72.1
Condition 1 (Overaged)	/	1088K (815°C)/ 75min/1°C/min	1005K (732°C)/ 30hrs/air cool	10.6	79.3
Condition 2 (Water Quenched)	1408K (1135°C)/ 2hrs/water quench	1088K (815°C)/ 2hrs/air cool	1005K (732°C)/ 8hrs/air cool	4.7	37.9

Heat treated samples were polished using standard metallographic techniques up to 1 μ m finish and etched for 15 seconds in AG-21 etchant to reveal gamma prime precipitates. Microstructure details of the as received, as well as, the modified microstructures are shown in Figure 1. These micrographs (Figure 1) were analyzed using image processing software to determine the average size and volume fractions of γ' particles. The average particle size is reported as an equivalent particle diameter. Results of this analysis are listed in Table 1. The volume fractions show little variation in the three heat treatments, thus, an average value of 26.7% for γ'_s and 7.5% for γ'_t was considered for all microstructure conditions. The second heat treatment condition, shown in Figure 1(e), where an additional stabilization and aging sequence is applied, thus allowing coarsening of both the γ'_s and γ'_t . The third condition, Figure 1(g), shows a decrease in the γ'_s particle size. In this case, the fast cooling rate from the solutioning temperature allows for precipitation of the γ'_s particles but does not

provide sufficient kinetics for coarsening. The aging condition, which is similar to that of the as received material, maintains the size of the γ'_t .

Figure 1: Secondary electron micrographs (SEM) of the as received IN100 microstructure, swabbed with AG-21 etchant for 15 sec, showing (a) γ'_p located along the grain boundaries and (b) γ'_s in the matrix. (c) Transmission electron micrograph (TEM) of the as received IN100 showing details of the γ'_t . Similarly, (d-e) are the overaged material and (f-g) are the water quenched material, corresponding to those listed in Table 1 for conditions 1 and 2; respectively.

The distribution of particle size is assumed to follow the Lifshitz-Sloyozov-Wagner (LSW) type of distribution, see Appendix A for details. This type of distribution assumes that the particles which have been coarsened during the heat treatment have reached a steady state distribution, P , and is given by Ardell as [25, 52]:

$$P(u) = \begin{cases} \frac{4u^2}{9} \left(\frac{3}{3+u} \right)^{7/3} \left(\frac{-3/2}{u-3/2} \right)^{11/3} \exp\left(\frac{u}{u-3/2} \right) & \text{for } u < 3/2 \\ 0 & \text{for } u \geq 3/2 \end{cases} \quad (1)$$

where u is the particle radius, r , normalized by the mean particle radius, $\bar{r}$. The particle size distributions of secondary γ' for the three conditions are shown in Figure 2.

Figure 2: Normalized γ'_s particle distributions of as received (a) and heat treated (b-c) IN100 microstructures, corresponding to conditions 1 and 2 listed in Table 1; respectively. Normalized γ'_t particle distributions of as received (d) and heat treated (e-f) IN100 microstructures, corresponding to conditions 1 and 2 listed in Table 1; respectively. The LSW distribution given by equation (1) is superimposed in each figure.

This distribution will be utilized in conjunction with a critical particle size to identify the relative volume fraction of the population corresponding to particles above and below this size. In the current analysis, the critical size corresponds to the transition between particles being sheared and those being looped. The distribution will be used to calculate the corresponding size and volume fraction of the particles above and

below the critical radius respectively. These dislocation/precipitate interactions affect the hardening behavior and are identified in terms of the yield stress and back stress.

3. Hardening behavior

3.1 Dislocation/precipitate interactions

There are contrasting views of the dislocation/precipitate interaction mechanisms operating as a function of γ' size and volume fraction in IN100. As previously stated in the introduction, the work of Shenoy et al [4] and Milligan et al [22] on IN100 attribute the all γ' particle/dislocation interactions to shearing mechanisms. In contrast, work by Heilmer et al [19] implies that in IN100, CRSS is incorporated through shearing by weakly and strongly coupled dislocations, and looping is apparent. Their work is carried out by measuring yield stress from compression tests at room temperature. The theoretical curves of CRSS versus particle size are plotted for shearing by weakly coupled and strongly coupled dislocations, as well as, looping by edge and screw dislocations. These curves represent a dislocation/particle interaction mechanism map as a function of particle size. For particles less than 100 nm, the experimentally measured CRSS is in good agreement with the theoretical CRSS, suggesting these particles are sheared by weakly coupled dislocations. For particles in the range of 100-400 nm, the experimentally measured CRSS is in good agreement with the theoretical CRSS, implying these are sheared by strongly coupled dislocations. For particles greater than 400 nm their graphs show a deviation of the experimental and theoretical yield stress data. The experimental values fall between shearing by strongly coupled dislocations and looping by edge dislocations. This suggests both shearing and looping mechanisms exist

simultaneously. Similar results are obtained by Reppich et al [41] on Nimonic 105. For this alloy, large overaged particles of a diameter greater than 120 nm (volume fraction 22%) and 450 nm (volume fraction 51%), show differences in the experimental and theoretical data which are related to particle shearing and looping. The authors also suggest that the transition between shearing and looping occurs over a range of particle sizes, rather than a distinct critical size. Thus, for a given mean particle size it is possible that both mechanisms are operating if the critical radius lies within the particle distribution. This is supported by TEM observations of Nimonic 105 with particle sizes of 75 nm, 220 nm and 320 nm, showing the existence of shearing, shearing and Orowan looping, and Orowan looping respectively. This implies that in the case of a particle size of 220 nm, both mechanisms are operating simultaneously. In addition, changes in deformation behavior from planar slip (γ' shearing) to uniform slip (γ' looping) has also been observed in Astroloy at 923K (650°C) by Gayda and Miner [53] associated with a variation from small to large γ' particle size, as well as, Del Valle et al [23] on Inconel X-750. The work carried out in the following sections will consider both shearing and Orowan by-passing mechanisms, in relation to a critical particle size, are operating in IN100 at 923K (650°C). This assumption is supported by the agreement between experimental and theoretical yield and back stress data.

3.2 *Internal state variable model formulations*

High temperature deformation response of superalloys is characterized by the viscoplastic behavior of the alloy. This behavior can be described using the concept of overstress or viscous stress [45-47] which represents the time-dependent difference

between the actual stress and the equilibrium stress. In tension/compression loading under an applied stress, σ , the viscous stress can be mathematically represented by:

$$\sigma_v = |\sigma - X| - R \quad (2)$$

The last two terms are the evolving hardening parameters where X and R are the kinematic and isotropic stress, respectively. The relation between plastic flow and the viscoplastic potential is determined by means of the normality rule to give plastic strain rate as:

$$\dot{\epsilon}_p = \left\langle \frac{\sigma_v}{K} \right\rangle^n \text{sign}(\sigma - X) \quad (3)$$

where n and K are material constants. The rules governing the evolution of the kinematic and the isotropic hardening variables are described below.

The isotropic stress is a scalar variable and is written as the summation of the initial size of the elastic domain, k , and a function of the accumulated plastic strain, p :

$$R = k + R(p) \quad (4)$$

The first term, k , is the initial value of the hardening stress and can be expressed as the summation of the different strengthening components, see references [4, 22]. This is written as:

$$k = k_f (k_{ss} + k_{\gamma'_t} + k_{\gamma'_s}) \quad (5)$$

where k_f is a fitting parameter (note that the term k is smaller than the macroscopic 0.2 % yield) and the first term, k_{ss} , is the stress required to mobilize a dislocation, as well as the stress concentration that builds up at boundaries. This term is also considered to comprise of the contribution of primary gamma prime particle/dislocation interactions. The work of Milligan et al [22] shows that the γ'_p particles are of comparable yield

strength to the matrix, thus their contribution is embedded in the k_{ss} term. The last two terms in equation (5), $k_{\gamma t}$ and $k_{\gamma s}$, due to intragranular γ' (tertiary and secondary) precipitates occurs as a result of dislocation/particle interactions involving particle shearing by weakly coupled dislocations and/or Orowan by-passing (looping) which are described in detail in Section 3.2. An emphasis is placed on the weakly coupled dislocations in the current study as this provides a simpler mathematical treatment for the condition when identifying the shearing/looping transition, as will be discussed later, through a single critical particle size.

The second term of equation (4), $R(p)$, describes the slow evolution of microstructure associated with cyclic hardening or softening of the material. This term can be written in a manner similar to references [45-47] as:

$$R(p) = Q(1 - e^{-\beta p}) \quad (6)$$

$$Q = Q_{\max}(1 - e^{-\lambda q}) \quad (7)$$

$$q = \max(|\varepsilon_p|, q) \quad (8)$$

where Q and β are the limiting values of the isotropic hardening variable. Q is the saturation limit of R . The constant, β , is a temperature and material-dependent parameter describing the rate by which R reaches Q . Q can be either a positive value, indicating cyclic hardening, or a negative value, indicating cyclic softening. $Q_{\max}$ is the limiting value of Q , and q is the maximum strain achieved during loading, which memorizes the previous plastic strain range [54].

The kinematic stress, X in equation (2), is the evolving hardening term resulting from dislocation/particle interactions. The components of these interactions

are solid solution and particles looped by dislocations. As such, X is expressed as the sum of two components:

$$X = X_1 + X_2 \quad (9)$$

The component X_1 is the short range back stress which is a fast saturated variable that rapidly becomes almost independent of the plastic strain. It represents, therefore, the contribution of solid solution. The second component, X_2 , is back stress due to long range interactions, which slowly evolves as a function of plastic strain and is, therefore, considered to be the product of the stress field resulting from γ' particles being looped by dislocations. A general mathematical expression for X_1 , can be written as an exponential function in the form:

$$X_1 = a_1 \left(1 - e^{-C_1 \epsilon_p} \right) \quad (10)$$

The constants, a_1 and C_1 , are material-dependent parameters. C_1 is associated with the hardening rate and a_1 , is the limiting value or saturation value of the short range kinematic hardening variables. This limiting value, a_1 , is taken as the contribution due to solid solution and grain size. In this study, grain size remained constant for all heat treatment conditions, thus a_1 is taken to be a constant.

The second term in equation (9), X_2 , which, as discussed earlier, is a slowly evolving term, is a function of the plastic strain that arises as a result of dislocations looping particles which are greater than the critical radius [23]. This stress can be calculated following an approach similar to that of Brown and Stobbs, [49] which is based on Eshelby [55] treatment of a spherical elastic inclusion within a matrix, as detailed in Section 3.3. A general form is written as:

$$X_2 = a_2 f \mu \left(\frac{b}{r} \right)^m \left(1 - e^{-C_2 \varepsilon_p} \right) \quad (11)$$

where a_2 and C_2 are material parameters. It should be noted that the parameter C_2 is much less than C_1 in equation (10), thus the X_2 term evolves slower than the X_1 term. The above equation indicates that the long range back stress is proportional to f/r^m , thus increases with higher volume fraction and decreases with larger size. For a constant particle size, an increase in volume fraction would cause a decrease in channel width resulting in a larger stress required to loop the particle. However, for a constant volume fraction, increasing particle size results in a larger channel width and thus requires a lower stress. This back stress term arises from particles greater than the critical radius which are looped by dislocations, thus the f^{loop} and r^{loop} are substituted into the above equation to give:

$$X_2 = a_2 f^{loop} \mu \left(\frac{b}{r^{loop}} \right)^m \left(1 - e^{-C_2 \varepsilon_p} \right) \quad (12)$$

where f^{loop} and r^{loop} are the volume fraction and average size of particles which are looped by dislocations respectively [23].

$$f^{loop}(r, \bar{r}) = \frac{f \int_{r_c}^{(3/2)\bar{r}} r^3 P(r, \bar{r}) dr}{\int_0^{(3/2)\bar{r}} r^3 P(r, \bar{r}) dr} \quad (13)$$

$$r^{loop}(r, \bar{r}) = \frac{\int_{r_c}^{(3/2)\bar{r}} r P(r, \bar{r}) dr}{\int_{r_c}^{(3/2)\bar{r}} P(r, \bar{r}) dr} \quad (14)$$

The term $\bar{r}_c$ is the critical particle radius identifying the transition between shearing and Orowan by-passing and can be determined from the yield stress, as will be discussed later, r is the radius random variable, the distribution of which has a mean radius $\bar{r}$. The limit of the integration is taken based on the limit of the distribution function, P , given in equation (1). The next two sections will detail the formulations for the isotropic and kinematic hardening terms in relation to particle size and volume fraction.

3.3 *Isotropic hardening stress as an explicit function of particle size and volume fraction*

The initial value of hardening stress, k , is expressed as the summation of different strengthening components as in equation (5). The first term arises from the contribution of solid solution and the remaining terms are from the intragranular γ' particles. The contributions to the yield strength due to secondary and tertiary precipitates occur as a result of dislocation/particle interactions involving precipitate shearing and/or Orowan-by-passing (looping) and are calculated as a function of the volume fraction, f and the particle size, r for each of the precipitates.

The first type of hardening occurs due to order strengthening, where the slip in the particle is accompanied by the formation of antiphase boundary and the increase in strength arises from the energy increase. Yield stress due to order hardening is given by Courtney [16] for early stages of precipitation and for situations when dislocations are widely separated as:

$$\sigma_{shear} = M 0.7 \mu \left(\frac{APB}{\mu b} \right)^{3/2} \left(\frac{fr}{b} \right)^{1/2} \quad (15)$$

where M is the Taylor factor, APB is the antiphase boundary energy, b is Burger's vector, f is volume fraction, r is particle radius, and μ is shear modulus.

The second type of hardening occurs when a particle size is above a critical radius and the favorable mechanism becomes dislocation looping. The yield stress due to dislocation looping is expressed as a function inversely proportional to the channel width, l , which can then be formulated in the following manner [16]:

$$\sigma_{loop} = M \frac{\mu b}{L - 2r} = M \frac{\mu b}{l} \quad (16)$$

where L is the mean spacing between particles (measured from center to center), and l is the channel size. The measured mean values of l are found to be both a function of r and f which can be fitted into an expression written as: $l = \alpha r(1 - f)/f$, where α is a fitting parameter. The fitting parameter is determined by comparing the theoretical value of l with experimental measurements. This expression is similar to several formulae of l reported in literature [21]. Substituting this expression into (16) we obtain:

$$\sigma_{loop} = M \alpha \mu b \frac{f}{r(1 - f)} \quad (17)$$

A peak stress exists at a critical radius, r_c , where the shear stress due to shearing is equal to the shear stress due to looping. This critical radius has the following characteristics:

- i) $r < r_c$; $(\sigma_{\gamma'})_{shear} < (\sigma_{\gamma'})_{loop}$, particle shearing occurs
- ii) $r = r_c$; $(\sigma_{\gamma'})_{shear} = (\sigma_{\gamma'})_{loop}$, transition between particle shearing and looping
- iii) $r > r_c$; $(\sigma_{\gamma'})_{shear} > (\sigma_{\gamma'})_{loop}$, particle looping occurs

iv) r_c increases with volume fraction

Using the volume fraction of intragranular γ' particles, the yield stress due to order hardening, using equation (15), and dislocation bowing, using equation (17), are calculated for radii between 0 and 250 nm. These are plotted against particle radius in Figure 3.

Figure 3: Yield stress of γ' particles as a function of secondary and tertiary γ' particle size; lines represent theoretical values and symbols are experimental values.

Figure 3 indicates for a volume fraction of 26.7%, particles with a radius less 63 nm are sheared by dislocations, because the stress required to shear the particle is less than the stress required to loop the particle. While particles with a radius greater than 63 nm are looped by dislocations, because the stress required to loop the particle is less than the stress required to shear the particle. Similarly, for a volume fraction of 7.5%, the critical radius is calculated as 35.3 nm. These critical radii will determine the average particle size and volume fraction contribution to both shearing and looping as defined by r^{shear} , r^{loop} , f^{shear} , and f^{loop} . Where r^{loop} and f^{loop} are given in equations (13) and (14) and f^{shear} and r^{shear} are the volume fraction and average size of particles which are cut by dislocations. These are expressed as:

$$f^{shear}(r, \bar{r}) = \frac{\int_0^{\bar{r}_c} r^3 P(r, \bar{r}) dr}{\int_0^{\bar{r}_c} r^3 P(r, \bar{r}) dr} \quad (18)$$

$$r^{shear}(r, \bar{r}) = \frac{\int_0^{\bar{r}_c} r P(r, \bar{r}) dr}{\int_0^{\bar{r}_c} P(r, \bar{r}) dr} \quad (19)$$

This treatment is similar to that of Del Valle [23] and Bhakta [24]. Substituting these into equations (15) and (17), the yield stress due to particle shearing and Orowan bypassing are described as:

$$\sigma_{shear} = M 0.7 \mu \left(\frac{APB}{\mu b} \right)^{3/2} \left(\frac{f^{shear} r^{shear}}{b} \right)^{1/2} \quad (20)$$

$$\sigma_{loop} = M \alpha \mu b \frac{f^{loop}}{r^{loop} (1 - f^{loop})} \quad (21)$$

The summation of equations (20) and (21), for both populations of γ' particles (tertiary and secondary) yields the contribution of yield stress due to γ' particles:

$$k_{\gamma't} = \sigma_{shear}^{\gamma't} + \sigma_{loop}^{\gamma't} \quad (22)$$

$$k_{\gamma's} = \sigma_{shear}^{\gamma's} + \sigma_{loop}^{\gamma's} \quad (23)$$

Next attention is turned to the remaining term in equation (5), k_{ss} , which is the contribution of solid solution. This term is assumed to be a collective effect of solid solution, carbides, and primary gamma prime particles. The solid solution hardening of the matrix is based on a approach given by Gypen and Deruyttere [7, 8] which assumes the hardening stress increases as a function of solute concentration [9, 10]:

$$k_{ss} = \sum_i \frac{d\sigma}{d\sqrt{C_i}} \sqrt{C_i} \quad (24)$$

where C is concentration of solute i and $d\sigma/d\sqrt{C_i}$ is the strengthening coefficient of solute i . Roth et al [10] determined the strengthening coefficients for various elements based on the work of Mishima et al [11] in which they studied binary Nickel alloys with varying atomic percents of solutes. Table 2 shows the strengthening coefficients for the elements which contribute to strengthening of the gamma matrix, along with the chemical composition of the gamma matrix. This value was measured at 0.2% flow stress at 77K by Mishima and coworkers [11]. This coefficient describes the strain induced in the lattice due to adding the impurity.

Table 2: Strengthening coefficient [10] and chemical composition [9] of the gamma matrix in IN100.

		Ni	Al	Ti	Co	Cr	Mo	V
Strengthening Coefficient	MPa $\sqrt{\text{frac}}$		225	775	39	337	1015	408
Chemical Composition of γ	wt %	Bal	2.25	0.93	27.8	24.5	5.73	0.05

Using the chemical composition and strengthening coefficients given in Table 2 in equation (24) yields a value of solid solution strengthening of 548 MPa. Substituting the experimentally determined yield strength and the values of $k_{\gamma/s}$ and $k_{\gamma/t}$ from equations (22) and (23) into equation (5), taking the k_f equal to 1 and solving this for k_{ss} yields an average value of 224 MPa. This value would represent the solid solution strengthening at 923K (650°C). In this paper the latter value for solid solutioning will be used.

3.4 Kinematic hardening stress as an explicit function of particle size and volume fraction

The back stress due to particle looping can be calculated following an approach similar to that of [49] which is based on Eshelby [55] treatment of a spherical elastic inclusion within a matrix. In this treatment, a spherical particle is assumed to be subjected to a uniform shear strain of $-\varepsilon_{sp}^*$, which is equal to the magnitude of matrix deformation around the particle. If the slip direction coincides with the x axis and the slip plane normal coincides with the z direction, the displacements inside a particle are given by [49]:

$$u_x = -(1-\gamma)\varepsilon_{sp}^*z; \quad u_y = 0; \quad u_z = -(1-\gamma)\varepsilon_{sp}^*x \quad (25)$$

where γ is a constraint factor or Eshelby accommodation factor. Considering a spherical particle with single slip this factor is written as [57]:

$$\gamma = [7 - 5\nu] / [15(1 - \nu)] \quad (26)$$

The strain in the particle is derived from the displacements given in equation (25); their non zero component is:

$$\varepsilon_{xz} = -(1-\gamma)\varepsilon_{sp}^* \quad (27)$$

As mentioned above, the particle is subject to a uniform shear strain of $-\varepsilon_{sp}^*$, where the matrix has deformed by ε_{sp}^* around the particle. Thus, the strain with respect to the unstrained condition is obtained by subtracting the strain, ε_{sp}^* , from the strain in equation (27) to give:

$$\varepsilon'_{xz} = \varepsilon_{xz} - \varepsilon_{sp}^* = \gamma\varepsilon_{sp}^* \quad (28)$$

The stress in the particle is derived from the strain using Hooke's law as:

$$\sigma_{xz} = 2\mu\gamma\epsilon_{sp}^* \quad (29)$$

where μ is the shear modulus. The mean value over the specimen volume is given as [49]:

$$\bar{\sigma}_{xz} = 2\mu\gamma f \epsilon_{sp}^* \quad (30)$$

where f is the volume fraction of particles, assuming the particles are uniformly distributed over that volume. The above equation considers that the particle and matrix have the same elastic constants. For the case of elastic inhomogeneity, the above equation is written as [49, 56, 57]:

$$\bar{\sigma}_{xz} = 2\mu D \gamma f \epsilon_{sp}^* \quad (31)$$

where D is the modulus correction factor, see [23].

$$D = \mu^* / [\mu^* - \nu(\mu^* - \mu)] \quad (32)$$

where ν is Poisson's ratio and μ^* and μ are the shear modulus of the precipitate and matrix respectively. In the current analysis D is taken equal to 1 since the shear modulus of precipitate and matrix are almost the same in IN100.

The symmetrical plastic shear strain is related to the number of Orowan loops by the equation [49]:

$$\epsilon_{sp}^* = (nb)/(4r) \quad (33)$$

where n is the number of Orowan loops, b is the Burger's vector and r is the particle radius. This is a measure of the incompatibility between the matrix and the particle [58]. Wilson's work [59] shows that the mean stress in the particle is proportional to the back stress developed in the matrix. The difference between the forward and

reverse stress-strain curve (back stress), $\Delta\sigma_{fr}$, is shown to be directly proportional to the mean stress value, given as [58]:

$$\Delta\sigma_{fr} = H\bar{\sigma}_{xz} \quad (34)$$

where H is a proportionality constant [58]. This difference, $\Delta\sigma_{fr}$, is measured at a value of plastic strain, ε_{sp} , where the reverse straining curve is at a value of stress equivalent to the yield strength. Substituting for the value of $\bar{\sigma}_{xz}$ using equation (31), the back stress is written as:

$$\Delta\sigma_{fr} = 2H\mu D\gamma f \varepsilon_{sp}^* \quad (35)$$

Solving the above equation for ε_{sp}^* yields:

$$\varepsilon_{sp}^* = \frac{\Delta\sigma_{fr}}{2H\mu D\gamma f} \quad (36)$$

Brown and Stobbs [49] related ε_{sp}^* to the plastic strain, ε_{sp} , measured experimentally at the value of $\Delta\sigma_{fr}$ and showed that this correlation can be written as, see also [49, 58]:

$$\varepsilon_{sp}^* = J \left(\frac{b\varepsilon_{sp}}{r} \right)^n \quad (37)$$

where J and n are constants that Atkinson et al [58] have determined to be 0.7 and 0.5 respectively for Copper-Silica. Substituting this value for ε_{sp}^* into equation (31) yields an equation that expresses the back stress in terms of measurable plastic strain. The difficulty in the above equation is that it does not saturate as the plastic strain ε_{sp} reaches very high values. This trend is incompatible with the evolution of the back stress, $\Delta\sigma_{fr}$, which ultimately reach a steady state conditions since the number of loops will reach a steady state value [60, 61]. A more physically accepted response of

ε_{sp}^* , considering a saturation response, is a modified form of equation (37) which can be written as:

$$\varepsilon_{sp}^* = K \left(\frac{b}{r} \right)^m \left(1 - e^{-P\varepsilon_{sp}} \right) \quad (38)$$

The parameters K , m and P are material dependent constants. This form allows ε_{sp}^* to reach a saturated limit equal to $K(b/r)^m$ at large plastic strain and provides flexibility in the control of the rate in reaching this limit through the material constant P . Substituting ε_{sp}^* into equation (31) yields an equation that expresses the back stress in terms of measurable plastic strain:

$$\bar{\sigma}_{xz} = HK \mu D \gamma f \left(\frac{b}{r} \right)^m \left(1 - e^{-P\varepsilon_{sp}} \right) \quad (39)$$

The macroscopic back stress and plastic strain can be expressed using the Taylor factor M , by the following relations:

$$X_2 = M \bar{\sigma}_{xz} \quad (40)$$

$$\varepsilon_p = 2\varepsilon_{sp} / M \quad (41)$$

where ε_p is plastic strain. Substituting equations (40) and (41) into equation (39) the back stress is written as:

$$X_2 = MHK \gamma f \mu D \left(\frac{b}{r} \right)^m \left(1 - e^{-\frac{MP\varepsilon_p}{2}} \right) \quad (42)$$

A general form is given by equation (11). Considering that the back stress term arises from particles greater than the critical radius, equations (13) and (14) for f^{loop} and r^{loop} are substituted to yield the final form, given in equation (12).

4. Determination of model parameters and simulations

The hardening formulations have been fully identified in equations (2) to (14). In order to determine the material parameters a series of strain-controlled tests were carried out, at 923K (650°C) on low cycle fatigue specimens that have been heat treated in the manner discussed in Table 1. The mechanical testing was carried out using a servo hydraulic test machine, equip with a high temperature furnace and quartz rod extensometer. Fully reversed cyclic stress-strain tests are performed, at a strain rate of $1 \times 10^{-5} \text{ sec}^{-1}$, with strain ranges varying from ± 0.6 to $\pm 0.9\%$. In addition, stress relaxation tests were performed at varying constant strain levels. From these tests, the kinematic and isotropic stress components are determined.

Isotropic Hardening Parameters: The initial value of the hardening stress, k , is determined as the 0.2% yield offset from the stress strain curve for each microstructure condition, details of this are shown in Section 3.2. The evolving isotropic stress, R , is a function of accumulated plastic strain, p , and is assumed to be the difference between the maximum stress of the current cycle, σ_M , and the maximum stress of the first loop, σ_{M0} . This difference becomes $Q = \sigma_{MS} - \sigma_{M0}$, when the current cycle is equal to the saturated cycle. By plotting R/Q versus p in Figure 4(a) and Q versus plastic strain in Figure 4(b), the material parameters β , λ and Q_{max} can be determined by fitting equations (6) and (7).

Figure 4: (a) Fit of equation (6) to experimental R/Q vs. log accumulated plastic strain and (b) fit of equation (7) to experimental Q vs. q for as received IN100 at 923K (650°C).

Kinematic Hardening Parameters: For each strain range, the hardening of the material in the first cycle of the cyclic stress-strain loops is assumed to be related to the kinematic hardening only. The kinematic stress is measured for each microstructure condition by measuring the center of the linear part of the first reversible cyclic loop at each strain range. As discussed earlier, the kinematic stress is expressed as the sum of two terms as given in equation (9). In order to define the average particle size and volume fraction contribution to both shearing and looping as defined by r^{shear} , r^{loop} , f^{shear} , and f^{loop} , the critical particle size must be determined. This

procedure is detailed in Section 3.2, which shows that the critical particle size is calculated as 63 nm and 35.3 nm for average volume fractions of 26.7% for γ'_s and 7.5% for γ'_t respectively. The critical particle size and the LSW distribution are shown in Figure 5 for the three microstructures (see Table 1) for both the tertiary and secondary γ' particle populations.

Figure 5: LSW particle distributions of the tertiary γ' particles for the as received (a) and heat treated (b-c) IN100 microstructures, corresponding to those listed in Table 1 for conditions 1 and 2; respectively. LSW particle distributions of the secondary γ' particles for the as received (d) and heat treated (e-f) IN100 microstructures, corresponding to those listed in Table 1 for conditions 1 and 2; respectively.

This critical γ'_t particle radius (35.3 nm) is greater than $1.5\bar{r}$ of γ'_t for all microstructure conditions, as shown in Figure 5(a) to 5(c), indicating that there is no Orowan by-passing of γ'_t . Thus, all the particles in the distribution contribute to shearing only and r^{shear} and f^{shear} for the tertiary γ' are equal to the mean tertiary particle radius and total tertiary volume fraction for each of the corresponding microstructures. For the γ'_s precipitates, the critical size (63 nm) is greater than $1.5\bar{r}$ only for the microstructure with a radius of 37.9 nm (Figure 5(f)). For this

microstructure, the r^{shear} and f^{shear} for the secondary γ' is equal to the mean secondary particle radius and total secondary volume fraction. In this case, X_2 is zero and equation (10) for X_l is fit to the experimental data, as shown in Figure 6(a). The other two microstructures have a critical γ'_s size which lies within the particle distribution, as shown in Figures 5(d) and 5(e). The relative shearing and looping sizes and volume fractions are indicated in Table 3 for each microstructure. By subtracting the previously determined X_l term from the total X measured, equation (12) for X_2 is fit to the experimental data, as shown in Figure 6(b), and an average value of the constant a_2 is determined for the two microstructure conditions.

Viscous Stress Parameters: A strain-controlled monotonic stress relaxation tests are performed in order to determine the time-dependent viscous stress term. During the periods of holding at a constant strain level, the stress as a function of time is acquired. Thus, under constant strain conditions during hold time, the plastic strain rate can be calculated as: $\dot{\epsilon}_p = -\dot{\sigma}/E$. The viscous stress can be expressed in terms of plastic strain rate by rearranging the equation for plastic flow, given by equation (3), to the form:

$$\text{Log}(\sigma - \sigma_i) = \frac{1}{n} \text{Log}\left(-\frac{\dot{\sigma}}{E}\right) + \text{Log}K \quad (43)$$

For arbitrary values of σ_i , one can plot $\log \sigma_v$ vs. $\log \dot{\epsilon}_p$. The parameters n and K are determined from fitting the linear part of the curve, as shown in Figure 7. For further details of the parameter determination see references [62, 63]. A summary of the parameters determined by the above procedure is shown in Table 4.

Figure 6: (a) Fit of equation (15) for X_1 to experimental kinematic stress vs. plastic strain for heat treated IN100 microstructure with a mean size of γ'_s of 37.9 nm. Note that this fit is representative of X_1 for all microstructure conditions. (b) Fit of equation (12) for X_2 to experimental kinematic stress vs. plastic strain for as received (with a mean size of γ'_s of 72.1 nm) and heat treated (with a mean size of γ'_s of 79.3 nm) IN100 microstructures.

Table 3: The average secondary γ' particle size and volume fraction contribution to both shearing and looping as defined by r^{shear} , r^{loop} , f^{shear} , and f^{loop} for the as received material and heat treated IN100 microstructures, corresponding to those listed in Table 1 for conditions 1 and 2; respectively.

HT Condition	r^{shear}	f^{shear}	r^{loop}	f^{loop}
	nm	%	nm	%
As received	50.1	2.2	79.2	24.6
1 (Overaged)	49.8	1.1	85.2	25.7
2 (Water Quenched)	37.9	26.7	0.0	0.0

Figure 7: Fit of equation (13) to experimental log viscous stress vs. log plastic strain rate for as received IN100 at 923K (650°C).

Table 4: Material parameters for IN100 at 923K (650°C).

Constant	Value	Unit	Reference
ν	0.3		
E	180620	MPa	
μ	69469	MPa	
K	1573	MPa	
n	5.8		
β	37		
Q_{max}	-38	MPa	
λ	927		
k_f	0.53		
k_{ss}	224	MPa	
M	3.06		[19, 23]
APB	0.14	J/m ²	[19, 64]
b	0.253	nm	[19]
α	2.775		
a_1	398	MPa	
C_1	1173		
a_2	52	MPa	
C_2	1		
m	0.5		[58]

Simulations: The validity of these relations and the model parameters will be examined by comparing the experimental stress versus strain results to numerical results. Utilizing the values listed in Table 4, modeling was achieved by carrying out a simultaneous integration of the set of viscoplastic constitutive equations described above. Fully reversed strain-controlled loading case studies, at a strain rate of $1 \times 10^{-5} \text{ sec}^{-1}$, and strain ranges of ± 0.006 , ± 0.007 and $\pm 0.008 \text{ mm/mm}$, were simulated and compared to those obtained experimentally as shown in Figure 8. Figure 8 shows that the simulated stress strain curves follow a similar trend as those obtained experimentally. Considering the narrow variations in microstructure achieved in this alloy, a set of case studies emphasizing the variations in volume fraction and size of the γ' particles are discussed in the following section.

Figure 8: Comparisons of experimental and numerical cyclic stress-strain loops at 923K (650°C) at a strain rate of 10^{-5} sec^{-1} , and strain ranges of (a) $\pm 0.006 \text{ mm/mm}$, (b) $\pm 0.007 \text{ mm/mm}$, and (c) $\pm 0.008 \text{ mm/mm}$. Microstructures of these loops correspond to (a) as received material and (b-c) heat treated microstructures listed as Conditions 1 and 2 in Table 1; respectively.

5. Discussion

The major outcome of the previous sections are the hardening relationships as explicit functions of particle size and volume fraction and the corresponding model parameters for alloy IN100 at 923K (650°C). In order to illustrate the model capabilities, a series of strain-controlled monotonic case studies at a strain rate of $1 \times 10^{-5} \text{ sec}^{-1}$ varying volume fraction and size of the γ' particles were performed utilizing the material constants obtained in Table 4. Since the above analysis shows that the tertiary γ' particles are sheared by dislocations, their only contribution is an additive component to the yield strength and not to the back stress. Thus, for the case studies performed, a unimodal particle distribution is assumed and only secondary γ' particles are considered. Although previous work [29, 30] has shown a limited variation in volume fraction can be achieved experimentally, the case studies performed here are examined over a wide range of volume fractions. The case studies examined here are at four volume fractions, 10, 20, 30 and 40%, with particle radii ranging from 1 to 300 nm.

The first factor that is dependent on volume fraction is the critical particle size. The critical particle size increases as a function of volume fraction. This size controls the relative particle size and volume fraction of particles which are sheared and looped by dislocations. The critical size for the four volume fractions, 10, 20, 30 and 40% are 40, 54, 68 and 82 nm; respectively. This critical size, in conjunction with the material constants obtained in Table 4, is used to simulate the strain-controlled monotonic behavior as a function of size and volume fraction. The highest stress-strain curve will correspond to the material with a volume fraction of 40% and a particle size

equivalent to the critical size. This peak behavior is clearly illustrated in Figure 9 by plotting the stress at a constant total strain value of 0.01 mm/mm as a function of particle size for the four volume fractions considered.

Figure 9: Stress at a total strain of 0.01mm/mm as a function of particle size, for four volume fractions, 10, 20, 30 and 40%.

Figure 9 shows that the peak stress occurs around the critical particle size for the given volume fraction. Furthermore, the stress increases as volume fraction increases. The trend shown in this figure arises from two factors, the yield strength and the long range back stress, each a function of particle statistics. The yield strength is described by the material constant, k , which is shown as a function of particle size for the four volume fractions in Figure 10. Similar to the trend in Figure 9, Figure 10 shows that the peak value of k occurs at a particle size equivalent to the critical particle size and k increases with volume fraction.

Figure 10: Material constant, k , as a function of particle size, for four volume fractions, 10, 20, 30 and 40%.

The second factor contributing to the total stress is the long range back stress, thus the X_2 and X at a constant plastic strain value of 0.002 mm/mm as a function of particle size for the four volume fractions are shown in Figure 11.

Figure 11: (a) Long range back stress component, X_2 , and (b) total back stress, X , at a plastic strain of 0.002mm/mm as a function of particle size, for four volume fractions, 10, 20, 30 and 40%.

The trend in Figure 11(a) is the same as that shown in Figure 11(b) except that Figure 11(b) has been shifted by a constant amount of stress approximately 360 MPa, which is equivalent to the X_I stress. The initial slope of the curves in Figure 11 is equal to zero, this corresponds to a microstructure with no Orowan by-passing and all particles are sheared by dislocations, thus only X_I exists. It can be noted here that the X_I is independent of particle size and volume fraction. As size increases, the volume fraction of particles which are looped increases until the total particle population is looped by dislocations. These figures illustrate that the peak X_2 and X occur at a

particle size greater than the critical size and for higher volume fractions. The peak back stress occurring at a particle size larger than the critical radius differs from the yield strength because the volume fraction dependency in equation (12) is different from that of the yield strength. The constant in equation (12) which describes the saturation value of the X_2 term is shown in Figure 12 as a function of particle size and volume fraction.

Figure 12: Size and volume fraction dependent material constant in the long range back stress, as show in equation (12), $a_2 f^{loop} \mu (b/r^{loop})^m$, as a function of particle size, for four volume fractions, 10, 20, 30 and 40%.

The combination of yield strength and back stress contribute to the trend of the total stress as a function of total strain. Each exhibit a peak behavior as a function of strain, this is attributed to the transition between particle shearing and Orowan by-passing. The increase in volume fraction increases the yield stress, back stress and total stress, while increasing the critical particle size. This implies that an optimum size and volume fraction exists which enhances the stress components.

6. Conclusions

A microstructure-explicit constitutive model has been developed to describe the hardening response of the P/M nickel-based alloy, IN100, as a function of γ'

precipitates size and volume fraction. The hardening contributions of these precipitates have considered dislocation/precipitate interactions in terms of particle shearing and/or Orowan by-passing mechanisms. The dominant deformation mode present is controlled by the γ' size and γ channel width in relation to a critical particle size. For particles less than the critical size, deformation is dominated by the particle shearing mechanism, while for particles greater than the critical size, deformation is dominated by the Orowan by-passing mechanism. The critical particle size is used to calculate the relative contribution of particles in a distribution based on their size being cut or looped by mobile dislocations. These mechanisms, defined with respect to the critical particle size, are incorporated into the isotropic and kinematic hardening laws as explicit functions of size and volume fraction. The critical shearing/looping particle size has been determined from the knowledge of the yield stress of heat treated specimens having different γ' sizes. Furthermore, the back stress has been examined through two terms; the first being a short range variable arising from solid solution and the second is a long range variable related to particle by-passing effects. The variations in the size of γ'_s are shown to influence both the isotropic and kinematic hardening of the material, while the influence of γ'_t only influence the isotropic hardening. A series of strain-controlled low cycle fatigue tests were carried out at 923K (650°C) on heat treated smooth specimens with variations in their secondary and tertiary γ' precipitate sizes while maintaining their volume fractions. Results of these tests were analyzed to identify isotropic, kinematic and viscous stress components. These tests provided the necessary material parameters to fully define the

microstructure-explicit constitutive model. The main conclusions of this study are summarized as follows:

- Variations in mean particle radius, ranging from 37.9-79.3 nm in γ'_s , can be achieved by selected heat treatments. Whereas variations in γ'_t range from 4.7-10.6 nm. The particle distributions are shown to follow the classical LSW type of distribution.
- The isotropic hardening in terms of yield stress is dependent on both γ'_s and γ'_t statistics. This is formulated with respect to a critical particle size, below which γ' shearing and above which Orowan by-passing mechanisms operate.
- For a volume fraction of 26.7% for γ'_s and 7.5% for γ'_t a critical particle size of 63 nm and 35.3 nm is been determined respectively. This size controls the particle/dislocation mechanisms operating. For all microstructure conditions the γ'_t particle size fall within the shearing regime, while γ'_s particle sizes fall within both the shearing and Orowan by-passing regimes.
- The kinematic hardening in terms of a two term back stress component is formulated as a function of particle size and volume fraction. The first term consists of contributions from solid solution and the second term is from the relative size and volume fraction of particles which are looped by dislocations.
- A constitutive material model incorporating γ' particle size and volume fraction in both the isotropic and kinematic hardening components has been developed. Low cycle fatigue tests on the three microstructure conditions are carried out in order to define the material constants for the model. The model shows good agreement with experimental results.

- Case studies performed over a wide range of volume fractions and sizes indicate the model is sensitive to particle statistics. In addition, results imply that for a given volume fraction, an optimum particle size exists which maximizes the contributions of isotropic and kinematic stresses to the total stress value.

Appendix A

The distribution of particle sizes is assumed to follow the LSW distribution given by the form P in equation (1), where u is the normalized particle size, $\bar{r}/r$. The function P has two features:

- i) a maximum at $u = 1.5$ ($r = 1.5 \bar{r}$)
- ii) $\int_0^{\infty} P(u) du = 1$

Below describes the process in order to plot the experimentally measured particle distribution in terms of P . Firstly, the equivalent particle radius was measured experimentally for a minimum of 350 particles for a given microstructure. The particle distribution is plotted in Figure A-1(a) as frequency (F) vs. r .

Figure A-1: (a) Frequency versus particle radius, (b) $z(u)$ versus u and (c) experimental particle distribution and superimposed LSW distribution, P , versus u for a microstructure with an average radius of 37.9 nm.

The axes in Figure A-1(a) are converted from F vs. r to $z(u)$ vs. u . Where $z(u) = Fu^2$.

This new distribution is shown in Figure A-1(b). The experimentally measured points

(defined by the gray bars) are then curve fit to a Gaussian type distribution of the form:

$$z(u) = Z_1 \exp \left(-0.5 \left(\frac{u - Z_2}{Z_3} \right)^2 \right) \quad (\text{A-1})$$

where Z_1 , Z_2 and Z_3 are constants. This curve is shown in Figure A-1(b), by the black

line. The integral, $\int_0^{3/2} z(u) du = \frac{1}{G}$, is calculated to determine the constant G . In order

to normalize the experimental data $z(u)$ is multiplied by a factor of G such that

$\int_0^{3/2} Gz(u) du = 1$. This can be plotted with the distribution P as in Figure A-1(c) as a

function of u .³

³ This is the end of the paper, Isotropic and kinematic hardening as functions of gamma prime precipitates in a nickel-based superalloy

Summary of Microstructure Explicit ISV and XP Models and Results

Based on the formulations described above, the main hardening equations for the microstructure explicit ISV model can be summarized as follows:

$$\begin{aligned}
 X &= X_1 + X_2; \quad \sigma_v = |\sigma - X| - R - k \\
 \dot{\varepsilon}_p &= \left\langle \frac{\sigma_v}{K} \right\rangle^n \exp \left(\alpha \left\langle \frac{\sigma_v}{K} \right\rangle^{n+1} \right) \text{sign}(\sigma - X) \\
 X_1 &= \left[a_{ss} + A_1 \left(f^{shear} r^{shear} \right)^{1/2} \right] \left(1 - e^{-C_1 \varepsilon_p} \right) \\
 X_2 &= a_2 f^{loop} G \left(\frac{b}{r^{loop}} \right)^m \left(1 - e^{-C_2 \varepsilon_p} \right) \\
 k &= k_{ss} + k_{shear} + k_{loop} \\
 q &= \max(|\varepsilon_p|, q); \quad Q = Q_{\max} \left(1 - e^{-\mu q} \right) \\
 \dot{R} &= b(Q - R) |\dot{\varepsilon}_p|; \quad \dot{\sigma} = E(\dot{\varepsilon}_t - \dot{\varepsilon}_p)
 \end{aligned}$$

In addition, the equations for the microstructure explicit XP model can be summarized as follows:

$$\begin{aligned}
x^{(\alpha)} &= x_1^{(\alpha)} + x_2^{(\alpha)} \\
\dot{\gamma}^{(\alpha)} &= \left\langle \frac{|\tau^{(\alpha)} - x^{(\alpha)}| - g^{(\alpha)} - g_0^{(\alpha)}}{K^*} \right\rangle^n \text{sign}(\tau^{(\alpha)} - x^{(\alpha)}) \\
x_1^{(\alpha)} &= \left[a_{ss}^* + a_1^* \left(f^{shear} r^{shear} \right)^{1/2} \right] \left(1 - e^{-C_1^* \dot{\gamma}^{(\alpha)}} \right) \\
x_2^{(\alpha)} &= a_2^* f^{loop} G \left(\frac{b}{r^{loop}} \right)^m \left(1 - e^{-C_2^* \dot{\gamma}^{(\alpha)}} \right) \\
\dot{g}^{(\alpha)} &= b^* \left(Q^{(\alpha)} - g^{(\alpha)} \right) \dot{\gamma}^{(\alpha)} \\
Q^{(\alpha)} &= Q_{\max}^* \left(1 - e^{-\mu^* q^{(\alpha)}} \right) \\
q^{(\alpha)} &= \max(|\dot{\gamma}^{(\alpha)}|, q^{(\alpha)}) \\
g_0^{(\alpha)} &= g_0^{(\alpha)}_{ss} + g_0^{(\alpha)}_{shear} + g_0^{(\alpha)}_{loop}
\end{aligned}$$

The set of equations describing the ISV model are applied to three microstructures of differing in their precipitate volume fraction and size. Results of the one-dimensional ISV model simulation using the equations mentioned above were compared with corresponding experimental data, for alloy IN100, as outlined in the paper above. The model seems to be in good agreement with experimental stress versus strain curves for the three microstructures considered. See Appendix C for details on the codes for implementation of the one-dimensional microstructure sensitive model. In addition, the formulations are in agreement with the deformation mechanisms observed through TEM examination of the dislocation substructures post-testing at 650°C, as discussed in reference [26]. For particles less than the critical size, deformation is dominated by particle shearing mechanisms and for particles greater than the critical size, deformation is dominated by the Orowan by-passing mechanism.

7.2.3 Influence of γ' on Grain Boundary Sliding and Crack Growth Rate

The influence of γ' statistics on grain boundary sliding and crack growth rate has been examined in alloy IN100 at 650°C in an air environment, as well as, ME3 alloy at 704°C in a vacuum environment. In both cases, the dwell crack growth rate is shown to be sensitive to γ' variations in the surrounding continuum. The size and volume fraction of intragranular γ' precipitates (secondary and tertiary γ') directly influence the stress field surrounding the crack tip. A higher constraint on the grain boundary sliding will decrease the crack growth rate. The influence of γ' size and volume fraction on crack growth rate, as well as, its effect on the grain boundary sliding is described in the following paper:

**INFLUENCE OF CONTINUUM PRECIPITATES ON INTERGRANULAR
FATIGUE CRACK GROWTH OF A P/M NICKEL-BASED SUPERALLOY**

by

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Abstract

This paper examines the role of microstructure, in particular, the size and volume fraction of secondary (γ'_s) and tertiary (γ'_t) γ' precipitates, on intergranular crack growth in P/M IN100. A series of different heat treatments were carried out on the as-received material to vary the γ' statistics through control of the different features of the solutioning, stabilization and aging parts of the heat treatment cycles. Dwell fatigue crack growth experiments have been performed on specimens having as received as well as heat treated microstructures at 650°C and 700°C in air with a 0.5 Hz loading frequency with and without a dwell time. Results of the as received material show that the dwell crack growth rate, while dependent on the temperature level, is independent of the hold time period. For the modified microstructures tested at 650°C, the dwell crack growth rate is shown to be sensitive to γ' variations in the surrounding continuum. This sensitivity was illustrated by correlating the crack growth rate and the corresponding continuum yield which shows the dwell crack growth rate decreases with the increase in the yield strength. The influence of the continuum yield on dwell crack growth was first examined numerically by modelling an intergranular crack path surrounded by a continuum region represented by a coarse crystal plasticity model while the far field continuum is represented by an internal state variable model. The grain boundary path considers grain boundary sliding and a critical sliding length which is implemented as a fracture criterion. Results of this model show that the grain boundary sliding is influenced by the viscous strain developing in the surrounding continuum. An increase in the continuum yield strength accompanied with a lower viscous strain results in higher constraint on the grain

boundary sliding and thus a decrease in the crack growth rate. This observation was further examined by calculating the individual contributions of the solid solution, γ'_s and γ'_t to the total yield strength of the continuum.

1. Introduction

Precipitation strengthened Nickel based superalloys have excellent resistance to creep and stress rupture up to 650°C. They are used in a wide range of high temperature design applications. Damage tolerance methodologies needed in these applications require accurate predictions of the crack growth behavior under typical service conditions. A considerable amount of research has been performed on crack growth damage mechanisms and their correlations with various loading and material parameters. A review of the role of each of these factors in crack growth damage mechanisms on high temperature alloys can be found in the work of many authors, see for example Refs. [65-70]. The general conclusion of these studies is that the fatigue crack growth fracture modes can be classified into two distinctive mechanisms, cycle-dependent damage and time-dependent damage processes. The cycle-dependent cracking takes place along slip planes and occurs at frequencies higher than the material's transitional frequency which is a function of the load ratio, temperature, environment and intrinsic factors of the microstructure. On the other hand, time-dependent crack growth, achieved at loading frequencies below the transitional frequency, is a thermally activated process which is characterized by an intergranular fracture path [70, 71]. Many studies have examined the influence mechanical and material parameters including grain boundary phases and gamma prime precipitates, on intergranular cracking, see for example Refs. [37, 72-76].

The precipitates in the continuum are identified as primary (γ'_p), secondary (γ'_s) and tertiary (γ'_t) gamma prime, these are studied in relation to the fatigue crack growth process, see Refs. [33, 35, 36, 38, 53, 77-79]. The work of Pang and Reed [33] have shown that large grain size, large coherent γ'_p precipitate size and higher volume fractions of both γ'_s and γ'_p precipitates improves fatigue lifetimes in P/M Ni-base alloys. They have also shown that in short crack tests, multiple temporary crack arrest events occur as the crack approaches a γ'_p precipitate. Telesman et al [35] found that a larger γ'_s precipitate size, increases the resistance to hold time crack growth. Schirra et al [36] have shown that the γ'_s and γ'_t precipitates had small effects on mechanical properties of two P/M superalloy materials, KM4 and SR3, at 649°C. The strength of both alloys decreased with larger grain sizes and larger γ'_s sizes. While there was no effect of γ'_t size on strength, increasing γ'_t size had beneficial effects on increasing the crack growth resistance. Merrick and Floreen [77] have also observed that large size, uniformly distributed γ' , as well as increased grain sizes, would decrease the intergranular crack growth rate. Knowles and Hunt [37] found that crack tip relaxation is induced by dissolution of fine γ'_t precipitates and selective coarsening of gamma prime decreases crack growth rate. Ma et al [78] in their work on IN783, did not observe a reduced intergranular crack growth rate due to the dissolution of particles but due to the increase of the secondary γ' size. Recent work by Telesman et al [38] has shown that there is no influence of the grain boundary $M_{23}C_6$ phase on hold time crack growth rate and that the key influence on crack growth rate arises from the size of γ'_t . The authors suggest that the γ'_t plays an important role in reducing the crack tip driving force by crack tip relaxation, especially during long hold times. Heat treatment

procedures on LSHR alloy were focused on variations in volume fraction and size of secondary and tertiary precipitates. Hold time crack growth tests were conducted with 90 second hold time period at 704°C and results showed lower remaining stress level (more stress relaxation) corresponds to a lower crack growth rate. Their work shows that the lower remaining stress is associated with larger γ'_t size. This work has dealt with a superalloy having only 5-10% of γ'_s while the majority of γ' is γ'_t , in the range of 50-55%. Similarly, Schirra et al [36], Gayda and Miner [53] have shown that increasing γ'_t size had beneficial effects on increasing the crack growth resistance. On the other hand, Jackson [79] considers that in Nickel based superalloys neither the size nor the volume fraction of the γ' precipitates influence intergranular cracking unless they are in the direct vicinity of the carbides along the grain boundaries.

The studies mentioned above point out to different views of the role of microstructure in the intergranular cracking process. The primary objective of this study is to examine the relationship between precipitates statistics and intergranular crack growth in IN100 alloy. This influence is assumed to exist on the basis of the view that intergranular crack growth, which occurs as the decohesion of the grain boundary interface in the crack tip region, is governed by the grain boundary traction-displacement relationships in both normal and shear directions. These displacements are related, through compatibility requirements, to the deformation field in the immediate continuum surrounding the affected grain boundary path. Localization of grain boundary stress and strain would then depend on the corresponding fields in the immediate neighboring continuum. From this point of view, it is important to examine the role of microstructure in relation to the intergranular crack growth process. In sub-

solvus heat treated IN100, the basic microstructural elements, in addition to grain boundaries and grain boundary precipitates, are the primary, secondary and tertiary precipitates. The γ' precipitates, $\text{Ni}_3(\text{Al,Ti})$, are a coherent phase with an ordered L1_2 crystal structure with a γ/γ' mismatch of 0.65 [80]. The primary γ' precipitates throughout the matrix, particularly at triple points along grain boundaries and have long-time stability. These particles have been reported to have the same nanohardness as the gamma matrix [22], so they are considered to effectively contribute to the deformation process similar to the γ matrix and do not contribute additional hardening effects. On the other hand, the secondary and tertiary precipitates are intragranular particles distributed within the γ matrix and act as barriers to dislocation motion and as such, contribute to the hardening processes. This paper focuses on effects of size and volume fraction of γ' on dwell crack growth rate in alloy IN100. This is achieved by performing a heat treatment study to identify conditions leading to variation in the size and volume fraction of γ' . These conditions are applied to compact tension (CT) specimens, which in turn have been examined under dwell loading cycles at 650°C and 700°C in air environment. The first section of the paper describes material and microstructure control followed by a description of intergranular crack growth experiments carried out on microstructures with different statistics of γ' . The effect of yield strength on intergranular crack growth rate is then examined numerically by utilizing a mechanistic based time-dependent crack growth model. This is then examined with analysis of the observed experimental results in terms of the γ' contributions to the total yield strength.

2. Experimental work

2.1 Material and microstructure control

The material employed in this study is Inconel 100 (IN100), a P/M processed Nickel based superalloy having the composition (in wt%): Al 4.85, Ti 4.24, Co 18.23, Cr 12.13, Mo 3.22, V 0.71, Zr 0.071, B 0.02, C 0.072 [81]. The heat treatment of the as received material is a three stage process consisting of: i) solutioning stage at 1149°C for two hours followed by oil quenching, ii) stabilization at 982°C for one hour followed by fan cooling, and iii) aging at 732°C for eight hours followed by air cooling. The average Rockwell C hardness of the as received material is 42.7 HRC and its microstructure, which is shown in Fig. 1, includes γ'_p located at the triple points of the grain boundaries as well as within the grains and carbides mostly located along the grain boundaries. In order to reveal the grain boundaries the specimens were etched for 3 minutes in Kallings waterless etchant (2g CuCl_2 , 40ml HCl, 40-80ml Ethanol). The average grain size is 5 μm (ASTM 12). The shape and distribution of the intragranular γ' particles, shown in Fig. 1a and 1b, exhibit a bimodal size distribution where the larger particles, γ'_s , appear cubical and the smaller ones, γ'_t , are spherical. The classification between secondary and tertiary γ' particles was determined using a threshold size of 50 nm. Micrographs revealing the precipitate phases were achieved with an etchant of 0.6g granulated Na_2O_2 and 50ml HCl applied for 7-10 minutes. Sizes and volume fractions of the γ'_p are 1.7 μm and 20%, respectively, while attributes of the γ'_s and γ'_t are shown in Table 1. The precipitate sizes are measured using Sigma Scan image analysis software and are reported as the average of the major and minor length of the particle [31].

Figure 1: (a) Secondary electron image of the as received IN100 microstructure, etched with 0.6g granulated Na_2O_2 and 50ml HCl applied for 7-10 minutes, showing γ'_s and γ'_t in the matrix, γ'_p and carbides located along the grain boundaries. (b) Transmission electron micrograph of the as received IN100 showing details of the γ'_t .

Table 1: Yield strength (0.2% proof stress at 650°C, σ_y) and statistics (size, d , and volume fraction, f) of secondary and tertiary γ' of the as received and heat treated IN100 microstructures.

Specimen (Heat Treatment Sequence)		$f_{\gamma'_t}$ (%)	$d_{\gamma'_t}$ (nm)	$f_{\gamma'_s}$ (%)	$d_{\gamma'_s}$ (nm)	σ_y (MPa)
1	As received (1149°C 2hrs oil quenched, 982°C 1hr fan cooled, 732°C 8hrs air cooled)	11	23	29	208	1042
2	Overaged (815°C 75min cooled 1°C/min, 732°C 30hrs air cooled)	24	23	16	155	1095
3	Long term exposure (650°C 1500hrs air cooled)	21	29	19	130	1119
4	Very slow cooling from solutioning (1065°C 2hrs cooled 1°C/min for 10°C with 10min hold, repeating this cooling cycle to room temperature, 982°C 75min air cooled, 732°C 8hrs air cooled)	14	29	26	258	941
5	Aging before stabilization (1065°C 2hrs ice water quenched, 732°C 8hrs air cooled, 982°C 75min air cooled)	25	20	15	209	1035

A set of heat treatment experiments are conducted on the as received material in order to identify conditions that produce maximum and minimum volume fractions and sizes of the γ'_s and to investigate the long term stability of the microstructure, see details in Ref. [31]. These heat treatment conditions, listed in Table 1, are carried out in an air environment and are applied to CT specimens that will be tested to correlate their microstructure features and the corresponding intergranular cracking behavior. All heat treatments carried out in this study are sub-solvus conditions, thus the grain

size, γ'_p size and volume fraction remained constant throughout the specimens. Microstructures corresponding to the selected heat treatment conditions are shown in Fig. 2.

Figure 2: Secondary electron images of etched IN100 microstructures showing size and volume fraction of γ' particles for different heat treated conditions; (a) overaged, (b) long term exposure, (c) very slow cooling from solutioning and (d) aging before stabilization, corresponding to those listed in Table 1.

These micrographs are quantified through the use of image analysis software, results of which are presented in Table 1 in terms of volume fraction and size of γ'_s and γ'_t as a function of the heat treatment processes, respectively. In addition to the CT specimens, three low cycle fatigue specimens, denoted A, B and C, were heat treated in order to study the dislocation/precipitate interactions acting in IN100 at 650°C [90]. In these three additional specimens, the primary, secondary, and tertiary γ' particles have volume fractions of 24.2%, 26.7%, and 7.5% respectively. For microstructures A, B and C, the secondary γ' sizes were (A: 144.2 nm, B: 158.6 nm, C: 75.8 nm) and the tertiary γ' sizes were (A: 9.3 nm, B: 21.1 nm, C: 9.3 nm). These three specimens

are utilized in Section 3.2 for identification of the particle shearing and Orowan bypassing mechanisms present in this alloy. It should be noted here that observations of the microstructure are taken in the interior of the specimens, greater than 5 mm from the surface. General observations on the quantitative analysis of the heat treated microstructures, related to the volume fraction and particle size are as follows; see Ref. [31] for details:

- (i). For the selected heat treatments, changes in γ'_s size were more significant than γ'_t variations. The size of γ'_t ranges from 20-29 nm while for γ'_s , it ranges from 130-258 nm. For all conditions, the sum of the total volume fraction of γ'_t and γ'_s remained almost constant, thus indicating a change between the two populations to maintain an equilibrium volume fraction of γ' . This observation is supported by the work of Jackson and Reed [82].
- (ii). Over aging (Specimen 2: 815°C / 75min / cooled at 1°C/min, 732°C / 30hrs / air cooled) as well as long term exposure at 650°C for a total time of 1500hrs (Specimen 3: 650°C / 1500 hrs several steps / air cooled) results in a reduction in size and volume fraction of the γ'_s which suggests that the precipitate forming elements either go back into the matrix or form the very small γ'_t . During over aging, the kinetics for the growth or coarsening of the γ'_t are sufficient and should, thus, be the driving force for the decrease in size of γ'_s . This is supported by the increase in both size and volume fraction of γ'_t in both the over aged and long term exposed conditions as compared to that of the as received.

(iii). Specimens which have been subjected to a complete heat treatment cycle starting with solutioning, followed by cooling increments $1^{\circ}\text{C}/\text{min}$ for 10°C and 10 min hold (Specimen 4: 1065°C / 2hrs / cooled at $1^{\circ}\text{C}/\text{min}$ for 10°C , with 10 min hold, 982°C / 75min / air cooled, 732°C / 8hrs / air cooled) showed a uniform distribution of γ'_s size. A possible mechanism responsible for particle size uniformity in these specimens is that solutioning at a lower temperature (1065°C) only reduces the size of γ'_s particles but does not dissolve them, thus generating a uniform distribution of small γ'_s . The remaining γ'_s would thus impede the supersaturation of the matrix, which in turn suppresses the precipitation of more γ'_s , so that γ'_s are coarsened again during cooling and subsequent stabilization. This would explain the almost identical volume fraction of the γ'_s under these heat treatment conditions but larger size as compared to the as received condition. The increase in size of γ'_t in this specimen could be due to the variation in cooling rate from the stabilization temperature, air cooling (Specimen 4) is slower than fan cooling (as received). During this part of the heat treatment sequence, the γ'_t particles grow at a slower rate allowing coarsening to take place prior to performing the aging cycle.

(iv). Specimens which were ice water quenched from solutioning followed by aging before stabilization (Specimen 5: 1065°C / 2hrs / ice water quenched, 732°C / 8hrs / air cooled, 982°C / 75min / air cooled), showed a smaller γ'_s volume fraction. In this case, the non-equilibrium condition following aging allows coarsening or growth to dominate during the stabilization stage which

would result in the same size γ'_s as that observed in the as received condition while the volume fraction remains small. The size of the γ'_t particles in this specimen are the smallest (20 nm). By reversing the stabilization and aging cycles, the γ'_t particles are coarsened and then dissolved again in the subsequent step, thus the reduction in size.

2.2 *Dwell fatigue crack growth rate*

The role of microstructure on the crack growth process has been examined by carrying out two sets of dwell-fatigue crack growth experiments. The first set is performed on CT specimens made of the as received material while the second set was carried out on specimens that have been subjected to the heat treatment procedures described in Table 1. All tests were performed on standard CT specimens with dimensions following ASTM E647 (width = 25.4 mm, thickness = 6.35 mm), using servohydraulic material testing systems with the crack growth measurements being monitored using the potential drop technique. Prior to testing, each specimen was prepared using standard metallographic techniques to a 1 micron finish. Pre-cracking was first performed at room temperature and testing was carried out at high temperature in a resistance heating furnace. The loading cycle consists of 1 second loading, 1 second unloading and a dwell time of 0s, 100s and 7200s superimposed at the maximum load level. All tests were performed at a stress ratio of 0.1 at 650°C and 700°C in air environment. Results of the as received material are plotted as da/dN versus ΔK in Fig. 3, which shows that the crack growth rate is proportional to the dwell time. The transition between transgranular and intergranular fracture is defined by a transitional frequency, f_t , which is in the range of 0.06 to 0.12 Hz for the

temperature range of 650°C to 700°C. For loading frequencies greater than f_t , the fracture mode is transgranular while for frequencies less than f_t , the fracture mode is intergranular, typical of that shown in Fig. 4. Focusing on the intergranular fracture region (i.e. frequencies less than transitional frequency), the corresponding da/dN versus ΔK can be plotted in terms of the crack growth speed, da/dt , versus K_{max} , shown in Fig. 5. This figure shows that for each of the two temperature conditions, the crack growth curves with different hold time cycles are consolidated into a single curve. This indicates that for the same temperature, the intergranular growth increment per second is independent of the hold time duration. In addition, crack length versus time and crack opening displacement versus time, detailed in Ref. [31], are shown to be continuous, thus indicating that the thermally activated crack growth process is continuous and does not involve a damage incubation stage, see Ref. [67], suggesting that oxide formation is not a possible environmental damage mechanism.

Figure 3: Crack growth rate as a function of temperature and loading frequency (loading time-dwell time-unloading time) as indicated in the legend, for as received material in terms of da/dN versus ΔK .

Figure 4: Typical intergranular fracture surface.

Figure 5: Crack growth rate as a function of temperature and loading frequency (loading time-dwell time-unloading time) as indicated in the legend, for as received material in terms of da/dt versus K_{max} .

The second set of crack growth experiments is carried out on the four specimens with modified microstructures, see Table 1. These tests were performed in air environment at 650°C at 700°C, with triangular loading frequencies of 0.5Hz and hold times at maximum load of 100s or 7200s. These frequencies are below the transgranular/intergranular transitional frequency and as such, the fracture path in each of the test specimens was intergranular and their da/dt versus K_{max} results are shown in Fig. 6.

Figure 6: Crack growth rate in terms of da/dt versus K_{max} for different loading frequencies (loading time-dwell time-unloading time) as indicated in the legend, conducted at (a) 700°C and (b) 650°C on the as received and modified microstructures, as listed in Table 1. Note an additional microstructure, labeled quenched, is a material with excessive yield strength and is discussed in Section 3.3.

The 700°C tests involved only the overaged microstructure at a loading frequency of 1s-100s-1s, while the 650°C tests were performed on all the modified microstructures. Fig. 6a shows that the crack growth curve of the modified microstructure coincides with that of the as received material. This would suggest that at 700°C, intergranular

cracking is governed solely by grain boundary localized deformation and damage events independent of grain boundary/continuum interactions. The work of Kirchhoff [31] on this alloy shows that environmental damage is the dominant intergranular cracking mechanism at this temperature level. On the other hand, contrary to observations in Fig. 5 and 6a, the crack growth rate of the modified microstructures at 650°C do not consolidate into a single curve, see Fig. 6b. This suggests that variations of γ' statistics in the bulk material have altered the time-dependent mechanisms controlling the intergranular crack growth rate. This can be understood by the fact that high temperature intergranular cracking is generally described as the sum of two damage events related to environment and creep. The environmental damage component results from oxide formation and fracture processes or dynamic embrittlement due to oxygen diffusion. The work of Kirchhoff [31] on IN100 alloy precludes the oxidation route and shows that dynamic embrittlement is the active environmental damage mechanism in this alloy at 650°C. Furthermore, since the subsolvus heat treatment procedures applied to the CT specimens listed in Table 1, did not result, as expected, in changes in grain size, grain boundary morphology or grain boundary phases, it could then be concluded that the grain boundary activation energy in these modified microstructures has not been altered and therefore, the environmental damage contribution at the crack tip is the same for all the microstructures examined here at 650°C. Variations in the observed da/dt in Fig. 6b is thus considered to be mainly the outcome of creep related deformation and damage, involving grain boundary sliding and/or cavitation. In this alloy, scanning electron microscopy observations carried out on the intergranular fracture surfaces show no

evidence of cavities and, thus, sliding is the dominant mode of the grain boundary deformation. In this, following the work of Ghonem and Maciejewski [83], the sliding rate, while dependent on the grain boundary viscous characteristic, is also restricted by requirements of strain compatibility with the adjacent bulk material. In this bulk, the viscoplastic deformation is calculated from the knowledge of the viscous stress as a function of the back stress, isotropic hardening and yield strength [83]. Among these parameters, the yield strength is a measurable material parameter which is also directly influenced by γ' statistics. Considering that changes in the yield strength would influence the bulk deformation which in turn, alters the constraints imposed on the GB sliding rate, it is then possible to correlate the intergranular crack growth rate and the corresponding continuum yield. This conclusion could also be reached by examining the da/dt versus $K_{\max}$ relationship which, being a thermally activated process follows the Arrhenius law and can be written as [84]:

$$\frac{da}{dt} = Ae^{-Q/RT} \quad (1)$$

where A is the frequency factor, Q is the apparent activation energy and is a function of $K_{\max}$, R is the universal gas constant, and T is temperature in Kelvin. Considering T to be inversely proportional to the yield strength of the continuum, one could then correlate the crack growth and the yield strength, σ_y for different $K_{\max}$. This plot is shown in Fig. 7 for $K_{\max}$ ranging from 30 to 50 MPa $\sqrt{m}$, for the modified microstructures plus the as received material, tested at 650°C.

Figure 7: Crack growth rate, at five different K_{max} values, as a function of experimentally measured yield strength (0.2% proof stress) at 650°C for the as received and the heat treated conditions.

This figure shows that the continuum yield and the intergranular crack growth rate are inversely related. This type of relationship is similar to that discussed by Gayda and Miner [53] in their work on Astroloy at 650°C. As mentioned above, this relationship is assumed to be a result of a constraint imposed by the continuum viscous strain, as influenced by its yield strength, on the affected GB slip deformation. This concept will be examined in the analysis section of this paper. In this analysis, a numerical simulation of intergranular crack growth is performed to illustrate the nature of interactions between the continuum hardening and the intergranular crack tip sliding response and related crack growth rate. The role of precipitates on the crack growth becomes apparent from the knowledge that in the precipitate hardened IN100 alloy, the strength arises from solid solution strengthening in the γ matrix and precipitation hardening due to the ordered γ' precipitates which are coherently embedded in the matrix. The yield stress, σ_y , is, therefore, the sum of the combined contributions of the solid solution, σ_{ss} , and the strengthening components associated with tertiary and secondary γ' particles; $\sigma_{\gamma't}$ and $\sigma_{\gamma's}$, respectively. Hence, for the same K_{max} , da/dt is

influenced by the sum, $(\sigma_{ss} + \sigma_{\gamma'_t} + \sigma_{\gamma'_s})$. This relationship can be used to determine the relative influence of γ'_t and γ'_s , through their corresponding yield component and will be discussed in the next section.

3. Analysis

The first part of this section deals with the numerical modelling of an intergranular crack path surrounded by a multi-scale viscoplastic continuum [83]. Results of this model will be used to illustrate the interactions between the continuum deformation, as governed by the corresponding yield and GB sliding. The second part of this analysis section will focus on identifying the individual contributions of γ'_s and γ'_t to the continuum yield strength [83].

3.1 *Effect of yield strength on GB sliding and da/dt : numerical simulation*

This section explores the influence of the continuum yield and related viscoplastic strain on the deformation of the grain boundary fracture path by performing a numerical simulation of intergranular crack growth using a cohesive zone model. In this, the grain boundary fracture path is modeled as an interface governed by viscoelastic traction-displacement law while the hardening behavior of the surrounding continuum is described by a multi-scale viscoplastic constitutive model. The grain boundary interface is in direct contact with a near field continuum (represented by crystal plasticity formulations) surrounded by a far field zone (represented by non-linear kinematic hardening formulations); details of this model are given in Ref. [83]. Focusing here on the grain boundary path, represented as an interface with a node to node contact, is described by traction-displacement laws. These laws follow a viscoelastic relationship in which the elastic displacement is the

difference between the total (u_n, u_t) and inelastic displacements (u_c, u_s) in the normal and tangential directions; respectively. The inelastic displacement is equivalent to the grain boundary sliding displacement, u_s , in the tangential direction and the opening displacement due to cavities, u_c , in the normal direction. These traction-displacement laws are described as:

$$T_n = k_n (u_n - u_c) \quad (2)$$

$$T_t = k_t (u_t - u_s) \quad (3)$$

where the subscripts n and t represent normal and tangential directions, respectively, $T_{n,t}$ is traction and $k_{n,t}$ is cohesive stiffness. In the current analysis, $u_c = 0$, due to the absence of cavity related deformation mechanisms. The grain boundary sliding mechanism is described by a viscous flow law, in which the relative velocity of two sliding layers is written as [85]:

$$\dot{u}_s = (\delta/\eta) T_t \quad (4)$$

where δ is the grain boundary thickness, η is viscosity and T_t is the tangential stress. The viscosity can be derived as a function of the relaxation time or characteristic time, t_c . This is the time a dislocation can climb a distance λ [86], as:

$$\eta = (\delta E_{GB} / w_c) t_c = (\delta E_{GB} / w_c) (\lambda / v_c) \quad (5)$$

where E_{GB} is the unrelaxed shear modulus and w_c is the characteristic length over which grain boundary sliding occurs, w_c is taken equal to 3 microns [83]. The climb velocity or dislocation velocity, v_c , is given by the Einstein mobility relation [87-89]:

$$v_c = M_d F = M_d T_t b \quad (6)$$

where M_d is the mobility of grain boundary dislocations and F is the force per unit length acting on the dislocation and b is the Burger's vector. Substituting equations (5) and (6) into equation (4) yields the sliding rate in terms of dislocation mobility as:

$$\dot{u}_s = w_c \frac{M_d b}{E_{GB} \lambda} T_t^2 \quad (7)$$

Fracture occurs when the grain boundary sliding displacement reaches a critical length, u_{crit} which corresponds to a critical strain estimated as 4% [83].

The model simulation was performed on three cases of the ME3 alloy, a powder metallurgy Nickel based superalloy, tested at 650°C under sustained loading conditions. The yield strength in these cases is selected as 0.75k, k and 1.25k, where k is the yield of the as received condition and the other two vary by factors of 0.75 and 1.25 relative to the as received. These values were selected in order to encompass the range in variation in yield strength experimentally achieved; see Table 1 and Fig. 7. Details of the full parameter determination for the near and far field continuum and grain boundary interface material models are given in [83]. Results of the finite element model are grouped into node and element components. The node components, such as grain boundary tractions and displacements, are outputs of the grain boundary interface. The element components, stresses and extensions of elements located within the crystal plasticity zone of the continuum material, are outputs of the elements surrounding the crack tip. The element extension is taken as the average displacement of the nodes of the continuum element. Since damage in the normal direction is not considered in the IN100 or ME3 alloy, only the components in the tangential direction are considered here. The tangential stress (S_{II}) and element extension (U_I) in the

element directly above the crack tip as a function of time are shown in Fig. 8a and 8b; respectively.

Figure 8: Tangential (a) stress and (b) element extension as a function of time in the element above the crack tip for a material with three different yield strengths, $0.75k$, k and $1.25k$, where k is the yield strength for the as received material.

These show that a material with higher yield strength, characterized by a larger stress and lower element extension or strain at the crack tip, has less stress relaxation at the crack tip. This is assumed to provide higher constraints on the grain boundary sliding. This is evident in simulation results pertaining to the grain boundary traction and displacement. Fig. 9 shows the tangential displacement (u_t), traction (T_t) and grain boundary sliding displacement (u_s) as a function of time in the grain boundary at the crack tip.

Figure 9: Tangential (a) displacement, (b) traction and (c) grain boundary sliding displacement as a function of time in the grain boundary at the crack tip for a material with three different yield strengths, 0.75k, k and 1.25k, where k is the yield strength for the as received material.

The total tangential displacement, shown in Fig. 9a, is lower for a high strength material. As a result, a material with high yield strength and low tangential opening displacement would have a lower tangential traction acting on the grain boundary and lower sliding displacement, as shown in Fig. 9b and 9c; respectively. Furthermore, grain boundary fracture is assumed to occur when the u_s reaches a critical displacement, u_{crit} . Thus, da/dt is proportional to $\dot{u}_s$, described by the slope of the curve shown in Fig. 9c. A material with lower yield strength has a higher u_s , and larger $\dot{u}_s$, reaching u_{crit} in a faster time, thus, has a faster crack growth rate. The crack growth rate in each of the simulated conditions is calculated from Fig. 9c as u_{crit}

divided by the time to reach this limit. Results for 5 simulated conditions are normalized relative to the as received microstructure and plotted in Fig. 10 in the form of normalized crack growth rate versus normalized yield. The trend in this figure is similar to that obtained experimentally in Fig. 7 indicating that the intergranular crack growth rate decreases with the increase in continuum yield strength.

Figure 10: Crack growth rate, da/dt versus yield strength normalized to the as received microstructure, for the five simulated conditions.

The next section will describe the influence of the precipitates' yield strength on da/dt by first calculating the yield strength due to tertiary and secondary precipitates; $\sigma_{\gamma't}$ and $\sigma_{\gamma's}$, respectively, and comparing their sum to the experimental yield. Once the validity of these theoretically calculated components are established, their individual contributions to da/dt will be examined.

3.2 *Relative influence of precipitates' yield strength on da/dt*

As mentioned in the previous section, the continuum yield is the sum of the contributions of the solid solution, σ_{ss} , and the strengthening components associated with tertiary and secondary γ' particles; $\sigma_{\gamma't}$ and $\sigma_{\gamma's}$, respectively. The work of Maciejewski [90] on the same alloy, IN100, has examined the solid solution hardening of the γ matrix, σ_{ss} , at 650°C on the basis of strengthening coefficients from elements

in γ matrix. This work showed that σ_{ss} is approximately 298 MPa which is applicable to all the microstructures considered here. The contributions to the yield strength due to γ'_s and γ'_t occur as a result of dislocation/ γ' particle interactions involving precipitate shearing and/or Orowan by-passing. These contributions are calculated as a function of the volume fraction, f and the particle size, d , for each of the precipitate populations. The first type of hardening occurs due to order strengthening, where the slip in the particle is accompanied by the formation of antiphase boundary and the increase in strength arises from the energy increase. Shearing of precipitates occurs when a particle is below a critical size, d_c , and the stress required for shearing, σ_{shear} , is less than that required for by-passing the particle. For the same volume fraction, the yield stress due to shearing is assumed to follow that for weakly coupled dislocations and is expressed as [15, 16]:

$$\sigma_{shear} = \theta MG \left(\frac{APB}{Gb} \right)^{3/2} \left(\frac{fd}{2b} \right)^{1/2} \quad (8)$$

where θ is a constant which equals 0.7, M is the Taylor factor equal to 3.06 [23], APB is the antiphase boundary energy, taken as 0.14 J/m² [19, 64], b is Burger's vector and G is the shear modulus. This equation is derived by considering a force balance across the leading and trailing dislocation. Once a coherent precipitate reaches a particle size greater than d_c , and taking into account the channel width, the stress required to loop the particle becomes less than that required to shear the particle, thus dislocation looping becomes the favorable interaction mechanism. The yield stress due to dislocation looping, σ_{loop} , is inversely proportional to the channel width, l , which can

then be formulated as a function of d and f leading to the following expression [16, 21, 90]:

$$\sigma_{loop} = M \frac{Gb}{l} = MaGb \left(\frac{2f}{d(1-f)} \right) \quad (9)$$

where a is a material constant. The sum of the above two expressions for each of the precipitates; secondary and tertiary, assumes that precipitate shearing and looping mechanisms are operative on both tertiary and secondary particles depending on their average particle size with respect to the critical shearing/looping particle size, d_c . Transmission Electron Microscopy was utilized in the work of Maciejewski et al [90] to identify the nature of dislocation/particles interactions in heat treated specimens of IN100 alloy subjected to different strain rate and strain ranges. Results of this work showed that both the shearing and Orowan by-passing of γ' particles are active deformation mechanisms in IN100. The occurrence of either mechanism depends on the relative size of the particle with respect to a critical size, d_c which is estimated to be 126 nm [90]. On the basis of these experimental observations, equations (8) and (9) are used to plot trends of yield stress for constant average volume fractions as a function of the average particle size for secondary and tertiary precipitates represented by solid lines in Fig. 11a and 11b; respectively. In these two figures, the ascending part of each of the curves belongs to equation (8) which represents an increase in yield strength as d increases, while the descending part of the curve which belongs to equation (9) indicates a decrease in yield strength with an increase in the particle size. The intersection of these two curves, for a constant volume fraction, identifies the critical particle size, d_c . Fig. 11a shows that the average d_c corresponding to γ'_s is in

the range of 100-126 nm for the volume fractions studied. Furthermore, Fig. 11b indicates that the γ'_t particles, for all microstructure conditions, fall within the shearing regime.

Figure 11: Yield stress of γ' particles as a function of the average (a) secondary and (b) tertiary γ' particle size corresponding to the CT specimens (black circles) and three additional specimens (gray circles) from reference [90], precipitate attributes are mentioned in Section 2.2.

The stress components, $\sigma_{\gamma'_t}$ and $\sigma_{\gamma'_s}$, in Fig. 11, in addition to σ_{ss} , are plotted as a bar graph in Fig. 12 for each modified microstructure, showing the relative contributions of each of these components to the total yield strength.

Figure 12: Bar graph of the total theoretically calculated yield stress components for the as received and heat treated IN100 microstructures.

This figure shows that the average yield strength contribution due to γ'_s is 445 MPa while that due to γ'_t is 313 MPa. These stresses, $\sigma_{\gamma'_t}$, $\sigma_{\gamma'_s}$ and σ_{ss} , are summed to give the theoretically calculated yield stress. The validity of this sum is compared with that obtained experimentally, see Fig. 13, showing a maximum error of 9.7%. These relative contributions of yield strength due to secondary and tertiary γ' particles on the intergranular crack growth rate are examined in Fig. 14a and 14b.

Figure 13: Theoretical versus experimental yield stress for as received and modified microstructure conditions; maximum error in this comparison is 9.7%.

Figure 14: Crack growth rate, at a $K_{\max}$ of 35 MPa $\sqrt{m}$, versus the (a) total theoretical and experimental yield strengths as well as the (b) secondary and tertiary γ' yield strength contributions.

The former figure show that the trend of the theoretical sum of the yield stresses of the continuum is similar to that of the experimental yield. The latter figure shows the

relative correlation between the individual components, $\sigma_{\gamma'_t}$ and $\sigma_{\gamma'_s}$, and the crack growth rate. It is apparent from this correlation that the increase in each of the precipitates' yield provides a higher resistance to intergranular cracking. It is important, however, to observe that da/dt is more sensitive to variations in contribution by the tertiary precipitates as indicated by the slope of $\sigma_{\gamma'_t}$ versus da/dt which is almost twice that of $\sigma_{\gamma'_s}$ versus da/dt .

3.3 Limitations of yield strength

Fig. 7 and Fig. 10 show that the intergranular crack growth rate decreases with the increase in yield strength. In order to explore the limitations of this trend, an additional heat treatment, aiming at generating a microstructure with very high yield strength is attempted. This heat treatment, termed quenched, consists of solutioning at 1135°C for 2hrs followed by water quenching, stabilization at 815°C for 2hrs followed by air cooling, and aging at 732°C for 8hrs followed by air cooling. This heat treatment sequence resulted in secondary and tertiary γ' particles with volume fractions of 27% and 13%, and sizes of 81 nm and 13 nm, respectively. The primary γ' has the same size and volume fraction as the as received microstructure. The yield strength of this heat treated microstructure is measured as 1252 MPa. A crack growth experiment was carried out on this microstructure at 650°C using a CT specimen of the same geometry as the ones used to generate Fig. 6b. Results of this test when plotted with the five microstructures listed in Table 1, see Fig. 6b, shows that a material with excessive yield strength has reversed the trend obtained in Fig. 14; i.e. an increase in the yield strength results in an increase in the crack growth rate. These two trends are illustrated in Fig. 15 for a K_{max} of 35 MPa $\sqrt{m}$.

Figure 15: Crack growth rate, at a K_{max} of 35 MPa $\sqrt{m}$, as a function of experimentally measured yield strength (0.2% proof stress) at 650°C for the as received and heat treated conditions, corresponding to those listed in Table 1; (1) as received (2) overaged, (2) long term exposure, (4) very slow cooling from solutioning and (5) aging before stabilization. An additional heat treatment, 1135°C / 2hrs / water quenched, 815°C / 2hrs / air cooled, 732°C / 8hrs / air cooled, has been applied to a CT specimen labeled (6) in the graph.

The two opposite trends of the crack growth rate versus yield shown in the above figure indicate that da/dt decreases with the increase of σ_y , until reaching σ_y around 1150 MPa, after which the trend is reversed. The first part of this trend ($\sigma_y < 1150$ MPa), can be explained in relation to the positive effect of the hardening of a soft phase, where the highest da/dt and lowest σ_y is expected to correspond to the position of solid solution which has minor additional strengthening from γ' precipitates. Along this trend, as the yield strength increases, due to effects of secondary and tertiary precipitates, the grain boundary sliding, being the dominant deformation mechanism, becomes relatively restricted resulting in a slower GB sliding rate, thus a lower da/dt . Once the yield strength reaches a sufficiently large value, approximately 1150 MPa in this alloy studied here, the excessive pinning of the grain boundary results in apparent grain boundary embrittlement and a decrease in stress relaxation leading to higher crack growth rate. This second trend for $\sigma_y > 1150$ MPa, is in agreement with results

of Telesman et al [38] which suggest that an increased stress relaxation at the crack tip corresponds to a lower crack growth rate.

4. Conclusions

A series of heat treatments were carried out on the IN100 alloy in order to identify conditions leading to variation in the size and volume fraction of γ' precipitates. These specific heat treatments are applied to compact tension specimens to regenerate the corresponding γ' statistics. Dwell-time fatigue crack growth experiments are performed on these specimens at both 650°C and 700°C in air. Concerning the microstructure control, the various heat treatment cycles carried out in this study show that modifications in volume fraction and precipitate size can be achieved by variations in parameters including cooling rate from solutioning and stabilization stages, stabilization time, aging time and post aging thermal exposure, as well as, the order of the heat treatment cycles. These variations in the size and volume fraction of γ'_s are shown to influence the dwell crack growth rate. The main conclusions of this study concerning the P/M IN100 are summarized as follows:

- Applying selected heat treatments result in variations in volume fractions, ranging from 15-29% and mean size, ranging from 130-258 nm in γ'_s , while corresponding variations in γ'_t range from 11-25% and 20-29 nm.
- For the as-received microstructure, the intergranular crack growth rate at 650°C and 700°C, while are shown to be temperature dependent, are independent of the hold time duration. Thus, indicating that the cracking mechanism is a continuous process governed by combined dynamic embrittlement due to environment and creep damage associated with grain boundary sliding.

- For the modified microstructures, the intergranular crack growth rate at 700°C, is independent of microstructure. It is, therefore, assumed that the grain boundary cracking process is dominated by environmental damage at this temperature. At 650°C, however, the crack growth rate is influenced by variations in the precipitate statistics.
- Focusing on the 650°C tests, a correlation is made between the crack growth rate, da/dt , and the continuum yield strength. This correlation is based on the ascertain that changes in precipitates' statics affect the yield strength of the continuum which in turn, due to deformation compatibility requirements in the crack tip region, would influence the sliding rate of the grain boundary fracture path.
- A mechanistic based intergranular crack growth model simulating the growth rate of an interface surrounded by a near and far field continuum zones represented by crystal plasticity and non-linear kinematic hardening formulations; respectively. Results from this simulation suggest that a material with a higher yield strength, characterized by a larger stress and lower element extension or strain at the crack tip, has less stress relaxation at the crack tip, thus introduces a relatively high pinning effect on the grain boundary motion. This simulation suggests that a material with a high yield strength has less grain boundary sliding displacement and consequently, a lower crack growth rate.
- The relative influence of precipitates' yield strength on da/dt has been established by calculating the contributions to the yield strength due to secondary and tertiary precipitates by invoking yield strength formulae that consider dislocation/ γ ' particle

interactions involving precipitate shearing and/or Orowan by-passing. The sum of these contributions, in addition to that of the solid solution, compared well with that obtained experimentally. The yield strength due to both precipitates is shown to be inversely proportional to da/dt . This relationship shows that the intergranular crack growth rate is more sensitive to the yield strength contribution from tertiary γ' compared with that of the secondary precipitates.

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⁶ This is the end of the paper, Influence of continuum precipitates on intergranular fatigue crack growth of a p/m nickel-based superalloy

In order to isolate the influence of Oxygen on the intergranular cracking process, the second part of this work is carried out on variants of alloy ME3 in a vacuum environment. The effect of γ' on the cracking process is performed by carrying out the cohesive zone model analysis for the two microstructure conditions. A focus is made on modifying the secondary γ' particle size. For this the original microstructure condition (M1-ME3) with a secondary size of 230 nm is compared to that with a size of 493 nm, with their relative microstructures shown in Figure 7-11. The microstructure with larger secondary γ' size is obtained through a 3 stage heat treatment procedure in which the cooling rate from solutioning is slow to promote coarsening of the secondary γ' particles. The as received M1-ME3 material (shown in Figure 7-11a) was subjected to solutioning at 1121°C for 30min and cooling at 0.1°C/min to 700°C then air cooling to room temperature, followed by the standard stabilization and aging, as discussed in Chapter 2. This has resulted in an increase in size as shown in Figure 7-11b. In addition, the γ' statistics for both the as received and modified microstructures are given in Figure 7-11. This heat treatment was performed on a CT specimen and the resulting condition is tested in a vacuum environment at 704°C at a loading frequency of 1.5s-300s-1.5s. The experimental results and simulation of these two microstructure conditions are shown in Figure 7-12. The graph in Figure 7-12a shows the total tangential displacement across the grain boundary. The microstructure with the large secondary γ' particles has resulted in a higher displacement than that with the smaller secondary γ' . This has resulted in a higher tangential traction, as shown in Figure 7-12b, and a higher grain boundary sliding rate, as shown in Figure 7-12c, for the microstructure with larger secondary γ' size.

Figure 7-11: Secondary electron image of the (a) as received M1-ME3 material and (b) heat treated ME3 material, etched with AG-21 for 15sec, with the corresponding microstructure statistics.

Figure 7-12: Simulation results for two microstructures, as received M1-ME3 and a heat treated condition, with secondary γ' sizes of 230 nm and 493 nm, respectively, under creep crack growth conditions at 704°C in a vacuum environment. (a) Tangential displacement, (b) tangential traction and (c) grain boundary sliding displacement as a function of time at a K_{max} of 45 MPa $\sqrt{\text{m}}$. (d) Experimental (symbols) and numerical (line) crack growth rate as a function of K_{max} for the two microstructures at 704°C in a vacuum environment at a loading frequency of 1.5s-300s-1.5s.

Thus, the material with higher grain boundary sliding rate will reach the critical sliding limit faster and result in a faster crack growth rate (see Figure 7-12d). The crack growth rate as a function of $K_{\max}$ experimentally obtained in a vacuum environment falls in a trend similar to that numerically predicted. The microstructure with larger secondary γ' results in a faster crack growth rate in vacuum than that with smaller secondary γ' .

7.3 Effect of Grain Boundary Serrations

7.3.2 Influence of Grain Boundary Serrations on Grain Boundary Viscosity

The second cohesive zone model sensitivity analysis is carried out by modification of the grain boundary viscosity through grain boundary serrations. The grain boundary serrations control the intrinsic grain boundary viscosity. This viscosity controls the resistance to grain boundary motion. The grain boundary serrations are generally characterized by a height (h) and wavelength (l). The two micrographs in Figure 7-13 show a planar grain boundary (M1-ME3) and a heavily serrated grain boundary (M2-ME3). The height or amplitude of the serrated grain boundary is measured on average as 2.3 microns. The wavelength is measured on average as 15.6 microns. These two factors (h and l) will increase the grain boundary viscosity by the factor β . Similar to references [85, 91, 92], the serration factor is given as:

$$\beta = 1 + \left(\frac{h}{l} \right)^n \quad (7-28)$$

Figure 7-13: Secondary electron micrograph of ME3 with a planar grain boundary (M1-ME3) and a serrated grain boundary (M2-ME3), with a schematic representation of the height and wavelength of each grain boundary.

For a planar grain boundary where the height of serration is 0, the term β is 1. For a serrated grain boundary the term β is greater than 1. This factor is embedded in the intrinsic grain boundary viscosity as:

$$\eta = \gamma\beta; \quad \gamma = \frac{\delta E_{GB} \lambda}{w_c T_t b M_d} \quad (7-29)$$

As detailed in Chapter 4, this viscosity is inversely proportional to the grain boundary sliding rate by the relation:

$$\dot{u}_s = \frac{\delta}{\eta} T_t \quad (7-30)$$

Thus, an increase in serration severity will increase β , increase η , decrease sliding rate and in turn reduce the crack growth rate.

7.3.2 Influence of Grain Boundary Serrations on Sliding and Crack Growth Rate

A higher viscosity will reduce the grain boundary sliding rate and thus, the relaxation of the tangential traction component will not be as rapid. This in effect is similar to reducing the testing temperature as we previously saw that the tangential traction at 650°C is higher than that at 760°C due to a lower grain boundary sliding. Figure 7-14a shows the numerically predicted da/dt for the three temperatures, 650, 704 and 760°C as a function of the factor β .

Figure 7-14: (a) Numerically predicted da/dt at a K_{max} of 55 MPa√m as a function of the serration factor, β at 650°C, 704°C and 760°C. (b) Numerically predicted da/dt as a function of K_{max} for selected serration factors at the above mentioned temperatures, where (A) is 650°C with $\beta = 1$, (B) 704°C with $\beta = 1$, (C) 760°C with $\beta = 1$, (A') is 704°C with $\beta = 1.23$, (B') 760°C with $\beta = 1.44$. The da/dt for condition A = A' and for B = B'.

β equal to 1 corresponds to the planar grain boundary condition and the increase in beta corresponds in an increase in severity of grain boundary serrations. This plot shows that da/dt at 704°C with a beta of 1.23 is equivalent to the da/dt for a planar grain boundary at 650°C and the da/dt at 760°C with a beta of 1.44 is equivalent to the da/dt for a planar grain boundary at 704°C. This phenomenon is further illustrated for

the conditions mentioned above, which collapse, as shown in Figure 7-14b. This implies that the introduction of grain boundary serrations can lessen the effect of temperature on grain boundary sliding and result in a similar condition as obtained for a material with a planar grain boundary at a lower temperature. The inclusion of grain boundary serrations will reduce the sliding rate and the grain boundary sliding displacement would reach a critical limit in either air or vacuum slower than that for a planar grain boundary.

For the microstructure with a serrated grain boundary considered here, β is calculated as 1.2. This has resulted in a reduction in grain boundary sliding and a decrease in crack growth rate as shown in Figure 7-15. The crack growth rate as a function of $K_{\max}$ experimentally obtained in a vacuum environment for a serrated microstructure falls in a trend similar to that numerically predicted.

Figure 7-15: Experimental (symbols) and numerical (line) crack growth rate as a function of $K_{\max}$ for M1-ME3 (planar grain boundary) and M2-ME3 (serrated grain boundary) at 704°C in a vacuum environment at a loading frequency of 1.5s-300s-1.5s.

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CHAPTER 8

CONCLUSIONS

The objective of this research project was to model the intergranular crack growth of a precipitate hardened Nickel-based disk superalloy as a function of the loading frequency and temperature, taking into account the role of microstructure as well as environment. The success in these efforts would provide a numerical tool for tailoring the alloy response to service conditions through the control of microstructure parameters including grain size, precipitate statistics (size and volume fraction), grain boundary geometry and phases. To achieve this, combined experimental, analytical and numerical studies were performed on ME3 alloy. In the experimental work, 32 fatigue crack growth tests were carried out at four temperatures, 650, 704, 760 and 800°C in both an air and vacuum environment. Results of these tests were presented in the form of crack length (a) versus number of cycles (N), crack growth rate per cycle (da/dN) versus ΔK , crack growth rate per time (da/dt) versus $K_{\max}$ and crack opening displacement versus load and N . A fracture surface analysis was conducted for each test condition and the corresponding fracture mode was identified as a function of loading frequency and temperature. This analysis provided the data input required to determine the transgranular/intergranular transitional frequency (f_t). Furthermore, the apparent activation energy (Q_{app}) was calculated from the slope of the crack growth rate as a function of temperature. Resulting values of Q_{app} were used to determine basic damage processes involved in the intergranular cracking mechanisms in ME3

alloy. These have been identified as grain boundary sliding coupled with dynamic embrittlement due to Oxygen diffusion. The grain boundary sliding mechanism was correlated with slip band/grain boundary interactions and the ensuing dissociation of lattice dislocations resulting in the presence of grain boundary gliding glissile dislocations which lead to a grain boundary sliding displacement. A criterion of this mechanism is that the slip band spacing (inverse of slip band density) exceeds a characteristic length, w_c . Analytical work was developed to correlate the slip band spacing and loading frequency on the basis of minimum strain energy accumulation within a slip band. Results of this experimental/analytical work represent the input parameters necessary for implementation and validation of a mechanistic based time-dependent crack growth model which considers the role of creep, fatigue and environment interactions on both the bulk and the grain boundary interface in the ME3 disk material. The model was established by considering a moving crack tip along a grain boundary path in which damage events are described in terms of the grain boundary deformation and related accommodation processes. Modeling of these events were achieved by adapting a cohesive zone approach in which the grain boundary dislocation network is smeared into a Newtonian fluid element. The deformation behavior of this element was controlled by the continuum in both far field (ISV model) and near field (XP model) and the intrinsic grain boundary viscosity which is characterized by microstructural parameters, including grain boundary precipitates and morphology and was able to define the mobility of the element by scaling up the motion of dislocations into a mesoscopic scale. In this, the motion of grain boundary gliding dislocations in the tangential direction is related to the

observed grain boundary sliding displacement, the rate of which is limited by the climb of dislocations over grain boundary obstacles. Effects of microstructural variation and orientation of the surrounding continuum are embedded in the tangential stress developing across the grain boundary. The mobility of the element in the tangential direction (i.e. by grain boundary sliding) characterizes the accumulation of irreversible displacement while the vertical movement (migration), although present, is assumed to alter stress by relaxation and, thus, is not considered to be a contributing factor in the damage events. These events were controlled by the rate at which the time-dependent sliding reaches a critical displacement and as such, a damage criterion was introduced by considering the mobility limit in the tangential direction leading to strain incompatibility and failure. This limit is diminished by environmental effects which are introduced as a dynamic embrittlement process that hinders grain boundary mobility due to Oxygen diffusion.

A brief summary of results obtained from the experimental / analytical / numerical studies performed here are listed below:

Experimental Low Cycle Fatigue Experiments

- The 0.2% offset yield stress is shown to decrease with the increase in temperature for the ME3 alloy in the range of 650°C to 760°C.
- The isotropic hardening describes how the peak stress evolves from cycle to cycle as a function of accumulated plastic strain. The isotropic stress is calculated as the difference between the initial monotonic and saturated cyclic stress strain curves. For temperatures 650 and 704°C, cyclic hardening is observed in ME3 where the isotropic stress increases with plastic strain, while at 760°C, cyclic softening is

observed. This variation in hardening behavior could be attributed to the fact that 760°C is the aging temperature for this alloy and thus, testing at this temperature condition may lead to unstable microstructure features, in particular the tertiary γ' particles.

- In addition to isotropic hardening, the cyclic stress strain loops for ME3 show evidence of the Bauschinger effect. The two temperatures, 704 and 760°C, have a similar kinematic stress and lower values at 650°C. Similarly, the components X_1 and X_2 are similar at 704 and 760°C. At 650°C, X_1 is lower and X_2 is higher, than X_1 and X_2 at 704 and 760°C. Since X_2 reflects the influence of γ' precipitates, it can be concluded that they are more influential at 650°C to the total back stress.
- The effect of temperature on stress relaxation has also been observed in terms of the viscous stress component. For the same total strain, stress relaxation is independent of temperature, indicating similar strain rate sensitivity as a function of temperature. Furthermore, the large magnitude of the viscous component indicates that time dependency plays an important role in the deformation behavior of ME3.
- The surface of the low cycle fatigue (LCF) specimens, post-testing, in ME3 alloy have indicated a large presence of slip band traces. These traces seem to correlate inversely with the loading rate. In addition, extensive cracking has been observed on the specimen surfaces, located primarily along grain boundaries normal to the loading direction and at the intersection between a slip band and grain boundary. Fracture surface features of broken LCF specimens have been shown to be dominated by intergranular facets.

- A series of heat treatments were carried out on smooth specimens of the IN100 alloy in order to vary the size of γ' precipitates. Low cycle fatigue experiments were performed on these specimens at 650°C under strain-controlled loading scenarios. TEM investigations of the dislocation substructures post-testing revealed that the dominant deformation mode present is controlled by the γ' size and γ channel width in relation to a critical particle size. For particles less than the critical size, deformation is dominated by particle shearing mechanisms and for particles greater than the critical size, deformation is dominated by the Orowan bypassing mechanism.

Continuum Material Modeling (ISV and XP Models)

- The continuum deformation behavior of ME3 alloy has been modeled at two scale levels. The macroscopic level is incorporated through a one dimensional and two dimensional internal state variable (ISV) type model. In addition, a two dimensional coarse crystal plasticity (XP) model was developed to describe the behavior of the continuum for a set of 100 grains. This model is formulated in a similar manner as the ISV model, considering isotropic hardening, as well as, a two term kinematic stress component. They also consider time-dependent recovery terms. The 100 grain XP model consists of a random array of grain orientations with 12 slip systems for the FCC crystal structure. Both the ISV and XP models are implemented in the Abaqus finite element platform through a UMAT user subroutine, in order to simulate uniaxial strain controlled loading scenarios including monotonic, cyclic and stress relaxation case studies. The outputs of the

numerical analysis compare well with those experimentally obtained over the temperatures considered in this work, i.e. at 650, 704, and 760°C.

- The material parameters required for the full identification of the ISV and XP models were obtained from results of 29 LCF tests carried out on the ME3 alloy under different monotonic and cyclic loading scenarios at three temperature conditions 650, 704, and 760°C.
- A microstructure explicit constitutive material model incorporating γ' particle size and volume fraction in both the isotropic and kinematic hardening components has been developed. The initial yield strength considers the contribution of solid solution, tertiary and secondary γ' particles. The contributions to the yield strength due to secondary and tertiary precipitates occur as a result of dislocation/particle interactions involving precipitate shearing and/or Orwan-by-passing (looping) and are calculated as a function of the volume fraction and the particle size for each of the precipitates. The kinematic hardening is in terms of a two term back stress component which is formulated as a function of particle size and volume fraction. The first term consists of contributions from solid solution and the second term is from the relative size and volume fraction of particles which are looped by dislocations. Low cycle fatigue tests on the three microstructure conditions for IN100 alloy were carried out in order to define the material constants for the model. The model shows good agreement with experimental results.

Experimental Crack Growth Curves

- The crack growth rate for the quasi brittle material ME3 is correlated with the stress intensity factor (ΔK). The growth rate per cycle (da/dN) ranges from 10^{-7} to

10^{-3} m/cycle in the range of temperatures mentioned above. An increase in temperature and/or dwell time at maximum load accelerates the crack growth rate. Furthermore, the crack growth rate in vacuum at both 704 and 760°C is generally lower than that in air environment. For the same ΔK , the reduction is almost an order of magnitude.

- A transgranular/intergranular transitional frequency (f_t) was determined as 0.01 Hz. The crack growth mode at frequencies below f_t is predominately intergranular whereas above this frequency, the fracture mode is transgranular. A mixed mode of failure with characteristics of both intergranular and transgranular fracture is observed in the region near the transitional frequency.
- Intergranular crack growth response in terms of the crack speed, da/dt versus K_{max} , lies in the range of 10^{-9} to 10^{-6} m/s for the four temperatures conditions. The da/dt curves of the 650 and 704°C tests show temperature dependency, but are independent of the hold time duration of the dwell cycle. Results at 760°C show anomalous response which was related to possible microstructure instability since the 760°C is the aging temperature of the heat treatment cycle.
- Crack opening displacements (COD) were measured and correlated with the applied force, P , as a function of number of cycles. The general trend of P versus COD curves is linear in both the loading and unloading directions, which indicates the absence of closure effects. This observation supports the view that the environmental effect at the crack tip in ME3 alloy is related to dynamic embrittlement and no oxide formation is involved. Furthermore, COD measurements during the hold time duration of the loading cycles show that the

crack growth is a continuous process. This observation supports the conclusion that the crack tip advancement by grain boundary slip occurs over a length smaller than the grain size.

- The stress exponent for da/dt versus K_{max} curves are 4.02, 3.72 and 5.71 for 650, 704 and 760°C respectively. These values show that the stress dependency of the crack tip is higher at both 650 and 760°C, indicating an increase in the relative role of stress dependent damage mechanisms (environmental effects). The lower stress sensitivity exponent at 704°C indicates a larger role of time-dependent mechanisms (creep by grain boundary sliding).
- The apparent activation energy, Q_{app} , for the intergranular cracking mechanism is measured as 130-140 kJ/mole in vacuum reaching 150-190 kJ/mole in air environment. Q_{app} is correlated in a nonlinear form with K_{max} , the analysis of which shows that the crack growth kinetics is a dual mechanism involving creep by grain boundary sliding and environmental degradation due to dynamic embrittlement by Oxygen diffusion.

Grain Boundary Deformation and Damage Mechanisms

- The deformation response of the continuum involves the formation of slip bands, the density of which is proportional to the loading frequency. The deformation of the grain boundary is explained in terms of activities of trapped extrinsic dislocations. Grain boundary sliding is only observed if a plastic incompatibility exists between the adjacent grains, producing dislocations of similar signs with no obstructions. A condition for the occurrence of this mechanism is having a slip band spacing equal or larger than a critical limit. Grain boundary sliding is

measured from surfaces of intergranular crack growth test specimens using a pre-test scribing method and is estimated to be in the range of 1 to 6 microns.

- An analytical model is developed to correlate slip band spacing (SBS) and loading frequency on the basis of minimum strain energy accumulation within slip bands. The model outcome in terms of SBS as a function of loading frequency is supported by experimental measurements at both high and low loading frequencies. Results of the model show that a saturation of SBS is reached at approximately 3 microns which is shown to coincide with the transitional loading frequency of 0.1 Hz. The critical slip band spacing is used in the numerical crack growth simulation study as the characteristic length of the cohesive zone element in the crack tip region.
- The accelerated damage in the crack tip grain boundary path in air environment is considered to be a result of grain boundary dynamic embrittlement by Oxygen diffusion, the effect of which is to pin the grain boundary dislocations and reduce their effective mobility. Fracture occurs once the accumulated Oxygen at the crack tip is sufficient to result in grain boundary immobility leading to a localized boundary decohesion. This embrittlement mechanism is the basis of a grain boundary fracture criterion formulated in terms of the critical sliding strain in vacuum reduced by a grain boundary mobility parameter which is a function of the Oxygen concentration.

Cohesive Zone Model Simulations

- A mechanistic based model was developed to simulate the intergranular crack growth in ME3 by considering the role of creep-fatigue-environment interactions

on bulk crystalline slip, grain boundary sliding and grain boundary diffusion of Oxygen. Modeling of these concepts was achieved by adapting a cohesive zone approach in which the grain boundary is set as an interface element governed by a linear traction-displacement relationship. The element displacement was calculated as the difference between the total displacement imparted by the surrounding continuum and the viscoplastic strain due to the grain boundary sliding. The continuum deformation is described by a multi-scale model which includes (i) a microstructure-explicit coarse scale crystal plasticity model applicable to higher stress levels near the crack tip, and (ii) a macroscopic ISV model for the far field response several grains removed from the crack tip. A finite element model was developed to simulate the cohesive zone element's deformation and fracture as a function of loading conditions and environment. Results of this simulation showed that the cohesive zone model was successful in capturing details of the crack growth details in vacuum and air. The outputs of this model are briefly summarized as follows:

- The numerical crack growth curves for 704°C and 760°C in vacuum were shown to follow a trend similar to that of the experimental data. The simulated data show an under prediction of the crack growth rates at lower K_{max} values, while the opposite is observed at high K_{max} . This stress dependency illustrates the sensitivity of the model to the power law of the grain boundary sliding rate.
- The crack growth rates for temperatures ranging from 650 to 760°C in air have been predicted and results are in agreement with the range and trends of the experimental data. In contrast to vacuum results, air simulated curves show a

slope slightly lower than that obtained in the experimental curves. This is possibly due to the wide variations in the magnitude of the frequency term in the Oxygen diffusivity function.

- In addition to the 650, 704 and 760°C, a simulation of the crack rate at 800°C has been carried out. The continuum and grain boundary interface material constants required for the simulation process were obtained by extrapolating the available 650-760°C data. The simulation results in the form of da/dt versus K_{max} at 800°C compares well with the experimental results. This simulation points to the potential use of the cohesive zone model, coupled with the available set of material parameters, as a tool to optimize the intergranular crack growth behavior of the ME3 alloy through modifications of microstructure variables at selected service temperatures in the range 650-800°C.
- The cohesive zone model is employed to examine the sensitivity of the ME3 alloy to grain boundary related deformation and damage parameters, specifically, the grain boundary diffusivity, D_{gb} , the grain boundary unrelaxed shear modulus, E_{gb} , and Oxygen partial pressure, C_s (surface concentration of Oxygen). This is intended to provide insight into possible approaches to enhance the alloy's resistance to time-dependent damage processes. Results of these simulations are as follows:
 - A 50% reduction of D_{gb} results in no noticeable effects at 650°C, while at 704°C, the influence of the reduction of the grain boundary mobility is more apparent as K_{max} increases. This influence becomes significant at

760°C, where the reduction in da/dt varies exponentially with K_{max} . These results indicate that an approach limiting the gliding of grain boundary dislocations may not be effective at the lower end of the temperature range, but deemed as a necessity as higher temperatures are applied. Furthermore, the influence of E_{gb} , on the grain boundary sliding has been examined and is shown to be the inverse of that of D_{gb} . These results provide a flexibility in controlling the grain boundary viscosity through routes including segregation of grain boundary elements to combine stiffness (carbide formers: C, Co, W, Cr, Ta, Hf) and lubrication by dislocation emission (B).

- The analysis of C_s has been performed by determining the hold time required to advance the crack tip a distance of 2 μm , t_{fail} , as a function of surface Oxygen concentration. Simulation results show that the rate of reduction of life with the increase in C_s is relatively higher at the lower temperature. At 55 MPa $\sqrt{m}$, a change of C_s from vacuum (0%) to 5%, results in a reduction of life (time to advance 2 μm) by 57%, 51% and 42% at 650, 704 and 760°C; respectively. This points out to the significance of limiting the Oxygen partial pressure within the crack tip region. It is recognized that planar slip bands have the ability to transport Cr atoms to grain boundary interfaces to form oxide phases, which in the presence of a carbide necklace structure, could suppress the Oxygen partial pressure. This would increase the time required for Oxygen concentration to reach the critical limit for grain boundary embrittlement.

- In addition to the sensitivity analysis described above, the cohesive zone model has also been examined in relation to the influence of grain boundary morphology and continuum precipitates. Results of these simulations and experiments are as follows:
 - The influence of grain boundary morphology, in terms of grain boundary serrations, on intergranular cracking, has been examined in ME3 alloy at 704°C in a vacuum environment. The grain boundary morphology (grain boundary serration height and wavelength) controls the grain boundary viscosity. The grain boundary sliding rate is proportional to 1/viscosity. grain boundary serrations increase the grain boundary viscosity. An increase in grain boundary viscosity decreases the grain boundary sliding rate, resulting in a decrease in da/dt . The addition of grain boundary serrations can reduce the effect of the temperature. Thus, a serrated grain boundary at 760°C can have a similar da/dt to a planar grain boundary at 704°C.
 - The influence of continuum precipitates, in particular the secondary γ' size, on intergranular cracking, has been examined in ME3 alloy at 704°C in a vacuum environment. The microstructure of the continuum (γ' precipitate size and volume fraction) controls the stress field surrounding the crack tip and as a result modifies the grain boundary sliding rate. With a focus on secondary γ' size, a decrease in size pins the grain boundary motion, reducing the amount of grain boundary sliding and decreasing the da/dt .

CHAPTER 9

FUTURE RECOMMENDATIONS

Lessons learned from the current studies reflect on issues related to the cohesive zone model as well as aspects concerning the alloy enhancement of damage resistance in an air environment; these are listed here:

- The cohesive zone model is built as a coupling of three different components; ISV, XP and grain boundary models. Details in these formulations were kept at a coarse microstructure scale that allow a smooth transition between components without sacrificing the sensitivity to material variables and provide the ability to capture the evolution of deformation and damage events at a macroscopic level compatible with experimental measurements. This coarse scale selection avoids formulations at a fine scale that, by necessity, would have to be smeared in order to extract global damage parameters. This would render the transition across scales into a sophisticated averaging technique which could mask, and complicate, interpretation of model outcomes. Even at the coarse scale levels exercised here, the full identification of the ISV and the XP models required 29 LCF tests and 229 parameters to be determined and validated. The coupling of the model components in a finite element routine to simulate a single step of the crack tip advancement, i.e. the length of a cohesive element, requires extensive computational efforts. While refinements are applied to the finite element platform to enhance performance, the application of the cohesive zone model to generate a continuous

crack growth path was found computationally inefficient and therefore, simulations were carried out as discrete steps. With keeping the model scale at the coarse level developed here, several steps are suggested to increase the computational efficiency. For example, the degree of influence of the far field continuum on the immediate crack tip deformation field has not been thoroughly examined. It is therefore, suggested to explore using a simple elastic formulation, adjusted for the thermal dependency, as opposed to the ISV. Furthermore, the XP model has been exercised using 100 grains. It is suggested that a bicrystal configuration, encompassing the spread of the crack tip viscoplastic zone, may be sufficient to provide an accurate stress magnitude and orientation at the crack tip.

- The grain boundary tangential traction is a function of the difference between the total strain developed in the adjacent grain boundary continuum and the viscoplastic strain in the grain boundary interface. The lower this difference is, the lower the net grain boundary displacement and the longer the life to failure. This can be achieved by increasing the yield strength of the continuum which can be accomplished by optimizing the volume fraction of the tertiary γ' precipitates at the expense of the secondary γ' and by attempting to reach a distinct trimodal precipitate distribution. This microstructure control is achieved through the optimization of the heat treatment cycle (e.g. increasing the cooling rate of the solutioning and stabilization cycle segments and increasing the temperature level of the aging treatment). This could provide the potential for developing a new generation of ME3 to be used as a stable microstructure at temperatures higher than 704°C. It should however, be mentioned that in the current work,

observations were made regarding the dissolution of the tertiary γ' in the crack tip zone under the influence of high stresses at elevated temperatures. This phenomenon deserves further examination.

- The dislocation/particle interaction has been extensively studied under unimodal particle distributions. The relative effects of the tertiary and secondary γ' precipitates have been treated in this thesis, and many other works, as an additive effect, following a rule of mixtures law. The contributions of either particle population to the isotropic hardening are assumed as the sum of their relative size and volume fractions. In the case where dislocations loop the secondary γ' particles, there is an assumption that the tertiary γ' precipitates between the secondary γ' act as the γ matrix material. Some other authors treat the tertiary γ' particles as a cluster size, thus acting as a secondary γ' particle. Further investigation of the influence of multimodal particle distributions could be examined. The influence of having tertiary γ' particles in the γ channel between secondary γ' particles can also be examined. This could be studied at multiple scale levels. The XP type models can be used with varying formulations considering size, volume fraction, cluster size, distributions, etc., to study the influence of particles on the global stress strain behavior of the material. A microscopic scale, at the particle dislocation level, in terms of phase field simulations can also be used. This will examine the intimate details of the particle dislocation interactions in relation to various deformation mechanisms, stress, temperature and orientation considerations.

- With regard to the grain boundary deformation and related intergranular cracking, a condition for the grain boundary sliding is established in terms of the slip band spacing approaching a characteristic length. Reaching this criterion during a hold time loading can be retarded by pre-straining that can be induced by forging isothermally under superplastic strain rate conditions or by forging at a few degrees colder than that at the conventional practice. Published work by Ghonem and co-workers has shown that a predeformed material of alloy 718 at 650°C, has experienced a deformation response under low loading frequency similar to that at high loading frequency. In addition, the fracture mode has shifted into transgranular fracture with a growth rate almost a decade lower than that in a material that has not been subjected to pre-straining. It is recommended to examine the effect of pre-straining on ME3 at the laboratory coupon size in relation to slip band spacing, loading frequency, fracture mode and grain boundary sliding. Another approach, in this regard, is to focus on increasing the grain boundary cohesive energy. This can be achieved through a selection of elements that segregate to the grain boundary. Several studies, similar to the work reported here, have shown that the dominant response of the grain boundary to the stress concentration associated with a dislocation pile-up is grain boundary fracture. In Boron-doped material, however, it was observed that grain boundary stress relief occurs by the emission of dislocations from the grain boundary. These results are consistent with Boron increasing the grain boundary cohesive energy. It is recommended that crack growth experiments be carried out on samples of ME3 that have been impregnated with Boron (or other selected elements) along its grain

boundary network in order to observe the influence of the segregated element on the grain boundary cohesive energy. In fact, literature shows that the topic of identifying the grain boundary cohesive energy as a function of grain boundary precipitates aiming at relaxing grain boundary stresses in order to reduce sliding has not been studied in depth.

- The grain boundary sliding mechanism has been related to the dissociation of extrinsic lattice dislocations allowing the glissile dislocations to glide along the grain boundary. This mechanism occurs, provided the slip band spacing is greater than its critical characteristic length. While this has been supported in literature in high angle boundary microstructures, it is recommended that the details of this mechanism including grain boundary dislocation transmission, annihilation and sliding as a function of loading frequency be examined.
- The numerical simulation has explored the role of the Oxygen partial pressure (PO_2) on the grain boundary mobility and related fracture limit. It has been shown that these parameters are sensitive to small variations in the PO_2 . As mentioned above, an approach combining an increase in the Cr content ($>13\%$) coupled with formation of carbides along the grain boundary could provide a means of controlling the Oxygen diffusion and related dynamic embrittlement damage (keeping in mind the effect of carbides on grain boundary stiffness and negative effects on deformation accommodation). It is recommended that an experimental program exploring effects of Oxygen partial pressure on ME3 specimens that have their grain boundary manipulated in the manner discussed above be carried out in

order to provide a fundamental knowledge on the critical Oxygen concentration and give insight into the diffusivity parameters.

- The cohesive zone simulation has explored the influence of grain boundary morphology (grain boundary serrations) on the grain boundary sliding and intergranular cracking processes. Other factors such as grain boundary carbides can be studied. In addition, the grain boundary serrations were assumed to only effect the grain boundary viscosity in this work, while the influence on Oxygen diffusion is treated as the same as that for a planar grain boundary. It is recommended that further exploration of the influence of grain boundary serrations on the diffusion of Oxygen be carried out.

The low cycle fatigue specimen, as described in Chapter 5, is used for strain controlled monotonic, stress relaxation and cyclic testing. These are smooth bar specimens with 0.8 in gage length and 0.25 in diameter, as shown in the drawing in Figure A-3.

Figure A-3: Low cycle fatigue specimen drawing.

APPENDIX B

CRACK GROWTH TESTING PROCEDURE AND DATA ANALYSIS

This appendix contains the description for the testing procedure for the crack growth tests, in terms of crack opening displacement and crack length measurements, as well as, MATLAB codes for data reduction.

Crack Opening Displacement Measurements

As described in Chapter 3, crack growth tests were carried out on compact tension type specimens. Figure 3-1a shows the experimental test setup for air tests. In this, the crack opening displacement (COD) is measured using a clip gage with a scissor extension for use at high temperature. A schematic illustration of the high temperature scissor is shown in Figure B-1 below.

Figure B-1: Schematic of high temperature scissor extension and distances where the clip gage measures, as well as, distance of COD in the specimen.

In this, the clip gage measures distance D_{gage} and the COD for the specimen is distance D_{spec} . The conversion to obtain the COD for the specimen can be obtained using similar triangles from the relationship:

$$COD = D_{spec} = D_{gage} \frac{A}{B} \quad (B-1)$$

where $A = 123.2$ mm and $B = 56.4$ mm.

Crack Length Measurements

Crack length is monitored using the direct current potential drop technique. In this, a constant current is applied to the specimen. As the crack grows, the cross-sectional area is reduced causing an increase in electrical resistance. The potential drop, or voltage, is measured in the specimen at two locations, an active potential, V_1 , spanning the crack and a reference potential, V_2 , located remotely from the crack, as indicated in Figure B-2. As the crack grows these voltage measurements will increase, the voltage ratio, V , is monitored as a function of number of cycles. This ratio is given as:

$$V = \frac{V_{1c} - V_{1o}}{V_{2c} - V_{2o}} \quad (B-2)$$

Where V_{1o} and V_{2o} are the voltage measurements when the relay is open (i.e. the current supply is cut off) and V_{1c} and V_{2c} are the voltage measurements when the relay is closed (i.e. the current is passing through the specimen). This method will eliminate the propensity of thermal electrometric forces as detailed in reference [Zheng 1991]. This ratio can be converted to crack length, a , by a calibration curve. This curve correlates V and experimentally measured a (by optical microscope), normalized with specimen width W , as a function of number of cycles, N , as shown in Figure B-3.

Figure B-2: Wire diagram for crack growth specimen, where *TC* are K type thermocouples and *V_i* are potential drop voltage wires (chromel type wire), both spot welded to the specimen, and *I* are current wires, spot welded to the load train (passing through the specimen through the loading pins).

Figure B-3: Voltage to crack length calibration curve.

The data in Figure B-3 can be curve fit to a polynomial form given by:

$$\frac{a}{W} = 1.0155 - 0.0512(V) - 0.0236(V)^2 \quad (\text{B-3})$$

This equation is used in the MATLAB program *CTVA.m* to convert a given set of data acquired from the potential drop method. This program will then output the *a* as a function of *N*. The crack length data is then cleaned using a 7 point running average and blocking technique, as detailed in MATLAB programs *smooth.m* and *block.m*. This is followed by converting the *a* versus *N* into crack growth rate and stress intensity factor using the MATLAB program *DK_dadN_Kmax_dadt.m*. These four programs are given below.

B-1: CTVA.m

```

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
% Program CTVA2.m
% By Kimberly Maciejewski
% Created on July 29, 2007
% This Program is based on file CT122.BAS by Dian Zheng that converts a set
% of data N vs V1/V2 to a set of data of N vs a for a compact tension
% specimen based on two measured points.
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
% Clear Command Window
clc; % to give you a clean screen
% Clear Workspace
clear; % to clear any previously stored variables
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
FigureCount = 0; % Initialize Figure Count
Font_Size = 20; % Set Font Size
%
% Input initial values at the beginning N = 0 cycles,
% and final values at N = final cycles
Vi = 3.81 ; % (ratio) Potential Ratio (V1c-V1o)/(V2c-V2o) at t = 0
aWi = 0.23382 ; % (ratio) Crack Length to Width Ratio a/W at t = 0
Vf = 2.6275 ; % (ratio) Potential Ratio (V1c-V1o)/(V2c-V2o) at t = final
aWf = 0.57544 ; % (ratio) Crack Length to Width Ratio a/W at t = final
W = 25.514 ; % (mm) Width of Specimen
Ni = 0 ; % (cycles) Number of Cycles at t = 0
Nf = 7215.7143; % (cycles) Number of Cycles at t = final
ai = 5.966 ; % (mm) Crack Length at t = 0 (After Precracking)
af = 14.682 ; % (mm) Crack Length at t = final
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%

```

```

%
% Input Constants from V1/V2 vs. a/W Calibration Curve of the form
%  $y = C1 + C2*x + C3*x^2$  where  $y = a/W$  and  $x = V1/V2$ 
% Note: constants came from fit quadratic polynomial in Sigma Plot
% from calibration curve 1.5-1.5 (M1G-10 @ 760C)
C1 = 1.0155;
C2 = -0.0512;
C3 = -0.0236;
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
% Calculate a/W from calibration curve for initial and final conditions
aWci = C1 + C2*Vi + C3*Vi^2;
aWcf = C1 + C2*Vf + C3*Vf^2;
% Calculate new constants for shifted calibration curve
A = ((aWci - aWi) - (aWcf - aWf))/(Vf-Vi);
B = (aWi - aWci) - A*Vi;
G1 = C1 + B;
G2 = C2 + A;
G3 = C3;
%
% Call N and V from experimental data file
Atemp = load('NV_exp.txt');
% create column vector N and column vector V
N = Atemp(:,1); % column 1 is N (cycles)
V = Atemp(:,2); % column 2 is Potential Ratio (V1c-V1o)/(V2c-V2o) (ratio)
%
P = size(N); % Total Number of Data points
P = P(1,1);
%
% Calculate a crack length from V potential data
for j = 1:P % Number of data points
    A(j) = W*(G1+G2*V(j)+G3*V(j)^2); % (mm) crack length
end
%
FigureCount = FigureCount + 1;
figure(FigureCount)
plot(N,A,'-ko',Ni,ai,'rx',Nf,af,'rx')
xlabel('Number of cycles, N (cycles)','FontSize',Font_Size)
ylabel('Crack length, a (mm)','FontSize',Font_Size)
title('a vs. N from CTVA.m','FontSize',Font_Size)
set(gca,'FontSize', Font_Size)
grid on
%
% Print File with N and a
temp = fopen('Na_by_CTVA.txt', 'wt'); %write text
for i = 1:P % Number of data points
    fprintf (temp, '%-20f \t %-20.10f \n', N(i), A(i));
    % tab cycle number tab crack length new line
end
fclose(temp);
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% End of CTVA.m
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%

```

B-2: smooth.m

```

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
% Program smooth.m
% By Kimberly Maciejewski

```

```

% Created on July 29, 2007
% This Program is based on file SMOOTH.BAS by Dian Zheng that smooths
% the data points based on a seven point moving average method
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
% Clear Command Window
clc; % to give you a clean screen
% Clear Workspace
clear; % to clear any previously stored variables
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
FigureCount = 0; % Initialize Figure Count
Font_Size = 20; % Set Font Size
%
% Call N and a from experimental data file
Atemp = load('Na_by_CTVA.txt');
% create column vector N and column vector a
N = Atemp(:,1); % (cycles)
a = Atemp(:,2); % (mm) crack length
%
P = size(N); % Total Number of Data points
P = P(1,1);
M = P(1,1) - 3;
%
% Calculate Y a column vector of crack length a, a 7 point average
% Skips the first 3 and last 3
J = 4; % Initialize J
while J <= M
    Y(J) = (a(J-3) + a(J-2) + a(J-1) + a(J) + a(J+1) + a(J+2) + a(J+3))/7;
    J = J + 1;
end
%
% Calculate Y 1, 2, 3, P, P-1, P-2 (the numbers that we had to skip before)
Y(1) = (13*a(1) + 10*a(2) + 7*a(3) + 4*a(4) + a(5) - 2*a(6) - 5*a(7))/28;
Y(2) = (5*a(1) + 4*a(2) + 3*a(3) + 2*a(4) + a(5) - a(7))/14;
Y(3) = (7*a(1) + 6*a(2) + 5*a(3) + 4*a(4) + 3*a(5) + 2*a(6) + a(7))/28;
B_1 = (a(P-6) + 2*a(P-5) + 3*a(P-4) + 4*a(P-3) + 5*a(P-2));
Y(P-2) = (B_1 + 6*a(P-1) + 7*a(P))/28;
B_2 = (-a(P-6) - 2*a(P-5) + a(P-4) + 4*a(P-3) + 7*a(P-2));
Y(P-1) = (B_2 + 5*a(P))/14;
B_3 = (-5*a(P-6) - 2*a(P-5) + a(P-4) + 4*a(P-3) + 7*a(P-2));
Y(P) = (B_3 + 10*a(P-1) + 13*a(P))/28;
%
FigureCount = FigureCount + 1;
figure(FigureCount)
plot(N,Y,'-ko',N,a,'-r')
xlabel('Number of cycles, N (cycles)','FontSize',Font_Size)
ylabel('Crack length, a (mm)','FontSize',Font_Size)
title('a vs. N from smooth.m','FontSize',Font_Size)
legend('smoothed','original','Location','NorthWest')
set(gca,'FontSize', Font_Size)
grid on
%
% print file with N vs. Y(a, average of 7 points)
temp = fopen('Na_by_CTVA_smooth.txt', 'wt'); %write text
for i = 1:P %Number of data points
    fprintf (temp, '%-20f \t %-20.10f \n', N(i), Y(i));
    % cycle number tab crack length new line
end
fclose(temp);
%

```

```

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% End of smooth.m
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%

```

B-3: block.m

```

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
% Program block.m
% By Kimberly Maciejewski
% Created on August 7, 2007
% This Program is based on file block2.m by Dian Zheng that takes smoothed
% a vs N data and reduces the number of data points
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
% Clear Command Window
clc; % to give you a clean screen
% Clear Workspace
clear; % to clear any previously stored variables
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
FigureCount = 0; % Initialize Figure Count
Font_Size   = 20; % Set Font Size
%
% Input initial values at the beginning N = 0 cycles,
% and final values at N = final cycles
Ni = 0 ; % (cycles) Number of Cycles at t = 0
Nf = 7215.7143; % (cycles) Number of Cycles at t = final
ai = 5.966 ; % (mm) Crack Length at t = 0 (After Precracking)
af = 14.682 ; % (mm) Crack Length at t = final
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
% Call N and a from smoothed data file
Atemp = load('Na_by_CTVA_smooth.txt');
% create column vector N and column vector a
N = Atemp(:,1); % (cycles)
A = Atemp(:,2); % (mm) crack length
%
P = size(N); % Total Number of Data points
P = P(1,1);
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
% Define Block
% Example: 1 2 3 4 5 6 7 8 9
% #1 Block size 4 will take points 1 through 4
% #2 Increment 2 will advance to 3 and take points 3 through 6
% #3 will be points 5 through 8, etc
b2 = 4; % 6The smallest block size (default=4)
d1 = 3; % 4The increment of each block (default=2)
%
s1 = (1-2.*b2/d1);
s2 = s1^2.;
s3 = 8.*P/d1;
p1 = (s1+sqrt(s2+s3))/2.;
i3 = fix(p1); % fix Rounds the number to the integer part, 1.3 to 1, 1.9 to 1
% Initialize Numbers for Counting
i = 0; % initialize i
v1 = 0; % initialize v1

```

```

bb = 0; % initialize bb
jj = 1; % initialize jj (count for number of reduced data points)
%
% Calculate Reduced N and a
while bb <= P
    if i3 >= 0
        b1 = fix(b2+(i3-1)*d1);
        for k = 1:b1
            v1 = v1 + A(bb+k);
            if k == b1
                v1 = v1/b1;
            end
            if (bb+k) > P
                v1 = v1/k;
                exit;
            end
        end
        N1(jj) = ((N(bb+1)+N(bb+b1))/2); % Reduced Cycle Number N
        A1(jj) = v1; % Reduced Crack Length a
        jj = jj+1; % Step jj (count for number of reduced data points)
    else
        break
    end
    v1 = 0;
    i3 = i3 - 1;
    bb = bb + b1;
end
P_reduced = jj - 1; % Number of points (without initial and final)
% Add in N=0 and a0, and N=final and afinal
NR(1) = N(1); % Initial Cycle
NR(P_reduced + 2) = N(P); % Final Cycle
AR(1) = A(1); % Initial Crack Length a0
AR(P_reduced + 2) = A(P); % Final Crack Length af
for k = 1:(P_reduced)
    NR(k+1) = N1(k);
    AR(k+1) = A1(k);
    k = k + 1; % Step k
end
%
FigureCount = FigureCount + 1;
figure(FigureCount)
plot(N,A,'-r',NR,AR,'-ko',Ni,ai,'bx',Nf,af,'bx')
xlabel('Number of cycles, N (cycles)','FontSize',Font_Size)
ylabel('Crack length, a (mm)','FontSize',Font_Size)
title('a vs. N from block.m','FontSize',Font_Size)
legend('smoothed','blocked','Initial Measured',...
       'Final Measured','Location','NorthWest')
set(gca,'FontSize', Font_Size)
grid on
%
disp('*****')
disp('If the blocked data does not match well with the')
disp('initial and final crack length measured from the')
disp('fracture surface, then go back to CTVA.m and')
disp('choose another Vi and Vf when converting the V to a data')
disp('and repeat the smooth.m and block.m')
disp('*****')
disp('If the slope da/dN is not always increasing, you must block data')
disp('differently, also da/dN cannot be negative')
disp('*****')
%
% print file with N vs. a (reduced number of points)

```

```

temp = fopen('Na_by_CTVA_smooth_block.txt', 'wt'); %write text
for i = 1:(P_reduced+2) %Number of data points
    fprintf (temp, '%-20f \t %-20.10f \n', NR(i), AR(i));
    % cycle number tab crack length new line
end
fclose(temp);
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% End of block.m
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%

```

B-4: DK_dadN_Kmax_dadt.m

```

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
% Program DK_dadN_Kmax_dadt.m
% By Kimberly Maciejewski
% Created on August 7, 2007
% This Program is based on file DADN_3.m by Dian Zheng that calculates
% da/dN vs. delta K and da/dt vs. Kmax
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
% Clear Command Window
clc; % to give you a clean screen
% Clear Workspace
clear; % to clear any previously stored variables
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
FigureCount = 0; % Initialize Figure Count
Font_Size = 20; % Set Font Size
%
% Enter Specimen Dimensions and Forces
pmax = 7600.; % (N) Pmax
pmin = 760.; % (N) Pmin
B = 6.320; % (mm) Thickness of the specimen
W = 25.514; % (mm) Width of the specimen
period = 303; % (sec) time/cycle
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
% Call N and a from blocked data file
Atemp = load('Na_by_CTVA_smooth_block.txt');
% create column vector N and column vector a
N = Atemp(:,1); % (cycles)
A = Atemp(:,2); % (mm) crack length
%
P = size(N); % Total Number of Data points
P = P(1,1);
%
for ii = 2:P-1
    % Adjusts for the fact that da and dN do not increase by a fixed
    % number, meaning the data we get da could be 1, then 3 then 2, etc.
    % a2-a1 + ( a2-a1 * (a3-a2 - a2-a1) )
    % N2-N1 ( a3-a1 (N3-N2 N2-N1) )
    dadn(ii)=(A(ii)-A(ii-1))/(N(ii)-N(ii-1))+...
        (A(ii)-A(ii-1))/(A(ii+1)-A(ii-1))*...
        ((A(ii+1)-A(ii))/(N(ii+1)-N(ii))-...
        (A(ii)-A(ii-1))/(N(ii)-N(ii-1))); % (mm/cycle)
    aW = A(ii)/W; % (Ratio) Calculate a/W Ratio
    aaa(ii)=A(ii); % (mm) New Array aaa for Crack Length
end

```

```

NN(ii)=N(ii); % (cycles) New Array NN for Cycle Number
% Calculate Mode I Stress Intensity Factor for Compact Tension
% Specimen (dK)
%
% Fracture Mechanics Fundamentals and Applications
% by T.L. Anderson, CRC Press, 1991, pg 76
%
% f(a/W) specific formula for compact tension specimen geometry:
% f(a/W) = ((2 + a/W) / (1-a/W)^(3/2)) * (0.886 + 4.64*(a/W)...
%         - 13.32*(a/W)^2 + 14.72*(a/W)^3 - 5.60*(a/W)^4)
% Stress Intensity Factor: dK = f(a/W) * (pmax - pmin) / B / sqrt(W)
faW = ((2.+aW)*(.886+4.64*aW-13.32*aW^2+...
        14.72*aW^3-5.6*aW^4))/(1.-aW)^1.5; % (Unitless)
delk(ii)=(faW*(pmax - pmin))/(B*sqrt(W)); % (N/mm * mm^0.5)
end;
%
% Convert delk and da/dN to m and MPa Units
delk = delk/sqrt(1000); % (MPa*m^0.5)
dadn = dadn/1000; % (m/cycle)
% Convert delk and da/dN to Kmax and da/dt (assuming R = 0.1)
Kmax = delk/0.9;
dadt = dadn/period;
%
FigureCount = FigureCount + 1;
figure(FigureCount)
loglog(delk,dadn,'-ko');
xlabel('\DeltaK (MPa\surdm)','FontSize',Font_Size)
ylabel('da/dN (m/cycle)','FontSize',Font_Size)
title('da/dN vs. \DeltaK','FontSize',Font_Size)
xlim([30 100])
set(gca,'XTick',[10 20 30 40 50 60 70 80 90 100 110 120 130 140 150])
set(gca,'XTickLabel',[10 20 30 40 50 60 70 80 90 100 110 120 130 140 150])
set(gca,'FontSize', Font_Size)
grid on
%
FigureCount = FigureCount + 1;
figure(FigureCount)
loglog(Kmax,dadt,'-ko');
xlabel('K_m_a_x (MPa\surdm)','FontSize',Font_Size)
ylabel('da/dt (m/sec)','FontSize',Font_Size)
title('da/dt vs. K_m_a_x','FontSize',Font_Size)
xlim([30 100])
set(gca,'XTick',[10 20 30 40 50 60 70 80 90 100 110 120 130 140 150])
set(gca,'XTickLabel',[10 20 30 40 50 60 70 80 90 100 110 120 130 140 150])
set(gca,'FontSize', Font_Size)
grid on
%
% print file with crack growth rate data
temp = fopen('dk_dadN_Kmax_dadt.txt', 'wt'); % write text
for i = 2:(P-1) % 1 is 0 so we want to start with 2 to Number of data points
    fprintf(temp, '%-20f \t %-20.10f \t %-20f \t %-20.10f \n',...
        delk(i), dadn(i), Kmax(i), dadt(i));
    % crack length tab delta k tab dadn new line
end
fclose(temp);
%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% End of DK_dadN_Kmax_dadt.m
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%

```


APPENDIX C

CODES USED FOR SIMULATIONS

This appendix contains a list of codes and descriptions of the files used for uniaxial strain controlled simulations, as well as, intergranular crack growth simulations.

C-1: Codes for one-dimensional uniaxial simulations using the ISV model

This appendix contains a list and short description of the main codes for one-dimensional uniaxial simulations using the internal state variable (ISV) type model (detailed in Chapter 5).

1. **rhab_mon_cyc.m** - The MATLAB script to run ODE for model under strain controlled conditions corresponding to the various sets listed in Chapter 5 including monotonic, stress relaxation and cyclic loading conditions. Once this is run it creates text files with the numerical data.
2. **chab_cyc.m** - The MATLAB function file, to be integrated outside the function with an ODE solver. This function file is called by the run file rhab_mon_cyc.m. This function file contains the rate forms of the hardening laws for the ISV material model.
3. **sqr.m** - The MATLAB function file, to be called for the strain rate calculation in the chab_cyc function file.

C-2: Codes for two-dimensional uniaxial simulations using the ISV model

This appendix contains a list and short description of the main codes for two-dimensional uniaxial simulations using the internal state variable (ISV) type model (detailed in Chapter 5).

1. **ISV.for** - The fortran file which contains the UMAT subroutine for the two-dimensional ISV material model. This contains the hardening formulations, as

developed in Chapter 5, along with other user defined subroutines including an integration scheme.

2. **OneElAxisSym.inp** - The ABAQUS input file for one element axisymmetric model, to be used with ISV.for for two-dimensional uniaxial simulations.
3. **Strain_Amp_Loading.m** - The MATLAB file to create strain controlled loading profile that is applied on the axisymmetric element. This file will define the strain controlled conditions corresponding to the various sets listed in Chapter 5 including monotonic, stress relaxation and cyclic loading conditions. Once this is run it creates text file with the amplitude data set which can be pasted into the input file.
4. **ODB_S22.py** - A python script to extract data from the ODB file once the simulation is complete.

C-3: Codes for two-dimensional uniaxial simulations using the XP model

This appendix contains a list and short description of the main codes for two-dimensional uniaxial simulations using the coarse scale crystal plasticity (XP) model, as detailed in Chapter 5.

1. **umatcrystal-kh.for** - The fortran file which contains the UMAT subroutine for the two-dimensional XP material model. The XP fortran code used in this work is based on a code developed by Huang [Huang 1991] , with the hardening formulations updated as detailed in Chapter 5, along with other user defined subroutines including an integration scheme.
2. **GrainOrientation.m** - This MATLAB file is used to generate a text file that defines the material properties for the user defined number of grains in an array. It will also assign random orientations for the grain assembly with $\pm 15^\circ$ orientation relative to the neighboring grains. The user must input the number of grains in a row, and number of grains in a column in the code.
3. **PolyCrystal_PythonInput.m** - This MATLAB file is used to generate text files which are pasted into the designated spot in the python code (GeneratePolycrystalCAE.py). The user must input the grain size, number of grains in a row, and number of grains in a column in the code.
4. **GeneratePolycrystalCAE.py** - This python code is used to generate the CAE model (PolyXModel.cae).

5. **PolyXModel.cae** - This is the CAE model for a polycrystal. It is generated by running the GeneratePolycrystalCAE.py code. The user must mesh the model here, then they can write an input file (PolyXModel.inp).
6. **PolyXModel.inp** - This is the input file for a polycrystal model, to be used with the umatcrystal-kh.for for two-dimensional uniaxial simulations.
7. **Strain_Amp>Loading.m** - The MATLAB file to create strain controlled loading profile that is applied on the polycrystalline array of grains. This file will define the strain controlled conditions corresponding to the various sets listed in Chapter 5 including monotonic, stress relaxation and cyclic loading conditions. Once this is run it creates text file with the amplitude data set which can be pasted into the input file.
8. **ODB_S22.py** - A python script to extract data from the ODB file once the simulation is complete.

C-4: Codes for cohesive zone simulation

This appendix contains a list and short description of the main codes for the intergranular crack growth simulations using the cohesive zone model. This includes both continuum (see Chapter 5) and interface modeling (see Chapter 4 and 6).

1. **CZM-SX-ISV-ENV.for** - Fortran file containing the continuum material models, interface material model and loading subroutine. The UMAT subroutine is a fortran code used with Abaqus to simulate the viscoplastic material deformation. This code is for 2-D simulations using either the internal state variable (ISV) or coarse scale crystal plasticity (XP) model, as detailed in Chapter 5. The XP fortran code used in this work is based on a code developed by Huang [Huang 1991], with the hardening formulations updated as detailed in Chapter 5. The UMAT code contains 161 state variables and 198 material parameters. The UINTER subroutine is used with Abaqus to simulate the interface traction-displacement relations considering grain boundary sliding, cavitation and environment (dynamic embrittlement by Oxygen diffusion) as detailed in Chapter 4. The UINTER code contains 11 state variables and 23 material parameters. The UTRACLOAD subroutine is used with Abaqus to simulate the loading behavior of a compact tension specimen using triangular load-time curve with holds superimposed at maximum and minimum. This subroutine is to define the loading traction at any time t during the step.
2. **ctxtal-czm.inp** - The ABAQUS input file for a compact tension specimen. This contains the XP model at the crack tip surrounded by the ISV material. The grain

boundary interface is modeled as node to node contact between the mid plane of the half compact tension specimen (as shown in Figure 6-1) and rigid plane.

3. **ODB_Nodes.py** - A python script to extract data from the ODB file once the simulation is complete.
4. **CZM.m** - A MATLAB file that plots the interface outputs.

C-5: Codes for one-dimensional uniaxial simulations using the microstructure sensitive ISV model

This appendix contains a list and short description of the MATLAB codes for one-dimensional uniaxial simulations using the microstructure explicit internal state variable type model (detailed in Chapter 7).

1. **IN100_CaseStudies.m** - The MATLAB script to run ODE for model under strain controlled conditions corresponding to a series of case studies with various γ' sizes and volume fractions.
2. **Frfun.m** - The MATLAB function file to calculate the relative fractions (f^{shear} , f^{loop}) and sizes (r^{shear} , r^{loop}) of particles in distribution P which are sheared and looped by dislocations, given by equations (7-26) and (7-27).
3. **Pfun.m** - The MATLAB function file to calculate the distribution P , given by equation (7-25).
4. **P1fun.m** - The MATLAB function file to calculate the integral in the numerator of equation (7-26) for f^{shear} and f^{loop} .
5. **P2fun.m** - The MATLAB function file to calculate the integral in the numerator of equation (7-27) for r^{shear} and r^{loop} .
6. **chab_cyc.m** - The MATLAB function file, to be integrated outside the function with an ODE solver. This function file is called by the run file IN100_CaseStudy.m. This function file contains the rate forms of the hardening laws for the microstructure explicit ISV material model.
7. **sqr.m** - The MATLAB function file, to be called for the strain rate calculation in the chab_cyc function file.

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