THE EFFECTS OF MICROSTRUCTURE EVOLUTION ON DEFORMATION AND DAMAGE MECHANISMS DURING CREEP-FATIGUE TESTING OF ALLOY 709

by
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A thesis submitted to the Faculty and the Board of Trustees of the Colorado School of Mines in partial fulfillment of the requirements for the degree of Doctor of Philosophy (Metallurgical and Materials Engineering).

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ABSTRACT

The improved economics and safety for next generation nuclear reactors depend, in part, on improved performance of advanced structural materials. Power plant structural components, for both fossil and nuclear fuels, are subject to thermal low cycle fatigue (LCF) during transient start-up and shut-down operations, and to creep during steady-state operation. Hence, the interaction of fatigue and creep degradation mechanisms (i.e. creep-fatigue) has been identified as a potential failure mode under such conditions. Alloy 709, a 20Cr-25Ni-1.5Mo-Nb-N solid solution and precipitation strengthened austenitic stainless steel, is a candidate material for structural components in Gen IV sodium cooled fast spectrum reactors (FSRs) due to its improved creep resistance over current qualified structural alloys. However, there are limited data on the creep-fatigue performance of this alloy. The objective of this research is to understand the deformation and damage mechanisms responsible for failure under creep-fatigue conditions relevant to nuclear service conditions.

Laboratory creep and creep-fatigue tests are typically performed under conditions where damage is accumulated in an accelerated manner; i.e. at stresses and temperatures higher than expected during service. Initial strain-controlled LCF and creep-fatigue tests were conducted at the expected service temperature of 550 °C, and at a higher accelerated test temperature of 650 °C. The results indicated that the deformation mechanisms and damage were significantly different between the two test temperatures, which resulted in particularly poor creep-fatigue performance at 550 °C. The reduction in number of cycles to failure in creep-fatigue relative to LCF was significantly greater at 550 °C, compared to 650 °C, despite a higher creep resistance at the lower temperature. Additionally, the microstructural evolution of solution annealed (SA) material, namely precipitation of carbides and nitrides during testing, was shown to be significantly different at 550 and 650 °C.

The effect of microstructural evolution on deformation and damage mechanisms in creep-fatigue was further investigated with SA material across a wider range of test temperatures (500 to 700 °C) and tensile hold times (0 to 30 min). Under conditions where dynamic precipitation is significant, a transition in slip behavior from planar to wavy leads to dynamic recovery at grain boundaries and a reduction of intergranular damage propagation as plastic deformation is enhanced at crack tips. Easier cross-slip is attributed to a consumption of solute atoms due to precipitation. Cyclic plastic deformation is also enhanced as precipitates coarsen.
during testing. The greatest number of cycles to failure occurred where the accumulation of grain boundary creep damage (i.e. voids) was minimized and/or balanced with a large magnitude of cyclic plastic deformation.

To further investigate the effect of microstructure evolution on creep-fatigue performance, a static aging treatment was used to produce a significant volume fraction of carbides and nitrides prior to creep-fatigue testing at 550 and 650 °C. Aging the alloy resulted in a three-fold increase in creep-fatigue life over the SA material at 550 °C, which was accompanied by more dynamic recovery and approximately the same amount of creep deformation per cycle compared to the SA condition. The significant improvement in creep-fatigue life at the expected FSR service temperature (550 °C) in an aged condition is encouraging for the applicability of Alloy 709 as a structural alloy. However, the results of this study indicate that the creep-fatigue performance during accelerated testing from a SA condition is not representative of long-term service performance due to the effects of microstructure on creep-fatigue deformation and damage mechanisms.
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ACKNOWLEDGEMENTS

This project was, in many ways, a team effort which would have not been possible without the support of the MME faculty and staff, colleagues, friends, and family. I would first like to express my gratitude to the ASPPRC and Karen Abt for the opportunity to participate in and contribute to this excellent research community. This research would not have been possible without the support and funding from the U.S. Department of Energy Nuclear Energy University Program (DOE-NEUP).

I would like to thank my thesis committee for support and guidance along this journey and my transition from mechanical engineer to metallurgist, especially Drs. Richard Wright and Mike Kaufman for all of the thoughtful discussions and ideas along the way. I would like to extend a special thanks to my advisor Dr. Kip Findley for guiding my development as a researcher and a professional, and for his endless patience, confidence, and encouragement.

This research would not have been possible without the support from Idaho National Laboratory (INL), specifically the use of the fatigue testing frames and the staff. A special thanks to Joel Simpson for his meticulous attention and care when setting up and running the fatigue tests and to Mike McMurtrey for his thoughtful discussions throughout.

I would like to especially thank my collaborators Drs. Ondrej Muransky and Zhiyang Wang at the Australian Nuclear Science and Technology Organization (ANSTO) and to the Australian Government for the funding to conduct our neutron experiments.

Several other people contributed to this work directly, or indirectly through guidance or training. Olivia DeNonno contributed significantly through hardness testing and metallography. Drs. Jonah Klemm-Toole, Bob Field, Dave Diercks, and Jie Song, and Tom Gallmeyer, all guided me with electron microscopy. Gary Zito developed countless TEM films. Matt Zappulla and Henry Geerlings helped me to develop the code used to process fatigue data in this study.

I am deeply grateful for the support of my family and friends, who provided support and encouragement throughout this process. A special thanks to my parents, Dave and Monique Porter, who taught me to solve problems, think critically, ask questions, and have always encouraged me to experiment.

Finally, and most importantly, I would like to thank my best friend and my wife, Tara Porter for her unconditional love and support, her confidence and inspiration, her endless patience, and for talking me off the ledge and keeping me sane throughout this journey.

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CHAPTER 1
INTRODUCTION

The improved economics and safety for next generation nuclear reactors depend, in part, on improved performance of advanced structural materials. Power plant structural components, for both fossil and nuclear fuels, are subject to complex stress cycles at elevated temperatures, which necessitates the use of highly corrosion- and creep-resistant materials. Alloy 709, a 20Cr-25Ni-1.5Mo-Nb-N solid solution and precipitation strengthened austenitic stainless steel, is a candidate material for structural components in Gen IV sodium cooled fast spectrum reactors (FSRs) due to its improved creep resistance over current structural alloys qualified for nuclear applications, Type 316 stainless steel and Grade 91 [1.1].

Currently, Alloy 709 is only qualified by the American Society of Mechanical Engineers (ASME) for use in fossil boiler applications [1.2]. Since current applications for this alloy are at temperatures greater than 650 °C, most of the available creep and microstructural data has been generated at 650 °C or above [1.3]. The maximum expected service temperature for structural components in FSRs is 550 °C. Components in FSR applications, such as pressure vessels, are made from plate product, which is in contrast to the tubular form that is currently in use in fossil boiler applications. Since the interaction of creep and fatigue damage is a potential cause of failure in nuclear applications, an analysis of creep-fatigue performance is required for ASME code qualification. However, this type of analysis is not required for fossil boiler applications, so little creep-fatigue data has been generated for Alloy 709. Therefore, significant gaps exist in the understanding of Alloy 709 mechanical properties and microstructural evolution under conditions relevant to nuclear service.

Accelerated creep and creep-fatigue testing, by means of testing at higher temperatures and larger stresses/strains than expected during service, is typical for generating a statistically significant amount of data in a reasonable time. Accelerated test data is then extrapolated to predict failure under less severe service conditions. Concerns for extrapolation of test data arise when different deformation and/or damage mechanisms are active across the conditions in which data is generated and those expected during service. Such extrapolation may result in non-conservative predictions of damage, resulting in premature failure [1.4], [1.5]. Additionally,
mechanical properties change with an evolving microstructure, especially in highly alloyed austenitic stainless steels, such as Alloy 709. The microstructural constituents, and therefore properties, during accelerated testing may differ significantly from those expected after long exposures to service temperatures.

The scope of this research is to develop an understanding of cyclic deformation and damage mechanisms of Alloy 709 under various testing conditions in order to provide a basis for confidently extrapolating short-term laboratory test data to long-term service conditions. Specifically, this research is focused on elevated temperature low cycle fatigue (LCF) and creep-fatigue properties, the interaction of fatigue (cycle dependent) and creep (time-dependent) deformation and damage mechanisms, and the correlation of mechanical behavior to microstructural evolution. A mechanistic understanding of the mechanical properties at various conditions will provide a pathway for careful design of laboratory testing and data extrapolation in the development of an ASME code case for the alloy, which is necessary prior to its adoption for use in nuclear pressure vessel applications [1.6]. This research is part of a multi-university collaborative effort, funded by the US Department of Energy Nuclear Energy University Partnership (DOE-NEUP) program, to establish an understanding of the material properties of Alloy 709 and their evolution during long-term nuclear service.

1.1 Research Objectives

The objective of this investigation is to understand the deformation and damage mechanisms responsible for creep-fatigue failure at the relevant service temperature of 550 °C and a higher accelerated test temperature of 650 °C, and the influence of microstructural evolution on creep-fatigue performance. Two research questions are posed to fulfill the broad research objective:

1. How do the cyclic and time-dependent deformation mechanisms at 550 °C, compared to 650 °C, affect the cyclic stress-strain response, internal damage formation and propagation, and reduction in creep-fatigue life compared to pure LCF?
2. How does initial microstructure and microstructural evolution (specifically precipitation) of Alloy 709 affect LCF and creep-fatigue behavior in accelerated lab testing compared to the behavior expected during long-term service exposure?
1.2 Dissertation Outline

The results and discussion chapters (4 through 7) of this dissertation are written and organized to be submitted as journal papers, with minor adjustments to make each chapter a stand-alone manuscript outside of this document. Chapter 4 was published in its entirety in the International Journal of Fatigue in February 2019.

Chapter 2 provides the background and motivation for this research. An introduction is presented on creep-fatigue and materials used in high-temperature structural applications for the energy generation industry. Relevant strengthening mechanisms and microstructure evolution of high-temperature alloys are also reviewed.

Chapter 3 details the experimental methods and analyses used in this study to address the research questions posed in Chapter 1. While Chapters 4 through 7 provide some experimental details, as they are intended to be stand-alone manuscripts to submit for publication, this chapter provides a greater level of detail of the experimental and analytical methods used in this investigation including materials used, testing matrix and methods, sample preparation, characterization techniques, and methods for analyzing fatigue data.

Chapter 4 is a preliminary study that focuses on the LCF and creep-fatigue performance at 550 and 650 °C for two different microstructures. Significant differences in cyclic behavior and life at the two temperatures are discussed with respect to deformation and microstructure evolution. The results from this initial investigation provided the framework for subsequent chapters, specifically through the formation of the research questions previously mentioned.

Chapter 5 is a study on the microstructural evolution of Alloy 709 during isothermal static aging. Precipitates are characterized using transmission electron microscopy (TEM) and small angle neutron scattering (SANS) for a range of aging conditions. The results are compared with numerical precipitation simulations conducted using Thermo-Calc® and TC-Prisma®. Additionally, the precipitate morphology and volume fractions from the static aging experiments are compared to those in the LCF and creep-fatigue microstructures. The results are discussed with respect to extrapolation of accelerated laboratory test data.

Chapter 6 builds upon the results of Chapter 4, focusing specifically on a wider range of temperatures and hold times for creep-fatigue testing. Many of the same characterization techniques are used in this study that are discussed in Chapter 4. A method for analyzing the
different components of stress (i.e. friction and back stress) is presented and applied to the data in this study.

Chapter 7 builds upon the results of Chapters 4, 5, and 6, focusing specifically on the effect of microstructure evolution on the creep-fatigue deformation and damage mechanisms in order to support the hypotheses presented in earlier studies. Using the results from Chapter 5, a microstructure with a stable precipitate population was produced by static aging prior to creep-fatigue testing. The same characterization and analytical methods used in Chapter 6 are used in this investigation.

Chapter 8 provides a summary of the results and conclusions of this study with respect to the specific research objectives.

Chapter 9 provides several possible pathways for future work based on the results of this study.

1.3 References


CHAPTER 2
BACKGROUND AND LITERATURE REVIEW

2.1 Creep-Fatigue Review

Structural components in the power generation industry are subject to complex loading cycles as a result of thermal cycling. Dynamic loading occurs due to large thermal gradients that typically exist during the start-up (heating) and shut-down (cooling) of the power plant. A schematic illustration of the thermal path in a power plant component, and the resulting strain path on the component surface, as a result of start-up, steady-state operation, and shut-down is shown in Figure 2.1 (a) [2.1]. During the transient operations (i.e. start-up and shut-down), a thermal gradient is introduced through thick-section components, as illustrated in Figure 2.1 (b). There is a large thermal expansion on the hot side of the component. However, the surface is constrained by the bulk material, which results in a compressive strain on one side of the component, a tensile strain on the other side, and a gradient through-thickness. Hence, the problem of thermal fatigue in structural components is strain-controlled rather than stress-controlled. Due to the complexities of replicating thermal fatigue in the laboratory, strain-controlled mechanical cycling, or low cycle fatigue (LCF), at isothermal conditions are typically used to simulate the service conditions. During the steady-state operation at elevated temperature, components such as pressure vessels or boiler tubes are exposed to static stresses, which introduces a time-dependent deformation component; i.e. creep. The steady-state period is represented by a constant strain dwell period, or hold time, in a laboratory test. Therefore, a complete thermal cycle of a power plant component is subject to a fatigue component and a creep component; hence, a creep-fatigue interaction.

Fatigue testing is often represented by continuous symmetrical cycling with equal strain rates in tension and compression. However, other waveforms exist including fast-slow and slow-fast cycles, which have higher strain rates in tension and compression, respectively. Failure in pure fatigue occurs in three stages: (I) crack nucleation at a free surface, (II) crack growth approximately normal to the load, and (III) final overload fracture from microvoid coalescence. Stages I, II, and III are respectively characterized by a featureless regime, striated regime, and ductile overload. Pure fatigue damage is generally observed as transgranular in nature.
Creep testing is generally carried out under constant load (i.e. stress) conditions at elevated temperatures (>0.4Tm). Similarly to fatigue, creep is typically described in three stages: (I – primary creep) a transient period of decreasing creep rate as a result of strain-hardening in the material, (II – secondary creep) a steady-state period of constant creep rate due to the balance of strain-hardening and recovery, and (III – tertiary creep) an increase in creep rate when recovery of the material is faster than strain-hardening or if there is a decrease in cross section due to necking or intergranular cracks. Creep damage is characterized by nucleation, growth, and coalescence of grain boundary cavities, leading to intergranular fracture. Several creep cavity nucleation mechanisms have been suggested: impingement of slip bands on grain boundaries, nucleation of cavities at grain boundary ledges, cavity nucleation at grain boundary particles, and nucleation of cavities at triple junctions (wedge-type) and on grain boundaries (round-type) [2.1].

The synergistic effects of fatigue and creep damage typically result in a shorter life compared to pure fatigue (cycles to failure) or pure creep (time to rupture). Therefore, the creep-fatigue interaction has been identified as an important failure mode for high temperature power plant components. Fatigue is generally characterized by transgranular fracture and creep damage is primarily intergranular (i.e. grain boundary cavitation), while the interaction of the two mechanisms is typically mixed in nature, as shown schematically in Figure 2.2. The interaction of the two failure modes results in some combination of three possible mechanisms:
(1) cavitation damage enhanced by cyclic loading, (2) crack initiation enhanced by cavitation damage, and (3) crack propagation enhanced by cavitation damage [1, 2].

Figure 2.2 Schematic fracture paths for fatigue dominated failure, creep-fatigue damage interaction, and creep dominated failure. Adapted from [2.2].

2.2 Creep-Fatigue Life Prediction

Reliable predictions of failure under various degrees of creep-fatigue interaction are of utmost importance for the safe design of structural components in fossil and nuclear fueled power plants. Although there is no single unifying life-prediction method, several accepted models have been compared and evaluated on their predictive capability based on extrapolation of laboratory test data [2.3]–[2.5]. This brief review will focus on three of the more commonly used approaches: strain-range partitioning, ductility exhaustion, and linear damage summation. An emphasis is placed on some of the more commonly considered variables in creep-fatigue testing including temperature, hold time, hold position, strain-rate, and strain range.

2.2.1 Strain-Range Partitioning (SRP) Method

Creep-fatigue analysis by strain-range partitioning (SRP), which was first proposed by Manson et al. [2.6] in 1971, is based on the premise that inelastic deformation at elevated temperatures is a result of two different mechanisms: plastic flow and creep. Any fully reversed inelastic strain range, $\Delta \varepsilon_p$, can be partitioned into four distinct components:

\[
\begin{align*}
\Delta \varepsilon_{pp} & = \text{tensile plastic flow reversed by compressive plastic flow} \\
\Delta \varepsilon_{cp} & = \text{tensile creep reversed by compressive plastic flow} \\
\Delta \varepsilon_{pc} & = \text{tensile plastic flow reversed by compressive creep} \\
\Delta \varepsilon_{cc} & = \text{tensile creep reversed by compressive creep}
\end{align*}
\]
The possible components of inelastic deformation in a hysteresis loop are tensile plastic flow, compressive plastic flow, tensile creep deformation, and compressive creep deformation, as illustrated in Figure 2.3. Any given inelastic strain range may be comprised of one, two, or three of the listed components ($\Delta\varepsilon_{pp}$, $\Delta\varepsilon_{cc}$, and $\Delta\varepsilon_{cp}$ or $\Delta\varepsilon_{pc}$), and the sum of the partitioned components must be equal to the total inelastic strain range, or the width of the hysteresis loop. In the example hysteresis loop, the total inelastic strain range is a-d. $\Delta\varepsilon_{pp}$ is the smaller of the two plastic components (a-c and d-b); in this case $\Delta\varepsilon_{pp}$ is a-c. Likewise, $\Delta\varepsilon_{cc}$ is the smaller of the two creep components (c-d and b-a); in this case $\Delta\varepsilon_{cc}$ is b-a. The difference between the two creep components is either $\Delta\varepsilon_{pc}$ or $\Delta\varepsilon_{cp}$ depending on which is larger; in this case the final component is $\Delta\varepsilon_{cp}$ (c-d minus b-a).

![Figure 2.3 Schematic hysteresis loop showing all possible components of inelastic deformation in the total plastic strain range, $\Delta\varepsilon_{p}$ (a-d), in a strain-controlled LCF test.](image)

Once partitioned, each of the components can then be analyzed individually to determine its unique relation to cyclic life. Though this method does not rely on a specific form of the relation between the individual strain range components and cyclic life, it is typically assumed to follow the Manson-Coffin relation, which takes the following form

$$N_{ij}\Delta\varepsilon_{ij}^\alpha = C$$

where $N_{ij}$ is the number of cycles for a given partitioned strain range, $\Delta\varepsilon_{ij}$. $\alpha$ and $C$ are unique fitting parameters for each component, determined from a plot of $\log(\Delta\varepsilon_{ij})$ versus $\log(N_{ij})$. 

8
There typically exists an upper bound on cyclic life, $\Delta \varepsilon_{pp}$, and a lower bound, $\Delta \varepsilon_{cp}$, for a given strain range. It is generally accepted that the introduction of creep strain to LCF decreases the cyclic life, and that typically tensile creep reversed by compressive plastic flow is the most damaging [2.1]. An estimation of the total number of cycles to failure, $N$, is then a summation of the number of cycles to failure due to each component, given by

$$\frac{N}{N_{pp}} + \left( \frac{N}{N_{cp}} \text{ or } \frac{N}{N_{pc}} \right) + \frac{N}{N_{cc}} = 1 \quad 2.2$$

One of the potential benefits to the SRP approach to cyclic life prediction is that it may be insensitive to test temperature for certain alloys. Halford et al. [2.7] examined the effects of temperature on the SRP approach for two different high temperature alloys, 2½Cr-1Mo and Type 316 stainless steels. The temperature independence of this method is demonstrated by considering a series of LCF tests at a constant strain rate over a range of temperatures. At the low temperatures, flow behavior is predominantly plastic and the strain range is only composed of $\Delta \varepsilon_{pp}$. In contrast, at higher temperatures the flow behavior is mostly creep, and the strain range is primarily composed of $\Delta \varepsilon_{cc}$. These strain ranges represent the bounds of the cyclic life. Therefore, fatigue lives are highly temperature and strain-rate sensitive. However, the failure criteria is temperature and rate independent since the cyclic life is governed by the flow behavior (i.e. the amount of each type of strain present). The implications of these results are that failure criteria can be determined from a set of tests at one temperature, with only spot checks at the different temperatures of interest for some materials. Figure 2.4 (a) shows the good agreement between predicted versus observed creep-fatigue lives at different test temperatures for Type 316 stainless steel based on the SRP method, illustrating the temperature independence of this failure criterion.

### 2.2.2 Ductility Exhaustion (DE) Method

The ductility exhaustion (DE) method has been used extensively in the UK for life-time predictions of components in both fossil and nuclear power plants [2.4], [2.5]. The creep and fatigue damage components are evaluated separately and life is estimated by a damage interaction diagram. The cycle dependent damage component, $D_i$, is determined simply by the number of cycles to failure in pure fatigue, $N_{fo}$, according to
The time-dependent creep damage, $D_c$, is evaluated as a function of creep ductility, $\delta$, and inelastic strain-rate, $\dot{\varepsilon}_{in}$, according to

\[
D_c = \int_0^{t_h} \frac{\dot{\varepsilon}_{in}}{\delta(\dot{\varepsilon}_{in}, T)} dt \tag{2.4}
\]

where $t_h$ is the hold time and $T$ is the test temperature. The creep ductility is a function of strain rate and test temperature determined from constant load creep and tensile test data. The interaction of the two damage components is determined by a linear summation such that the creep-fatigue life, $N_{fo}$, is given by

\[
D_f + D_c = \frac{1}{N_{fo}} \tag{2.5}
\]

The DE method has been shown to have the highest degree of accuracy among several methods reviewed, which is primarily due to the large amount of creep damage predicted at small strain ranges [2.5]. Figure 2.4 (b) shows very good agreement between the predicted and experimental life in creep-fatigue for Type 316 stainless steel tested at 550 °C with different tensile hold times and strain ranges [2.5]. It should be noted that the accuracy of this life prediction method relies on creep ductility data for long-time, slow strain rate tests.

### 2.2.3 Linear Damage Summation (LDS) Method

Similar to the DE method, the linear damage summation (LDS) method for predicting creep-fatigue life involves evaluating fatigue and creep damage in an uncoupled manner based on the assumption that the individual damage mechanisms are independent in nature. The ASME Boiler and Pressure Vessel Code (BPV) [2.8] has adopted this method, specifying damage to be evaluated by a linear summation of fractions of fatigue and creep damage according to the criterion

\[
\sum_j \left( \frac{n}{N_{d,j}} \right) + \sum_k \left( \frac{\Delta t}{T_{d,k}} \right) \leq D \tag{2.6}
\]
where $D$ is the allowable combined damage fraction (typically less than 1), $n$ and $N_d$ are the number of cycles of type $j$ and allowable number of cycles of the same type, respectively, and $\Delta t$ and $T_d$ are the actual time at stress level $k$ and the rupture time at that stress level, respectively.

The fatigue and creep damage terms are evaluated independently and the interaction is accounted for empirically by the $D$ term through strain-controlled creep-fatigue test data at various temperatures, strain ranges, and hold times. The main difference between the LDS and the DE methods is the way in which creep damage is evaluated; creep damage is related to the creep rupture life at a given temperature with LDS and to the creep ductility at a given temperature and strain rate with DE. Since strain-rate is not considered in the LDS method, the life predictions are typically non-conservative at low temperatures and small strain ranges where creep strain rates are typically low. Figure 2.4 (c) shows the relatively poor agreement between predicted and experimental creep-fatigue life for Type 316 stainless steel at 550 °C, particularly at the small strain ranges (higher fatigue lives) [2.5].

### 2.2.4 Creep Damage Component

In a creep-fatigue test, time-dependent damage occurs during the hold period where stress relaxes as a result of the conversion of elastic strain to inelastic strain. The inelastic strain rates during relaxation are typically on the order of those associated with creep deformation ($<10^{-4}$ s$^{-1}$) [2.2]. In both LDS and DE methods, the determination of the time-dependent damage component is based on data from constant stress creep tests (and tensile tests in the case of DE). For the LDS method, the time-dependent damage is essentially evaluated by normalizing the time at a given stress during relaxation by the time to rupture at that stress. Therefore, it is necessary to evaluate rupture times for all stresses during the relaxation period. This evaluation is commonly done using a Larson-Miller plot [2.9]. The Larson-Miller Parameter (LMP), which relates rupture time and temperature through a semi-log linear relationship, is plotted against stress in this type of plot. A Larson-Miller plot for NF709, which was generated from specimens from several heats over a range of test temperatures from 600 to 900 °C, is shown in Figure 2.5 [2.10]. This figure shows a good fit between the calculated LMP and the compiled data. A linear regression analysis of the data in this plot gives rupture time as a function of stress for a given temperature, which is described in more detail in Appendix A. Using a Larson-Miller plot generated from test data over
Figure 2.4 Experimental versus predicted creep-fatigue life by the (a) SRP approach, (b) DE method, and (c) LDS method for Type 316 stainless steel. In (a), different test temperatures, hold times, and hold time locations in the hysteresis curve are shown. $t_t$ and $t_c$ are hold times at peak tensile strain and peak compressive strain, respectively. In (b) and (c), the test temperature is 550 °C. Symbols represent different tensile hold times and the number next to the symbol represents total strain range. Adapted from [2.5].
a wide range of temperatures and stresses, one can predict the creep-rupture life under conditions that are not frequently tested, such as low temperatures and stresses.

Figure 2.5 Larson-Miller plot of NF709 constructed from stress-rupture data at various conditions. The predicted LMP values fit the experimental trend well for this alloy [2.10].

A key assumption in using the LMP approach to determine rupture time as a function of stress is that the mechanism for which creep deformation occurs is the same across all test conditions that make up the Larson-Miller plot. Creep deformation is a thermally assisted deformation mechanism which can occur by either diffusional flow of vacancies (and mass) or by climb and glide of dislocations [2.11]. The former is often referred to as diffusional creep, while the latter is called power-law creep. Diffusional creep is based on either lattice (Nabarro-Herring) or grain boundary (Coble) vacancy diffusion. Power-law creep is based on vacancy assisted climb and glide of dislocations and generally occurs at higher temperatures and higher stresses than diffusional creep. In the power-law creep regime, the stress dependence of the steady-state creep rate, $\dot{\varepsilon}_{ss}$, is described by the following power-law relation

$$\dot{\varepsilon}_{ss} = A\sigma^n \exp\left(\frac{-Q}{kT}\right)$$  

where $A$ is a dimensionless material constant, $\sigma$ is the applied stress, $Q$ is the activation energy for self-diffusion, $k$ is Boltzmann’s constant, and $n$ is the creep rate exponent, which typically ranges from 3 to 10. Therefore, the relation between steady-state creep rate and applied stress is
linear in log-log space, with the slope equal to \( n \). Note that for viscous diffusional creep, \( n \) is unity. At high stresses, the power-law relation can no longer describe the stress dependence of creep rate; this regime is often referred to as power-law breakdown [2.12]. Thus, the dominant (rate-controlling) mechanism for creep deformation is identified by the value of the creep-rate exponent.

Conditions where the dominant creep mechanism changes can be easily identified by a change in \( n \) on a plot of steady-state creep rate versus stress for a given temperature. Figure 2.6 (a) shows this type of plot, schematically, with three distinct regions of different creep mechanisms, where \( n_1 < n_2 < n_3 \). The concept of separating dominant deformation mechanisms was extended by Frost and Ashby to relate stress, strain-rate, and temperature on a single plot, separated into regions of different mechanisms [2.13], [2.14]. An example deformation mechanism map for Type 316 stainless steel is shown in Figure 2.6 (b) [2.14]. Note that the region where creep data is available is primarily in the power-law regime, while the expected conditions for several nuclear reactor components lie is in the Coble creep regime. If the steady-state creep rate is predicted for diffusional creep conditions (\( n_1 \)) from accelerated tests conducted in the power-law creep regime (\( n_2 \)), the results would be non-conservative. In other words, the extrapolated creep rates would be lower than the actual creep rates due to the change in mechanism at the inflection between lines \( n_1 \) and \( n_2 \) in Figure 2.6 (a). Rieth et al. [2.15] have concluded that the inflection in the steady-state creep rate versus stress behavior of Type 316L(N) at low temperatures and stresses, shown in Figure 2.7, is due to a transition in deformation mechanisms from diffusional to power-law creep.

One of the key objectives in studies of elevated temperature mechanical behavior is understanding the implications of using accelerated laboratory test data to extrapolate the life of components in long-term service. The non-conservative predictions of creep-fatigue life at low strain ranges based on the ASME accepted LDS method, as shown in Figure 2.4 (c), can lead to premature component failure in service. The calculated creep damage component in the LDS method does not take damage mechanism into consideration, which may be part of the reason for the poor agreement between predicted and experimental life at conditions close to those expected in service (i.e. low strain ranges, strain rates, and temperatures). Therefore, understanding the mechanisms involved in deformation and damage across a wide range of conditions is critical for safe and reliable design of power plant components.
Figure 2.6 (a) Schematic plot of steady-state creep rate, $\dot{\varepsilon}_{ss}$, versus stress, $\sigma$, in log-space with three different creep rate exponents, $n$, representing different deformation mechanisms ($n_1 < n_2 < n_3$). (b) Stress versus temperature deformation mechanism map for Type 316 stainless steel with iso-strain rate contours shown. Note that the available creep data is primarily in the dislocation creep mechanism regime, and nuclear reactor components are in the diffusional flow regime [2.14].

Figure 2.7 Steady-state creep rate as a function of stress at various temperatures for Type 316L(N) stainless steel [2.15].
2.3 Austenitic Stainless Steels for High Temperature Applications

2.3.1 General Review of Properties and Strengthening Mechanisms

Austenitic stainless steels are the most widely used class of alloys for structural applications in the power generation industry due to their excellent weldability, corrosion resistance, high temperature strength, and lower cost than nickel-based superalloys. The FCC austenite phase is typically stabilized at room temperature by significant Ni or Mn additions, which overcome the stabilization of BCC ferrite from Cr additions greater than 12 wt pct. While room temperature yield strength is similar to ferritic stainless steels, the ultimate strength, elongation, and toughness are generally better in austenitic steels. The superior creep resistance in austenitic stainless steels, compared to ferritic steels, is largely due to fact that the self-diffusivity in close-packed FCC iron is approximately two orders of magnitude smaller than in BCC ferrite [2.16]–[2.18]. Additionally, austenitic steels have comparatively higher solubility and lower diffusion rates of solute atoms, which retards precipitation kinetics and allows for solid solution strengthening to be effective at high temperatures. For example, the solubility of C in austenite is ~40 times that of ferrite at 723 °C and the solubility of N is ~20 times greater at 590 °C [2.17].

One of the drawbacks with austenitic steels is their relatively high coefficient of thermal expansion (CTE) and low thermal conductivity compared to ferritic and martensitic stainless steels. For example, the CTE for austenitic steels is approximately 50 pct higher than ferritic steels, and the thermal conductivity is approximately 33 pct lower. This combination of properties in austenitic steels results in large thermal strains and gradients in thick section components during transient temperature fluctuations (i.e. start-up and shut-down of power plants). Hence, LCF performance is an important field of study for austenitic stainless steels.

Strengthening in austenitic steels is achieved by solid solution, precipitation, grain size refinement, and cold deformation (i.e. dislocation hardening). Note that for elevated temperature performance, both grain size refinement and cold working may be detrimental. For example, the diffusional creep rate is a function of grain size since diffusion along boundaries is higher than through the matrix [2.11]. Therefore, smaller grains result in higher grain boundary surface area and higher diffusional creep rates. Cold work prior to elevated temperature service introduces dislocations and provides a high driving force for recovery and recrystallization, both of which
are detrimental to creep properties [2.19]. Therefore, solid solution and particle strengthening are the most important mechanisms in elevated temperature applications.

Solid solution strengthening in austenitic steels is achieved by alloying with both interstitial atoms, like C and N, and substitutional atoms, such as W and Mo [2.16]. Interstitial solutes interact strongly with the strain fields associated with edge dislocations in FCC metals since they occupy the octahedral sites in the lattice, which induces a hydrostatic stress state as a result of the size misfit. The elastic strain energy associated with both the interstitial and the dislocation is reduced when the solute atom is in the vicinity of an edge dislocation strain field, specifically, on the tensile side of an edge dislocation. C and N atoms consequently pin dislocations by raising the stress required for dislocation movement. Note that the movement of screw dislocations is not directly affected by interstitial atoms in FCC metals due to the purely shear stress state of screw components. Substitutional atoms, on the other hand, can interact with screw dislocations through a mismatch of the shear modulus with the solvent atoms. In general, strengthening from substitutional atoms in FCC alloys is primarily due to the size misfit interaction with edge dislocations. For both interstitial and substitutional solution strengthening, the increase in strength is proportional to the solute concentration [2.18].

Particle or dispersion strengthening in austenitic stainless steels is achieved by the addition of carbides, nitrides, intermetallic compounds, metallic phases, and oxides within the matrix. Precipitates are strong barriers for dislocation motion. Except for metallic second phases, like Cu precipitates, the particles within the matrix are usually not shearable by dislocations due to their significantly higher shear modulus compared to the matrix. Therefore, dislocations must bow between the precipitates in order to bypass the obstacles. Strengthening by hard particles is referred to as the Orowan bowing mechanism and is described by the following relation

\[ \tau_{or} = 0.84 \frac{Gb}{\lambda_s - d} \]

where \( \tau_{or} \) is the increase in resolved shear stress, \( G \) is the shear modulus, \( b \) is the magnitude of the Burger’s vector, \( d \) is the particle diameter, and \( \lambda_s \) is the particle spacing [2.20]. Particle spacing can be estimated by
where \( r \) is the particle radius (assuming a sphere) and \( f_V \) is the volume fraction. From Equations 2.8 and 2.9, it is clear that strengthening from Orowan bowing increases with larger volume fractions and finer precipitates. At sufficiently high temperatures, dislocations may also bypass precipitates by climb. Although dislocations may climb around particles, the interfacial energy of the particle with the matrix may cause an attraction with the climbing segment of the dislocation, leading to an additional threshold stress [2.20]. Similar to Equation 2.8, the threshold stress is inversely related to the particle spacing. Therefore, increases in volume fraction of small particles also increases the degree of precipitate strengthening under creep conditions.

2.3.2 Microstructural Instabilities

High-temperature properties of austenitic stainless steels are highly dependent on the microstructural evolution and stability during service, specifically the formation, dissolution, and coarsening of precipitates. In addition to Cr and Ni, austenitic stainless steels typically have a host of other alloying elements including Mn, Si, Mo, W, Nb, Ti, Cu, B, C, and N [2.17]. Austenitic alloys are usually solution annealed prior to service so that the maximum strengthening from solid solution is achieved. However, the concentration of solutes typically exceeds the solubility in austenite and therefore, other phases may be stable at service temperatures. During elevated temperature service, second phases may form which can be either beneficial or detrimental to properties, depending on the phase and location (i.e. grain boundary versus matrix). Sourmail et al. [2.21] and Lo et al. [2.22] have compiled a review of the different carbides, nitrides, and intermetallic phases that form in austenitic stainless steels, which are summarized in Table 2.1.

\[
\lambda_s = r \sqrt{\frac{2\pi}{3f_V}}
\]

\( \lambda_s \) 2.9

\( M_{23}C_6 \) is a well-documented carbide that commonly forms in austenitic stainless steel, where M is largely Cr, with varying amounts of Fe, Mo, and Ni. Its precipitation is well understood and occurs in the following order: on grain boundaries, incoherent twin boundaries, coherent twin boundaries, and intragranularly. Precipitation of this carbide can cause intergranular corrosion (i.e. sensitization) in stainless steels when it forms on grain boundaries due to the local depletion of Cr. The \( M_{23}C_6 \) carbide precipitates early during aging and has a
cube-to-cube orientation relationship with austenite and a lattice parameter that is approximately three times larger than austenite. \(\text{M}_{23}\text{C}_6\) is most beneficial for creep properties when it forms in fine dispersions intragranularly. Precipitation on grain boundaries may also reduce grain boundary sliding under creep conditions. However, since this carbide typically coarsens rapidly and causes sensitization, precipitation is typically avoided.

One common way to stabilize stainless steel against intergranular corrosion is through alloying additions of Ti, Nb, and/or V to promote MC precipitation, thereby consuming C available for \(\text{M}_{23}\text{C}_6\) precipitation. MX carbonitrides, where X is C or N, also provide good creep resistance when precipitated intragranularly on dislocations. In order to achieve maximum strengthening with MX, a solution treatment is typically performed to dissolve as much of the precipitates as possible prior to service so that subsequent precipitation occurs during service. MX has a NaCl FCC structure and precipitates on dislocations, stacking faults, and twin and grain boundaries. High temperature creep and creep-fatigue strength of several advanced austenitic stainless steels, including HT-UPS and Sanicro 25, is attributed to nano-scale MX precipitates that form on dislocations and do not coarsen significantly during testing and service [2.23]–[2.25].

Z-phase is another precipitate that provides significant creep strength when nucleated in fine dispersions on dislocations [2.21], [2.26], [2.27]. Z-phase is a complex nitride with the stoichiometry CrNbN and a tetragonal crystal structure that forms at high temperatures in Nb-stabilized austenitic stainless steels with high levels of N. It has been proposed that Z-phase form from pre-existing MX (NbN) precipitates, but it has also been reported to form directly

<table>
<thead>
<tr>
<th>Precipitate</th>
<th>Crystal Structure</th>
<th>Lattice Parameters (nm)</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>NbC</td>
<td>FCC</td>
<td>a = 0.447</td>
<td>NbC</td>
</tr>
<tr>
<td>NbN</td>
<td>FCC</td>
<td>a = 0.440</td>
<td>NbN</td>
</tr>
<tr>
<td>TiC</td>
<td>FCC</td>
<td>a = 0.433</td>
<td>TiC</td>
</tr>
<tr>
<td>TiN</td>
<td>FCC</td>
<td>a = 0.424</td>
<td>TiN</td>
</tr>
<tr>
<td>(\text{Z-phase})</td>
<td>Tetragonal</td>
<td>a = 0.3037, c = 0.7391</td>
<td>CrNbN</td>
</tr>
<tr>
<td>(\text{M}_{23}\text{C}_6)</td>
<td>FCC</td>
<td>a = 1.057 - 1.068</td>
<td>(\text{Cr}_{16}\text{Fe}_5\text{Mo}_2\text{C}_6) (e.g.)</td>
</tr>
<tr>
<td>(\text{M}_6\text{C})</td>
<td>Diamond Cubic</td>
<td>a = 1.062 - 1.128</td>
<td>(FeCr)$_{21}$\text{Mo}_3\text{C}_4, Fe$_3$Nb$_3$C, M$_5$SiC</td>
</tr>
<tr>
<td>Sigma ((\sigma))</td>
<td>Tetragonal</td>
<td>a = 0.880, c = 0.454</td>
<td>Fe-Ni-Cr-Mo</td>
</tr>
<tr>
<td>Laves</td>
<td>Hexagonal</td>
<td>a = 0.473, c = 0.772</td>
<td>Fe$_2$Mo, Fe$_2$Nb</td>
</tr>
<tr>
<td>Chi ((\chi))</td>
<td>BCC</td>
<td>a = 0.8807 - 0.8878</td>
<td>Fe$<em>{36}$Cr$</em>{12}$Mo$_{10}$</td>
</tr>
<tr>
<td>G-phase</td>
<td>FCC</td>
<td>a = 1.12</td>
<td>Ni$_{16}$Nb$_6$Si$<em>7$, Ni$</em>{16}$Ti$_6$Si$_7$</td>
</tr>
</tbody>
</table>
from solution. Although precipitation of Z-phase is not well understood and is seldomly reported, it is generally agreed that it improves creep strength due to its relatively high stability against coarsening at service temperatures.

Intermetallic compounds, such as Sigma and Laves phase, form in austenitic stainless steels after long times at elevated temperatures. While the literature is inconclusive on whether or not Laves is detrimental to creep properties, Sigma phase is well-known to cause embrittlement when it forms on grain boundaries in Fe-Cr systems \[2.21\], \[2.22\]. Precipitation of Sigma is very sluggish in austenitic steels due to its very low solubility of C and N and its incoherence with austenite \[2.28\]. Sigma formation occurs when the C concentration in the matrix is very low, which can be a result of carbide precipitation.

2.3.3 Cyclic Deformation

The cyclic stress-strain behavior of materials is characterized by hardening, softening, or a steady-state response with increasing number of cycles. Cyclic behavior is related to both loading and environmental conditions (\textit{i.e.} strain range, strain rate, temperature, etc.), as well as the microstructure (\textit{i.e.} crystal structure, second phases, solute concentration, etc.). In FCC metals and alloys, cyclic hardening is typical for annealed materials that are relatively defect free, while softening is more common for cold-worked, solute, or precipitation strengthened materials. Furthermore, the deformation behavior may change during cycling, which is related to changes in the dislocation interactions and structures. For example, the onset of cyclic softening after initial hardening has been correlated with the formation of low energy dislocation structures (LEDS) such as persistent slip bands (PSBs) or cellular structures \[2.29\], \[2.30\]. The formation of LEDS requires extensive cross-slip of screw dislocations. The deformation of FCC metals is characterized as either planar, where cross-slip is difficult, or wavy, where cross-slip is relatively easy. In general, materials with high stacking fault energy (SFE) deform in a wavy manner due to the small distance between partial dislocations. Low SFE materials, like austenitic stainless steels, usually deform in a planar manner.

However, it has been shown that this generalization of SFE and slip character does not always hold true and that low SFE austenitic steels may exhibit wavy slip during fatigue under certain conditions \[2.31\], \[2.32\]. Wavy slip can be thermally-activated or stress-induced. In Type 316 stainless steel, for example, high stresses at grain boundaries from dislocation pile-ups
can activate secondary slip systems and the formation of LEDS at room temperature [2.31]. There are also strong compositional effects on the deformation character in austenitic stainless steels. For example, N has a strong effect on the slip planarity in Type 304L(N) and 316L(N) stainless steels, where the low interstitial alloys develop LEDS and exhibit cyclic softening during LCF [2.33], [2.34]. Hong and Laird [2.35] proposed that the effect of interstitial solutes on cross-slip in FCC alloys is frictional in nature. Due to a strong strain field interaction, solute atmospheres form around the edge components of dissociated partial dislocations. In addition to the repulsive force of the same-sign partial dislocations, the force required for constriction and subsequent cross-slip includes a frictional force that is a function of solute content. This model indicates that there is a critical concentration of solutes where the slip mode transitions from wavy to planar for a given applied stress.

Dynamic strain aging (DSA) has been reported for several austenitic stainless steels during LCF at various temperatures and strain-rates, often encompassing conditions relevant to service [2.33], [2.36], [2.37]. DSA is a time-dependent phenomenon that occurs in many alloys in which solute atoms interact with mobile dislocations, leading to dynamic pinning and unpinning of dislocations during deformation. The effects of DSA generally occur over a wide range of temperatures and strain rates where the mobility of solute atoms are on the order of the dislocation velocity. In LCF, DSA is manifested in several ways including: serrations in the hysteresis loops, increase in maximum tensile stress, increase in the number of cycles to maximum stress, decrease in plastic strain range, increase in cyclic strain-hardening rate, and a reduction in the number of cycles to failure. There is an optimal temperature and strain rate combination where DSA effects are most pronounced. For a given temperature, large deviations in strain-rate away from the optimum result in a disappearance of serrated flow and other DSA related phenomena. Also, for a given strain-rate, the same can be said for deviations of temperature away from the optimum [2.19].

DSA is considered as another time-dependent mechanism, along with creep, oxidation, and metallurgical instability, which leads to a reduction in fatigue life at high temperatures and low cycle frequencies. DSA may be due to dislocation interactions with either interstitial or substitutional solutes, each with different activation energies and temperature ranges of interaction. The cyclic deformation and fracture mechanisms have also been shown to be strongly influenced by the presence of DSA. It has been shown for austenitic stainless steels in
the regime where DSA is active that slip planarity is enhanced and the formation of cells and subgrains is retarded since cross-slip and climb are inhibited by the strong dislocation-solute interactions [2.36]. Strong planar slip leads to the formation of dense slip bands and strain localization. Impingement of slip bands on grain boundaries causes stress concentrations resulting in grain boundary crack nucleation and fatigue life reduction in the DSA regime [2.1].

The cyclic deformation behavior in austenitic stainless steels is highly sensitive to microstructure, composition, and testing conditions. Predicting cyclic life from accelerated testing using the LDS method does not consider microstructural effects and their evolution on deformation behavior, similarly to the extrapolation of creep behavior across boundaries of deformation mechanisms. Therefore, understanding fatigue deformation and damage across a wide range of testing conditions and microstructures is critical for the reliable long-term extrapolation of test data to service conditions.

2.4 Alloy 709

Alloy 709 is a candidate alloy for structural components in Gen IV FSRs due to its superior creep properties over current qualified alloys Type 316 stainless steel and Grade 91 [2.38]. Alloy 709 is a 20Cr-25Ni-1.5Mo-Nb-N solid solution and precipitate strengthened austenitic stainless steel based off of NF709, which is produced by Nippon Steel (Japan) and ASME BPV code qualified for use in fossil boiler tubes [2.39]. The full composition of NF709 is shown in Table 2.2. Although the composition of Alloy 709 does not differ significantly from NF709, the target aims and thermo-mechanical processing (TMP) parameters are different.

The creep rupture strength versus time of NF709 and Type 316H at 700 °C are shown in Figure 2.8. For a given rupture time, NF709 has an order of magnitude larger creep strength compared to 316H. The improved creep properties of NF709 are attributed to solid solution strengthening from Mo and N and precipitation strengthening from fine Z-phase particles. Z-phase reportedly forms on dislocations during creep testing and resists significant coarsening after long times at high temperatures [2.26], [2.27]. The lack of detrimental phase formation, like Sigma phase, during long-term exposure is also attributed to the improved creep properties over other austenitic stainless steels. The microstructural evolution of NF709 has been studied after static aging at 750 and 800 °C for up to 10,000 h [2.40]. The results of this aging study showed that significant differences in the precipitation of Z-phase and Sigma existed for small
compositional differences in NF709. Specifically, Z-phase is more stable at higher temperatures and a significantly higher fraction of Sigma phase precipitates are observed in the variant with higher Cr and higher C. This study indicates that the microstructural features responsible for the improved properties in NF709 are sensitive to composition.

Table 2.2 Compositional Limits of NF709 (wt pct) [2.39]

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Ti</th>
<th>Nb</th>
<th>N</th>
<th>S</th>
<th>P</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.10</td>
<td>1.50</td>
<td>1.00</td>
<td>22.0</td>
<td>19.0</td>
<td>1.0</td>
<td>0.20</td>
<td>0.10</td>
<td>0.10</td>
<td>0.010</td>
<td>0.030</td>
<td>0.002</td>
</tr>
<tr>
<td>max</td>
<td>max</td>
<td>max</td>
<td>to</td>
<td>to</td>
<td>to max</td>
<td>to max</td>
<td>to</td>
<td>to</td>
<td>max</td>
<td>max</td>
<td>to</td>
</tr>
</tbody>
</table>

Figure 2.8 Creep stress rupture time for NF709 and Type 316H stainless steel tested at 700 °C. NF709 has an order of magnitude increase in strength over 316H for a given rupture time [2.38].

2.5 References


3.1 Experimental Design

The scope of this research is to investigate the cyclic deformation and damage mechanisms of Alloy 709 under accelerated testing conditions in order to confidently predict long-term creep-fatigue behavior for structural components in next generation nuclear reactors. Providing a mechanistic understanding of the mechanical properties at various conditions will allow for careful design of laboratory testing and data extrapolation in development of an ASME code case for the alloy, which is necessary prior to its adoption for use in nuclear pressure vessel applications.

The initial test matrix, shown in Table 3.1, included LCF and creep-fatigue tests at two different temperatures and with two different initial microstructures, holding strain rate, strain amplitude, and hold times constant (i.e. 0 min hold for LCF and 30 min tensile hold for creep-fatigue). Chapter 4 is dedicated to the results and analysis for the test matrix shown in Table 3.1.

Based on the results of the initial LCF and creep-fatigue tests, two specific research questions, discussed in Chapter 1, were proposed to answer the broad project objective. In order to answer the specific research questions, the test matrix was expanded to include a wider range of temperatures, hold times, strain amplitudes, and initial microstructures.

The complete creep-fatigue test matrix is shown in Table 3.2. Specifically, if a difference in time-dependent deformation mechanisms exists at 550 °C compared to 650 °C, expanding the matrix to temperatures above, below, and in-between the initial tests would provide reinforcement for this hypothesis and determine more accurately at which temperature the mechanism change occurs. Similarly, different hold time tests are designed to expose differences in time-dependent deformation at 550 and 650 °C. Finally, the effect of microstructure evolution on creep-fatigue performance is assessed by changing the initial condition of the microstructure through thermal aging, in addition to testing at different temperatures where precipitate evolution is expected to vary differently with time. Chapters 6 and 7 are dedicated to the results and analysis for the expanded test conditions, specifically with respect to the initial test results.
Experimental Materials

Several different heats of Alloy 709 were used in this study with compositions shown in Table 3.3. Small-scale experimental heats, identified as 011439, 011502, 011593 and 011594, were produced by vacuum induction melting (VIM) and electro-slag remelting (ESR) followed by hot forging and hot rolling, solution annealing at 1100 °C, and water quenching. Of the initial small-scale experimental heats, only 011593 and 011594 were used for LCF and creep-fatigue testing. Heats 011439 and 011502 were used in aging studies for microstructural analysis. Heat 011593 did not meet the microstructural specifications determined by Oak Ridge National Laboratory due to the inhomogeneity of the grain structure; thus, this plate was scrapped from further mechanical testing at the national laboratory.

The first large scale commercial heat of Alloy 709, identified as 58776, was produced with argon-oxygen decarburization (AOD) followed by hot rolling, solution annealing at 1100 °C, and water quenching. An important compositional difference between the commercial heat and the small-scale laboratory heats is the Ti content. The slightly higher Ti content in heat 58776 results in stringers of coarse cuboidal Ti(C,N) in the as-received microstructure, which are not frequently observed in the as-received microstructures of the small-scale heats with lower Ti contents. Specimens from heat 58776 were primarily used in the expanded test matrix, as detailed in Table 3.2.

All plates received for this study were nominally 25 mm thick. The length and width dimensions varied for each plate.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Tensile Hold Time (min)</th>
<th>Total Strain Amplitude, $\varepsilon_{aT}$ (pct)</th>
<th>Heat ID</th>
<th>Initial Microstructure</th>
<th>Test End Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>650</td>
<td>0</td>
<td>1.0</td>
<td>011593, 011594</td>
<td>Solution Annealed</td>
<td>To Failure</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>011593, 011594</td>
<td>Solution Annealed</td>
<td>To Failure</td>
</tr>
<tr>
<td>550</td>
<td>0</td>
<td>1.0</td>
<td>011593, 011594</td>
<td>Solution Annealed</td>
<td>To Failure</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>1.0</td>
<td>011593, 011594</td>
<td>Solution Annealed</td>
<td>To Failure</td>
</tr>
</tbody>
</table>
Table 3.2 Complete Creep-Fatigue Test Matrix

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Tensile Hold Time (min)</th>
<th>Total Strain Amplitude, ε\text{aT} (pct)</th>
<th>Heat ID</th>
<th>Initial Microstructure</th>
<th>Test End Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>700</td>
<td>30</td>
<td>1.0</td>
<td>58776</td>
<td>Solution Annealed</td>
<td>To Failure</td>
</tr>
<tr>
<td>650</td>
<td>0</td>
<td>1.0</td>
<td>011593, 011594</td>
<td>Solution Annealed</td>
<td>To Failure</td>
</tr>
<tr>
<td>650</td>
<td>5</td>
<td>1.0</td>
<td>58776</td>
<td>Solution Annealed</td>
<td>To Failure</td>
</tr>
<tr>
<td>650</td>
<td>15</td>
<td>1.0</td>
<td>58776</td>
<td>Solution Annealed</td>
<td>To Failure</td>
</tr>
<tr>
<td>650</td>
<td>30</td>
<td>1.0</td>
<td>011593, 011594</td>
<td>Solution Annealed</td>
<td>To Failure</td>
</tr>
<tr>
<td>650</td>
<td>30</td>
<td>1.0</td>
<td>011594</td>
<td>Solution Annealed</td>
<td>Interrupted at various intervals</td>
</tr>
<tr>
<td>650</td>
<td>30</td>
<td>1.0</td>
<td>58776</td>
<td>Aged at 750 °C, 100 h</td>
<td>To Failure</td>
</tr>
<tr>
<td>650</td>
<td>30</td>
<td>1.0</td>
<td>58776</td>
<td>Aged at 650 °C, 500 h</td>
<td>To Failure</td>
</tr>
<tr>
<td>650</td>
<td>30</td>
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<td>30</td>
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<td>58776</td>
<td>Solution Annealed</td>
<td>To Failure</td>
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<tr>
<td>550</td>
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<td>011593, 011594</td>
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<td>To Failure</td>
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<td>550</td>
<td>5</td>
<td>1.0</td>
<td>58776</td>
<td>Solution Annealed</td>
<td>To Failure</td>
</tr>
<tr>
<td>550</td>
<td>15</td>
<td>1.0</td>
<td>58776</td>
<td>Solution Annealed</td>
<td>To Failure</td>
</tr>
<tr>
<td>550</td>
<td>30</td>
<td>1.0</td>
<td>011593, 011594</td>
<td>Solution Annealed</td>
<td>To Failure</td>
</tr>
<tr>
<td>550</td>
<td>30</td>
<td>1.0</td>
<td>011594</td>
<td>Solution Annealed</td>
<td>Interrupted at various intervals</td>
</tr>
<tr>
<td>550</td>
<td>30</td>
<td>1.0</td>
<td>58776</td>
<td>Aged at 750 °C, 100 h</td>
<td>To Failure</td>
</tr>
<tr>
<td>550</td>
<td>30</td>
<td>1.0</td>
<td>58776</td>
<td>Aged at 650 °C, 500 h</td>
<td>To Failure</td>
</tr>
<tr>
<td>550</td>
<td>30</td>
<td>0.4</td>
<td>011594</td>
<td>Solution Annealed</td>
<td>Interrupted</td>
</tr>
<tr>
<td>500</td>
<td>30</td>
<td>1.0</td>
<td>58776</td>
<td>Solution Annealed</td>
<td>To Failure</td>
</tr>
</tbody>
</table>

Table 3.3 Compositions of Experimental Heats of Alloy 709 (wt pct)

<table>
<thead>
<tr>
<th>Heat ID</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Ti</th>
<th>Nb</th>
<th>N</th>
<th>S</th>
<th>P</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>011439</td>
<td>0.066</td>
<td>0.89</td>
<td>0.29</td>
<td>25.09</td>
<td>19.84</td>
<td>1.51</td>
<td>&lt;0.01</td>
<td>0.26</td>
<td>0.15</td>
<td>&lt;0.001</td>
<td>&lt;0.005</td>
<td>0.0026</td>
</tr>
<tr>
<td>011502</td>
<td>0.063</td>
<td>0.88</td>
<td>0.28</td>
<td>25.00</td>
<td>19.69</td>
<td>1.46</td>
<td>&lt;0.01</td>
<td>0.23</td>
<td>0.14</td>
<td>&lt;0.001</td>
<td>&lt;0.005</td>
<td>0.0022</td>
</tr>
<tr>
<td>011593</td>
<td>0.073</td>
<td>0.90</td>
<td>0.39</td>
<td>24.98</td>
<td>19.84</td>
<td>1.51</td>
<td>&lt;0.01</td>
<td>0.25</td>
<td>0.13</td>
<td>0.0008</td>
<td>&lt;0.005</td>
<td>0.004</td>
</tr>
<tr>
<td>011594</td>
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<td>0.90</td>
<td>0.39</td>
<td>25.01</td>
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<td>1.51</td>
<td>&lt;0.01</td>
<td>0.25</td>
<td>0.14</td>
<td>0.0006</td>
<td>&lt;0.005</td>
<td>0.0037</td>
</tr>
<tr>
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<td>0.91</td>
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<td>0.148</td>
<td>&lt;0.000</td>
<td>0.014</td>
<td>0.0045</td>
</tr>
</tbody>
</table>
3.2.1 Fatigue Test Specimen Machining

The specimens used for LCF and creep-fatigue testing, referred to as “button-head” specimens, were machined according to INL drawing number 771961. Details of the specimen are shown in Figure 3.1. Some of the relevant notes on the drawing are as follows:

1. Low stress grinding shall be used on the reduced section during final machining. The last 0.009 (in) removed by wet grinding and no more than 0.0002 per pass.
2. Longitudinal grinding or polishing shall be used to eliminate circumferential machining marks in reduced section.
3. Smooth blend from reduced section to fillet radius, no undercut permissible.

These specific machining instructions are provided to ensure that fatigue crack initiation will not be affected by the surface preparation of the reduced (gage) section.

Button-head specimens were machined from the as-received plates with the axial direction parallel to the rolling direction (RD) of the plate, as shown schematically in Figure 3.2. The center axis of the specimen was nominally on the center plane of the plate thickness.

3.3 LCF and Creep-Fatigue Testing

Strain-controlled LCF and creep-fatigue tests were performed on MTS servo-hydraulic frames at Idaho National Laboratory (INL) according to ASTM E2714 [3.1]. The loading in this type of test was carried out in a pull/push (tension/compression) manner along the specimen axis. All tests in this investigation were conducted in air using a symmetrical (i.e. fully reversed, \( R = -1 \)) triangular waveform, illustrated in Figure 3.3, and a strain rate, \( \dot{\varepsilon} \), equal to \( 10^{-3} \) s\(^{-1} \). The majority of the tests had a total strain amplitude, \( \varepsilon_{aT} \), of 1 pct (0.01), although a smaller strain amplitude of 0.4 pct was also investigated and is discussed in Appendix B. Various hold times, \( t_h \), were introduced at peak tensile strain in the creep-fatigue tests, as shown in Figure 3.3 (b).

The typical experimental test setup used in this study is shown in Figure 3.4. Elevated temperature was maintained using a resistance box furnace and monitored using two thermocouples spot welded outside either end of the gage section of the specimen. The specimen was held at each end using high temperature water cooled grips. Strain was measured using an air-cooled knife-edge type extensometer with a 12 mm gage length. The ceramic extensometer
Figure 3.1 Button-head test specimen drawing details for LCF and creep-fatigue testing. Dimensions are in inches.
Figure 3.2 Schematic showing the orientation of the machined specimens with the plate rolling direction (RD) and transverse direction (TD).

Figure 3.3 Schematic test profiles for (a) LCF and (b) creep-fatigue with fully reversed \((R = -1)\) strain-controlled loading. \(\epsilon_{a,T}\) is the total strain amplitude, \(t_h\) is the tensile hold time, and \(\dot{\epsilon}\) is the strain rate. Schematics are not to scale.

Figure 3.4 LCF test setup details showing a specimen instrumented with spot-welded thermocouples and a two-pronged extensometer in a servo-hydraulic testing frame at INL.
prongs reach the specimen through a small opening in the furnace. The tests that were conducted
to failure were terminated when the peak tensile load was approximately 60 pct of the value
determined after 20 cycles. Note that the test termination did not always result in separated
specimens at the fracture. Prior to testing, the diameter of the gage section was measured in five
different locations using an optical comparator and the average value was used to determine
engineering stress in subsequent analyses.

3.4 Sample Preparation for Microstructural Analysis

Following LCF and creep-fatigue tests, the fractured specimens were carefully separated,
sectioned, and prepared for microstructural characterization. This section details the sample
preparation for characterization including light optical (LOM), scanning electron (SEM), and
transmission electron microscopy (TEM), X-ray diffraction (XRD), small angle neutron
scattering (SANS), and micro-hardness indentation.

3.4.1 Fracture Surfaces

The specimens tested to failure typically first need to be separated at the primary fracture,
since separation does not always happen at the end of the test. A typical specimen cross-section
after failure has a fractured region and an unfractured connected ligament, as illustrated in Figure
3.5. To separate the specimen without damaging the fracture surface or imposing significant
plastic deformation within the gage section, the connected ligament was carefully cut from the
surface towards the fractured region using a bandsaw. Once a sufficient notch was cut through
the majority of the connected ligament, the specimen was easily separated using a tensile testing
frame. The fracture surfaces were cut from the gage section, as close to the fracture as possible
using an MSX abrasive saw, making sure to preserve the fracture surface. The fracture surface
pieces were then cleaned in an ultra-sonic bath in acetone to remove coolant from the sawing
operation, followed by an ethanol rinse. Immediately prior to analysis, fracture surfaces were
cleaned using the same ultra-sonic method.

3.4.2 Mounted Samples for Hardness, LOM, SEM, and XRD Analysis

A single sample from each test specimen was used for hardness, LOM, SEM, and XRD
characterization. After cutting off the fracture surfaces, the remaining gage section halves were
Figure 3.5 An illustration of a typical fatigue specimen cross-section after test termination which contains a fractured region and an unfractured connected ligament.

cut from the specimen. Typically, each gage section half was approximately 10 mm long, depending on where the fracture was. The section closest to 10 mm long was cut along the centerline parallel to the cylinder axis using a slow-speed saw. One half of the cylinder section was mounted in Bakelite® so that the exposed sample was ~10 mm long by ~7 mm wide (i.e. the gage section diameter). The long edges of the mounted sample were the specimen surface and usually contained secondary micro-cracks, depending on the test condition. Mounted samples were ground up to 1200 grit and then polished with suspended diamond media up to 0.5 µm. Hardness testing was performed first on the mechanically polished and un-etched mounted samples. Details of hardness testing are given in Chapter 4. Following hardness testing, the samples were ground and polished again to remove the indents.

Dislocation density analysis using XRD and dislocation structure analysis using electron channeling contrast imaging (ECCI) and electron backscatter diffraction (EBSD) in the SEM are highly sensitive to surface deformation from mechanical polishing. The sample preparation for these techniques requires electropolishing after the mechanical polishing to remove surface deformation. Samples were electropolished in a solution of 10 pct perchloric acid in methanol at approximately -30 °C with a voltage of 30 V for ~60 to 90 sec. After electropolishing, samples were vibratory polished with suspended 0.02 µm colloidal silica for up to 16 h to remove surface topography introduced during electropolishing. Following the vibratory polish, samples were carefully washed with warm water and soap and then ultrasonically cleaned in ethanol to remove
colloidal silica particles. This sample preparation was also used for crack density analysis with LOM.

For grain size and precipitate analysis, samples were etched after mechanical polishing. For precipitate analysis, samples were chemically etched in a 3:1 molar ratio solution of HCl:HNO₃ (i.e. Aqua Regia) for 90 s, followed by an ethanol rinse. Aqua Regia preferentially attacks the austenite and leaves carbides and nitrides exposed. However, grain boundaries and twin boundaries are not easily distinguished after chemical etching with Aqua Regia. For grain size analysis, polished samples were electro-chemically etched using a solution of 60 pct HNO₃ in DI water with a current density of ~10 mA/cm² for up to 120 sec, according to a procedure from literature [3.2]. Electrochemical etching with this procedure does not reveal the twin boundaries to the same extent as grain boundaries, allowing for more precise grain size analysis.

3.4.3 TEM Sample Preparation

TEM analysis was performed on either traditional electropolished thin film disks or micro-milled (i.e. lift-out) samples using a focused ion beam (FIB). The half of the gage section that was not sectioned for mounting was used for TEM samples. Thin transverse sections (i.e. normal to the cylinder axis) were cut from the gage section using a slow speed saw and subsequently ground to a thickness of ~100 µm with a final finish of 800 grit. 3 mm disks were punched from the thinned sample for subsequent electro-polishing. The disks were electropolished using a Fishcione® twin-jet electropolisher with a solution of 10 pct perchloric acid in methanol at approximately -40 °C with a voltage of ~30 V. The voltage was adjusted during polishing to maintain a current of ~30 mA as the solution heated up.

FIB lift-out samples were extracted from the mounted samples, typically across a grain boundary. The procedure for lifting out a TEM sample are detailed elsewhere [3.3]. The final lift-out sample area is typically ~10 by ~15 µm. The long dimension of a lift-out sample crosses the grain boundary and is parallel to the loading direction (specimen axis), as shown in Figure 3.6.

3.4.4 SANS Sample Preparation

Sample preparation for SANS analysis is detailed in Chapter 7.
Figure 3.6 SEM image showing an example FIB lift-out area selection across a grain boundary that is parallel to the stress axis. The specimen was etched with Aqua Regia to reveal grain boundaries and precipitates.

3.5 X-Ray Diffraction (XRD)

XRD was used to characterize dislocation density from line broadening analysis. Two diffractometers, PANalytical® Empyrean and PANalytical® X'Pert, were used in this analysis with Cu Kα radiation, a step size of 0.008°, a time per step of 100 s, and a 2θ range from 40 to 100°. Each sample was scanned a total of four times, with 90° rotations between the scans. The 2θ range captures the first five peaks of FCC austenite, which are 111, 200, 220, 113, and 222. An example of the raw scan data is shown in Figure 3.7.

It is well known that peak broadening in an X-ray diffraction (XRD) line profile is due to three factors: instrument broadening, crystallite size, and micro-strain from defects like dislocations [3.4], [3.5]. Instrument broadening can be easily characterized as a function of angle and subtracted from the profile of interest. Characterization of the instrument broadening is done by scanning a standard specimen (polycrystalline Si in this study) with large grains and approximately zero strain over a large range of 2θ (45 to 100°) and determining full width at half-maximum intensity (FWHM) as a function of angle. A plot of instrument broadening (FWHM v. 2θ) is shown in Figure 3.8 for both the diffractometers used in this study. A second order polynomial fit was determined for each instrument, which results in the following instrument broadening functions

36
Figure 3.7 Typical XRD line profile of diffracted intensity versus scan angle, $2\theta$, from 40 to 100° capturing the first five peaks in austenite.

Figure 3.8 Instrument broadening, FWHM, as a function of scan angle, $2\theta$, for both diffractometers used in this study described by second order polynomial functions.
where $\beta_{\text{inst.}}$ is in degrees. Note that $\beta_{\text{inst.}}$ must be converted to radians for the following analysis.

Deconvolution of the remaining peak broadening into the other two components, $\beta_{\text{strain}}$ and $\beta_{\text{crystallite}}$, can be done using various approaches proposed by Warren and Averbach [3.6] and Williamson and Hall [3.7]. Ungar et al. [3.8] modified the Williamson-Hall method to account for dislocation strain anisotropy by introducing a dislocation contrast factor, $C$. Since broadening from small crystals, or diffracting domains, is not a function of angle and strain broadening is, a plot of broadening versus angle produces a positive slope that is directly related to the amount of micro-strain, and thus, dislocation density in the sample. The intercept is related to the crystallite size. Contrast factors are used to account for strain anisotropy caused by dislocations of different character (i.e. edge and screw). The contrast factors are specific to crystallographic planes and, therefore, the individual peaks analyzed. Contrast factors are calculated based on crystal structure, elastic constants, peak order, and dislocation character according to the procedure outlined by Ungar et al. [3.8]. In untextured polycrystalline cubic metals, the average contrast factor, $\overline{C}$, for each peak is

$$\overline{C} = \overline{C}_{h00}(1 - qH^2)$$  \hspace{1cm} (3.2)

where $\overline{C}_{h00}$ and $q$ are constants dependent on dislocation character and elastic constants of the crystal and $H^2$ is a function of crystallographic plane

$$H^2 = \frac{(h^2k^2 + h^2l^2 + k^2l^2)}{(h^2 + k^2 + l^2)^2}$$  \hspace{1cm} (3.3)

where $h$, $k$, and $l$ are the Miller indices of the plane. Hence, $\overline{C}$ is a function of dislocation character and elastic constants. For this study, the elastic constants were assumed to be the same as 304 stainless steel from literature; $C_{11} = 205$ GPa, $C_{12} = 138$ GPa, and $C_{44} = 126$ GPa [3.9]. $\overline{C}_{h00}$ and $q$ were then determined, as a function of elastic constants, from tables in literature [3.8] for both pure edge and pure screw dislocations. Finally, $\overline{C}$ was determined for each of the five peaks
scanned, for both pure edge and pure screw character. The value of $\bar{C}$ used in the following analysis is a weighted average of $\bar{C}_{\text{edge}}$ and $\bar{C}_{\text{screw}}$ depending on the fraction of each dislocation type. Dislocation density was plotted for two data sets as a function of percentage of screw dislocations, which is shown in Figure 3.9. From this figure, it can be seen that the variation in calculated dislocation density as a function of dislocation character is not significant, especially with respect to the measurement error. Hence, for all calculations, it was assumed that 50 pct of each character exists in deformed samples, which gives the average contrast factors, $\bar{C}_{\text{average}}$, shown in Table 3.4.

![Figure 3.9 Dislocation density as a function of dislocation character (pct screw dislocations). The variation in calculated density versus character is small with respect to the error of measurement.](image)

<table>
<thead>
<tr>
<th>Peak, {h,k,l}</th>
<th>$\bar{C}_{\text{edge}}$</th>
<th>$\bar{C}_{\text{screw}}$</th>
<th>$\bar{C}_{\text{average}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>{111}</td>
<td>0.133</td>
<td>0.059</td>
<td>0.096</td>
</tr>
<tr>
<td>{002}</td>
<td>0.308</td>
<td>0.326</td>
<td>0.317</td>
</tr>
<tr>
<td>{022}</td>
<td>0.176</td>
<td>0.125</td>
<td>0.151</td>
</tr>
<tr>
<td>{113}</td>
<td>0.225</td>
<td>0.200</td>
<td>0.213</td>
</tr>
<tr>
<td>{222}</td>
<td>0.133</td>
<td>0.059</td>
<td>0.096</td>
</tr>
</tbody>
</table>

In the modified Williamson-Hall method for deconvolution of the sample broadening into the strain and crystallite size components, the measured profile broadening (less the instrument
contribution) is related to the crystallite size, $D$, and dislocation density, $\rho$, by the following equation:

$$\frac{\cos \theta_B \cdot (\beta_{\text{strain}} + \beta_{\text{crystallite}})}{\lambda} = \frac{0.9}{D} + \left( \frac{\pi M^2 b^2}{2} \right)^{1/2} \cdot \rho^{1/2} \cdot \frac{2 \sin \theta_B}{\lambda} \bar{c}^{1/2}$$ \hspace{1cm} (3.4)

where $\lambda$ is the radiation wavelength ($\lambda_{Cu} = 0.154$ nm), $\theta_B$ is the Bragg angle of the peak, $b$ is the magnitude of the Burgers vector ($b_{\frac{a}{2}<110>} = 0.254$ nm), $C$ is the contrast factor for the specific peak, and $M$ is a dimensionless constant related to the outer cutoff radius and arrangement of dislocations. For an analysis on a cyclically deformed stainless steel, $M$ is assumed to be 2 for relatively randomly distributed dislocations [3.10]. Equation 3.4 can be simplified as

$$\Delta K = B + mKC\bar{c}^{1/2}$$ \hspace{1cm} (3.5)

The modified Williamson-Hall plot is then made by plotting $\Delta K$ v. $KC\bar{c}^{1/2}$ where the slope, $m$, is related to the dislocation density and the intercept, $B$, is related to the crystallite size according to the first two terms on the right side of Equation 3.4. An example modified Williamson-Hall plot with data from failed creep-fatigue tests at 550 and 650 °C is shown in Figure 3.10. The slope of the linear fit of the data at 550 °C is greater compared to 650 °C, indicating a higher dislocation density in the sample tested at 550 °C. Note that the intercept is negative in this plot, which indicates a negative crystallite size. This is typical for the data analyzed in this study. The grain sizes in the material tested are on the order of 30 to 60 µm, which is significantly larger than the size (∼1 µm) where broadening occurs from small crystals. In the test conditions where small subgrains form, the negative intercept indicates that the volume of the recovered microstructure is small compared to the volume that is not recovered.

Therefore, the intercept should be approximately zero. A negative intercept may be due to error in determining the instrument broadening. The mass absorption coefficient (MAC) of Si is ∼6.4 compared to Fe which is ∼36, which may lead to error in the characterization of instrument broadening using a Si standard rather than a LaB$_6$ standard which has a MAC of ∼32. Using a standard with the same MAC as the material tested most likely shifts the curves in Figure 3.10 upwards rather than change the slope.
Figure 3.10 Example modified Williamson-Hall plot of $\Delta K$ v. $KC^{1/2}$ for failed creep-fatigue samples tested at 550 and 650 °C. The greater slope in the 550 °C sample corresponds to a higher dislocation density.

### 3.6 Internal Damage

Creep-fatigue testing with 30 min holds results in internal damage in all conditions studied here. The grain boundary damage consists of either round (or elliptical) voids or cracks along boundaries and triple points. In general, the cracks are normal to the loading axis and do not have any components that are parallel to the loading direction. Grain boundary cracks were quantified using a line intercept method with light optical micrographs. The stereological method involves overlaying a grid of vertical lines over a micrograph containing horizontal cracks and counting intersections of grid lines and cracks. ImageJ® was used to overlay a random grid of vertical lines on the micrographs. The effective crack length per unit area, $L_{\text{eff}}$, is then given by the following equation:

$$L_{\text{eff}} = \frac{NA}{l_{\text{grid}}}$$  \hspace{1cm} (3.6)

where $N$ is the number of intersections, $A$ is the image area (mm$^2$), and $l_{\text{grid}}$ is the total grid length (mm) [3.11]. For each sample measured, ten micrographs were taken 1 mm from the surface and ten were taken 2 mm from the surface along the gage sections of each sample. Micrographs were carefully selected to include only internal cracks and no cracks that extended to the surface. For
the effective crack length analysis, only intersections of cracks were counted; \textit{i.e.} voids were not considered. An example of a light optical dark field micrograph from a failed creep-fatigue specimen tested at 550 °C is shown in Figure 3.11 with a random vertical grid overlay.

![Micrograph Image](image)

Figure 3.11 Example dark field light optical micrograph of grain boundary cracks after creep-fatigue failure at 550 °C with an overlaid vertical grid for intersection counting. The right side of the micrograph is taken 1 mm from the specimen surface.

Voids were quantified more simply by an area density (\textit{i.e.} number of voids per unit area) using higher magnification scanning electron micrographs. Scanning electron micrographs were used for void analysis so that spherical precipitates were not confused for voids. ImageJ® was used to count the voids. Ten random micrographs at a magnification of 500X were counted for each sample.

### 3.7 Grain Size

Grain size analysis was carried out on four laboratory produced heats (011439, 011502, 011593, and 011594) according to the circular intercept method outlined in ASTM E112 [3.12]. This standard applies to microstructures of normal grain size distribution only. However, microstructures in heats 011502 and 011593 were found to be non-uniform, with bands of very large (up to 600 µm) non-recrystallized grains in-between bands of relatively uniform equiaxed grains. Thus, for each heat of material, five light optical micrographs were selected from random areas where the grain size was relatively uniform; \textit{i.e.} banded non-recrystallized regions were
avoided for this analysis. All images were taken at 100X magnification. Electrolytic etching of samples with 60 pct nitric acid in water resulted in better accuracy than chemical etching since annealing twins were not etched to the same degree as grain boundaries, allowing for reasonable distinction between the two boundary types. Using ImageJ®, three concentric circles were randomly overlaid on each micrograph with perimeters of 447.4, 894.8, and 1342.1 µm, as shown in the example micrograph in Figure 3.12. The ASTM grain size can then be determined by the total line length and number of intersections with grain boundaries. Finally, ASTM grain size is converted to diameter in µm, assuming the grains are tetrakaidecahedron shape [3.12].

Figure 3.12  Example light optical micrograph of an etched sample with three overlaid concentric circles, randomly centered, for grain size analysis.

3.8  Fatigue Data Analysis

Raw fatigue data is recorded and saved in two files: one which contains peak tensile and compressive loads for every cycle in a test and one which contains load, displacement, and time data at regular time intervals for selected cycles. In the former file, peak loads are converted to engineering stress, given the specimen geometry. From this file, peak tensile and compressive stress versus cycle number can be plotted for each test. Additionally, the stress ratio (i.e. peak tensile stress divided by peak compressive stress) can be obtained from the first file, which is used to determine the numbers of cycles to failure.

The second file contains the raw data that makes up the hysteresis loops; i.e. engineering stress and strain at a given frequency for a complete loading cycle, including the hold time, if applicable. Each complete hysteresis loop is recorded for the first 100 cycles, after which
complete loops are recorded only every 50 cycles. Cyclic deformation behavior is analyzed by partitioning the hysteresis loops into the different components of stress and strain during the loading and unloading portions of the loops. Time-dependent deformation behavior is analyzed through the stress relaxation and associated strain rates, which occur during the hold period.

### 3.8.1 Stress and Strain Partition Analysis

Schematic hysteresis loops for fully reversed LCF and creep-fatigue cycles are shown in Figure 3.13. For all tests in this study, the total strain amplitude, $\varepsilon_{aT}$, is fixed (i.e. strain-controlled). The amplitude of plastic strain, $\varepsilon_{ap}$, in LCF is simply the width of the hysteresis loop at zero stress and varies from cycle to cycle, depending on the deformation behavior (i.e. hardening or softening). The stress amplitude, $\sigma_a$, also varies for each cycle depending on the cyclic hardening ($\sigma_a$ increasing) or softening behavior ($\sigma_a$ decreasing).

In contrast to LCF, $\varepsilon_{ap}$ is composed of two components for each creep-fatigue cycle: the plastic deformation during cycling (i.e. loading and unloading), $\varepsilon_{ap,cyc}$, and the time-dependent (i.e. creep) deformation that occurs during the constant tensile strain hold period, $\varepsilon_{cr}$. Thus, the total plastic strain amplitude is

$$\varepsilon_{ap} = \varepsilon_{ap,cyc} + \varepsilon_{cr} = \varepsilon_{aT} - \varepsilon_{ae}$$  \hspace{1cm}  (3.7)

where $\varepsilon_{ae}$ is the elastic strain amplitude. As with LCF, $\varepsilon_{ap}$ is measured by the width of a given hysteresis loop at zero stress (i.e. the width of the loop on the strain axis). However, it is important to distinguish between the different components of $\varepsilon_{ap}$. Stress relaxes during the tensile hold as a result of the conversion of elastic strain into inelastic strain, which typically occurs by creep mechanisms (i.e. dislocation climb or vacancy flux). Therefore, the inelastic creep strain during the hold, which is realized at the expense of elastic strain, can be determined from Hooke’s law according to

$$\varepsilon_{cr} = \varepsilon_{rlx,e} = \frac{\Delta \sigma_{rlx}}{E}$$  \hspace{1cm}  (3.8)
where $E$ is the elastic modulus at the test temperature, $\varepsilon_{rlx,e}$ is the elastic strain converted to inelastic strain, and $\Delta\sigma_{rlx}$ is the magnitude of stress relaxation. $\Delta\sigma_{rlx}$ is measured simply by the difference between the tensile stress at the beginning and end of the hold for each cycle, as shown in Figure 3.13. Therefore, once $\varepsilon_{ap}$ is determined by the hysteresis loop width at zero stress and $\varepsilon_{cr}$ is determined from $\Delta\sigma_{rlx}$, $\varepsilon_{ap,cyc}$ can then be calculated from Equation 3.7. A MATLAB® script, shown in Appendix D, has been developed for partitioning hysteresis loops into the plastic strain components, as described above.

![Figure 3.13 Schematic fully reversed hysteresis loops for (a) LCF and (b) creep-fatigue with a hold at peak tensile strain showing the different strain components in a given cycle.](image)

The evolution of cyclic flow stress was investigated for all test conditions by partitioning the total stress amplitude, $\sigma_a$, into the friction and back stress components, $\sigma_f$ and $\sigma_b$, respectively. The method for stress partitioning of a fully reversed hysteresis loop used in this investigation, originally proposed by Cottrell [3.13] and extended by Kuhlmann-Wilsdorf and Laird [3.14], is a widely recognized approach for understanding dislocation behavior in fatigued metals. Friction stress (or effective stress) is considered to be an isotropic and thermal component of flow stress that represents the stress to move a dislocation through the matrix in the presence of obstacles, such as solute atoms, precipitates, and other dislocations. Based on the stress partitioning method, the friction stress is essentially the cyclic yield stress. Back stress (or internal stress) is the athermal component of flow stress associated with long range dislocation...
pile-ups. Back stress is associated with the increase in shear stress from strain-hardening. Thus, partitioning the total flow stress into the two components can provide an understanding of the mechanisms responsible for the evolution of stress and strain during cyclic deformation.

The basic principle of stress partitioning will be described, along with the schematic hysteresis loop shown in Figure 3.14. First, friction stress can be considered isotropic and independent of strain direction, while the back stress changes sign upon strain reversal. Back stress is elastic in nature and always reaches a maximum at the peak strain value. Upon reversal of the loading, the back stress acts in the direction of strain reversal to aid the reverse deformation (i.e. to lower the yield stress), which leads to the well-known Bauschinger effect. At the start of plastic deformation in each half cycle, the back stress generated in the previous half cycle acts in the same direction of the applied stress. Thus, in the presence of a friction stress on mobile dislocations, the following relation is assumed:

\[
\sigma_{ys} = \sigma_f - \sigma_b \tag{3.9}
\]

where \(\sigma_{ys}\) is the yield stress in the direction of loading. At the maximum stress, \(\sigma_{\text{max}}\), the back stress and flow stress act in the same direction such that:

\[
\sigma_{\text{max}} = \sigma_b + \sigma_f \tag{3.10}
\]

Combining Equations 3.9 and 3.10 yields the following:

\[
\sigma_f = \frac{(\sigma_{\text{max}} + \sigma_{ys})}{2} \tag{3.11}
\]

\[
\sigma_b = \frac{(\sigma_{\text{max}} - \sigma_{ys})}{2} \tag{3.12}
\]

It should be noted that an underlying assumption in this stress partition analysis is that the hysteresis loops are “reversible”, which means that the dislocation behavior, and the internal stress and strain distribution, remain relatively constant in successive cycles (at the same strain values) and from forward to reverse half cycles (disregarding the small difference in stress magnitude between tension and compression) [3.14]. This assumption may not be valid in the
context of elevated temperature creep-fatigue hysteresis loops, where the tensile hold time likely influences the dislocation behavior and distribution of stress and strain. An assumption in the formulation of Equation 3.9 is that $\sigma_b$ at yielding (i.e. $\sigma_{ys}$) is approximately equal to $\sigma_b$ at the strain reversal (i.e. at peak strain), which may not be the case at elevated temperatures or slow strain rates where stress relaxation occurs during the elastic loading to reduce $\sigma_b$. Other methods for stress partitioning have been proposed to compensate for overestimation of $\sigma_f$ values where a large viscous stress component ($\sigma_v$) exists and/or where $\sigma_b$ changes on loading due to stress relaxation [3.15]–[3.17]. For example, the method proposed by Handfield and Dickson requires an initial partition of the linear elastic region into the true elastic component and a “pseudo-elastic” component. For the hysteresis loops studied here, a pseudo-elastic region could not be identified accurately and repeatably for all conditions studied. Therefore, for comparison purposes among all conditions, the simple Cottrell method was used to partition the hysteresis loops and the implications of the assumptions in the analysis are discussed where appropriate. Only the half of the loop following peak compression strain (i.e. tensile loading) was analyzed for each cycle to minimize the effects from stress relaxation that occurs during peak tensile strain.

Figure 3.14 Schematic LCF hysteresis loop illustrating the partitioning of maximum stress, $\sigma_{max}$, into friction and back stress components, $\sigma_f$ and $\sigma_b$, respectively.
Partitioning of the flow stress requires an accurate and repeatable method for extraction of $\sigma_{ys}$. For this study, the tensile loading portion of each hysteresis loop was extracted and fit to a Ramberg-Osgood relation

$$\varepsilon_a = \frac{\sigma_a}{E_{eff}} + \left(\frac{\sigma_a}{K'}\right)^{1/n'}$$

where $\varepsilon_a$ is the strain amplitude and $K'$ and $n'$ are constants related to the cyclic hardening behavior. The left term on the right side of the equation represents the elastic strain and the right term is the plastic strain. $E_{eff}$ is the effective elastic modulus measured from reversal to 0.15 pct strain after reversal, which was determined for each cycle since it is commonly different than the monotonic elastic modulus and it evolves with cycle number [3.16]–[3.18]. Using the hardening constants, $K'$ and $n'$, the yield stress was determined at a plastic strain amplitude offset, $\varepsilon_{ap} = 0.001$, according to the following relation

$$\sigma_{ys} = K' (\varepsilon_{ap})^{n'}$$

Finally, $\sigma_f$ and $\sigma_h$ are determined according to Equations 3.11 and 3.12.

A MATLAB® script, detailed in Appendix D, has been developed for partitioning hysteresis loops into the different components of stress, as described above. In order to fit the tensile half of the hysteresis loop to the relationship shown in Equation 3.13, the data from peak compressive strain to peak tensile strain is extracted and all data is translated so that the stress and strain at reversal is zero. In other words, all stress and strain data in the half loop are shifted to positive (tensile) values, similar to standard tensile test data. Finally, a non-linear regression solver is used to fit the tensile data, as shown in Figure 3.15. The fitting constants are saved for each cycle and extracted for subsequent analyses.

### 3.8.2 Strain-Hardening Rate Analysis

Strain-hardening behavior in materials is indicative of the deformation behavior and the strengthening mechanisms. Hence, an analysis of the strain-hardening behavior during cycling in LCF or creep-fatigue tests can provide an understanding of the cyclic deformation mechanisms.
The strain-hardening rates are evaluated for the tensile half of the hysteresis loops in this study. Once the tensile half of the loop is fit to the relation given in Equation 3.13, the fitting constants \( K' \) and \( n' \) are obtained. Since strain-hardening is a result of plastic deformation, only the right term in Equation 3.13 is used to determine the rate. The strain-hardening rate is defined as

\[
\frac{d\sigma}{d\varepsilon} = n'K'(\varepsilon_{ap})^{n'-1}
\]

where the plastic strain amplitude, \( \varepsilon_{ap} \), starts at the strain amplitude offset which defines yielding (i.e. 0.001 offset). A MATLAB® script, detailed in Appendix D, has been developed to solve for the strain-hardening rate, based on the Ramberg-Osgood relation, in the tensile half of any hysteresis loop from the defined onset of plasticity.

![Figure 3.15 Example complete hysteresis loop in (a) and the Ramberg-Osgood fit relation (black line) to the tensile portion of the loop (blue markers) in (b).](image)

### 3.8.3 Stress Relaxation Analysis (Creep Stress Exponent)

The time-dependent deformation of the material is assessed through analysis of the stress relaxation behavior during the constant strain tensile hold of each cycle in a creep-fatigue test. Analysis of creep deformation mechanisms includes an analytical approach from stress relaxation data. During a tensile hold period in a creep-fatigue test, the strain is held constant and elastic strain is converted to inelastic strain, which is manifested as a relaxation of the stress. Since strain is constant, strain-rate must be zero, allowing for one to equate elastic strain rate from Hooke’s law, \( \dot{\varepsilon}_e \), and the plastic creep strain rate, \( \dot{\varepsilon}_{cr} \), according to
where \( \sigma \) is instantaneous stress, \( E \) is Young’s modulus, \( B \) is a constant with temperature that contains activation energy for vacancy formation and diffusion, and \( n \) is the creep-rate exponent [3.19]. Equation 3.16 can be rearranged to give instantaneous stress as a function of time according to

\[
\dot{\varepsilon} = \dot{\varepsilon}_e + \dot{\varepsilon}_{cr} = 0 = \frac{d\sigma}{dt} \frac{1}{E} + B\sigma^n
\]

3.16

where \( \sigma \) is instantaneous stress, \( E \) is Young’s modulus, \( B \) is a constant with temperature that contains activation energy for vacancy formation and diffusion, and \( n \) is the creep-rate exponent [3.19]. Equation 3.16 can be rearranged to give instantaneous stress as a function of time according to

\[
\sigma = \left[ tBE(1 - n) + \sigma_0^{1-n} \right]^\frac{1}{1-n}
\]

3.17

where \( \sigma_0 \) is the initial stress at the start of relaxation. Equation 3.17 can be further simplified by assuming \( B, E, \) and \( n \) are constants for the relaxation period, taking the form

\[
\sigma = a[t + b]^c
\]

3.18

where \( a, b, \) and \( c \) are constants [3.20]. Note that if the deformation mechanism changes during relaxation, \( B \) and \( n \) are not necessarily constant and the previous assumption may be erroneous; thus, this analysis assumes constant mechanisms and microstructure during the hold period. Raw stress relaxation data (i.e. stress and time) are then fit with a non-linear least-squares approach to the form of Equation 3.18. A MATLAB® script, detailed in Appendix D, has been developed to perform the non-linear fitting of the stress relaxation data for every cycle where stress relaxation data is recorded. An example of raw data and the function specific curve fit is shown in Figure 3.16.

The creep rate exponent can be estimated from the fitting constant \( c \). It has been shown that the value of the \( n \) is indicative of the creep deformation mechanism [3.21]. A creep rate exponent of 1 is indicative of a diffusion-based deformation mechanism, whereas an exponent of 5 is typical for climb-then-glide deformation. This type of analysis becomes more complicated when precipitates are present, resulting in exponent values above 5 for climb-then-glide deformation, due to the interaction of the climbing dislocation and the matrix/precipitate interface [3.22].
Figure 3.16  Example stress relaxation data and function specific curve fit for a 650 °C creep-fatigue mid-life cycle (N=500). The creep rate exponent, $n$, in this case is 13.8.

### 3.8.4 Stress Relaxation Analysis (Creep Strain Rate)

The time-dependent strain rate is indicative of the mechanisms responsible for the inelastic deformation. For example, strain rates during the hold time that are greater than approximately $10^{-4}$ s$^{-1}$ are associated with matrix plasticity (*i.e.* dislocation glide) [3.23], [3.24]. Therefore, the highest strain rates, which occur at the beginning of the hold time in a creep-fatigue test, may not be associated with creep deformation or damage. However, creep cavitation develops when the strain rates are less than $10^{-4}$ s$^{-1}$, where deformation occurs by diffusional mechanisms (*i.e.* dislocation climb or vacancy/mass flux). Hence, it is important to determine the strain rates during relaxation to ensure that creep damage is estimated accurately.

According to Equation 3.16, the inelastic strain rate is equal to the elastic strain rate during a constant strain hold time. Therefore, the rate of inelastic strain can be calculated from the elastic strain rate according to Hooke’s law (the left term in the right side of Equation 3.16). Differentiating the relation in Equation 3.18 gives

$$\dot{\varepsilon}_{cr} = \dot{\varepsilon}_e = \frac{d\sigma}{dt} \frac{1}{E} = ac[t + b]^{c-1}$$  \hspace{1cm} 3.19

Thus, the rate of time-dependent inelastic deformation can be determined for each cycle where stress relaxation data is recorded.
3.9 References


CHAPTER 4
ASSESSMENT OF CREEP-FATIGUE BEHAVIOR, DEFORMATION MECHANISMS, AND
MICROSTRUCTURAL EVOLUTION OF ALLOY 709 UNDER ACCELERATED
CONDITIONS

A paper published in *The International Journal of Fatigue*, February 2019\(^1\).
Ty Porter\(^2,3,4\), Kip Findley\(^5\), Michael Kaufman\(^5\), and Richard Wright\(^6\)

4.1 Abstract

Alloy 709 is a leading candidate structural material for sodium-cooled fast spectrum reactors (FSR), where creep-fatigue is a potential failure mode. Low cycle fatigue (LCF) and creep-fatigue tests with 30 min tensile hold times have been conducted to failure at 550 and 650 °C, corresponding to potential service and accelerated test temperatures, respectively. Creep-fatigue life is reduced relative to LCF at both temperatures, though significantly more at 550 °C compared to 650 °C. Differences in slip behavior, dynamic recovery, precipitate evolution, and strain-aging effects result in less stress relaxation and sustained high tensile stresses in creep-fatigue at 550 °C; thus, there is more intergranular damage compared to 650 °C.

4.2 Introduction

The improved economics of next generation nuclear reactors depend, in part, on improved performance of advanced structural materials. Power plant structural components, for both fossil and nuclear fuels, are subject to complex stress cycles at elevated temperature, which necessitates the use of highly corrosion- and creep-resistant alloys. An increase in operating

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temperature and component lifetime demands advancements of high-temperature structural alloys. Alloy 709 is a 20Cr-25Ni-1.5Mo-Nb-N solid solution and precipitation strengthened austenitic stainless steel developed by Nippon Steel for fossil boiler applications, implemented under ASME Code Case 2581 [4.1]. Alloy 709 is a candidate alloy for structural components in sodium cooled fast spectrum reactors (FSR) due to its improved creep resistance over the current structural alloys Type 316 stainless steel and Grade 91 [4.2]. Currently, Alloy 709 is only ASME code qualified in tubular form for fossil boiler applications, whereas nuclear structural components will be made from plate product form. ASME Boiler and Pressure Vessel (BPV) Code requires analysis of creep-fatigue performance for thick section nuclear components due to the thermomechanical fatigue experienced during start-up and shut-down of the reactor [4.3]. Creep-fatigue analysis is not required under the BPV code for fossil boiler applications, so little creep-fatigue data has been generated for performance assessment for this alloy.

In addition to the difference in product form, the proposed service conditions differ significantly between fossil and nuclear applications. The expected service temperature for structural components of next generation FSRs is 550 °C, whereas boiler tubes typically experience service temperatures greater than 650 °C. Additionally, fossil boiler tubes have shorter life cycles than those required for nuclear structural components. Therefore, most of the published creep and microstructural data for Alloy 709 is at higher temperatures than typical FSR operating temperatures.

Accelerated creep and creep-fatigue testing, by means of testing at higher temperatures and greater strains than expected during service, is typically necessary to generate a statistically significant amount of data in a reasonable time. The creep-rupture data from accelerated creep tests are extrapolated to expected service conditions using a Larson-Miller plot to predict component life for creep conditions and as part of the creep-fatigue life analysis. However, it has been shown that extrapolation of accelerated test results to lower temperatures and smaller strains produces non-conservative creep rupture and creep-fatigue life predictions for Type 316L(N) stainless steel [4, 5]. The non-conservative predictions are attributed to a change in time-dependent (creep) deformation mechanisms from power-law creep (climb then glide creep) in accelerated conditions to diffusional creep (Nabarro-Herring and Coble creep) in near-service conditions. Additionally, dynamic strain aging (DSA) is active in several nitrogen-containing stainless steels in the intermediate temperature range relevant to service conditions [5 - 10]. DSA
is known to affect the deformation and damage mechanisms of stainless steels in low cycle fatigue (LCF) by promoting planar slip and preventing dynamic recovery [6, 7, 11, 12]. The enhanced slip planarity, which contributes to high rates of strain hardening and slip band impingement on grain boundaries, can be detrimental to creep-fatigue life [8, 9, 13].

An initial investigation of creep-fatigue in Alloy 709 by the present authors showed significantly different behavior and cyclic life reduction compared to LCF at 550 and 650 °C [4.14]. The objective of the current investigation is to assess the deformation and damage mechanisms and microstructural evolution during creep-fatigue testing of Alloy 709 at an accelerated testing condition (650 °C) and the relationship to the near-service condition (550 °C). Differences in deformation behavior between accelerated testing and service conditions can result in significantly different creep-fatigue cyclic stress response, damage formation, and cyclic life. Additionally, the stability of the microstructure (i.e. second phase formation and dissolution) in austenitic stainless steels is known to have a significant effect on elevated temperature mechanical behavior. Evolution of the microstructure of Alloy 709 is also investigated at 550 and 650 °C to determine the effects on creep-fatigue performance. A comprehensive understanding of creep-fatigue behavior and microstructural evolution at accelerated testing and near-service conditions will provide a basis to develop reliable extrapolation models for Alloy 709 in nuclear reactor design.

4.3 Experimental Procedure

Strain-controlled LCF tests, without and with (creep-fatigue) 30 min tensile hold periods, have been performed according to ASTM E2714 [4.15], using servo-hydraulic testing frames at Idaho National Laboratory (INL) on specimens machined from two solution annealed heats of Alloy 709, labeled 011593 and 011594. Tests were conducted to failure at the expected service temperature of 550 °C and an accelerated test temperature of 650 °C. Additionally, creep-fatigue tests were interrupted at various cycle intervals corresponding to regions of cyclic hardening, a maximum in cyclic tensile stress, and cyclic softening. All test conditions are summarized in Table 4.1. Fatigue tests to failure were performed on both experimental heats of material and all of the interrupted tests were performed on heat 011594. All tests were conducted in air using a symmetrical triangular waveform profile with R = −1, a strain rate (\(\dot{\varepsilon}\)) of 10^{-3} s^{-1}, and a total strain range (\(\Delta\varepsilon_T\)) of 1 pct. Strain was measured using a two-pronged extensometer. During
testing, temperature was maintained by resistance heaters and monitored by thermocouples welded outside the gage section. Cylindrical button-head specimens were machined from the as-received annealed plates with the stress axis parallel to the rolling direction and longitudinal final polishing in the gage section to eliminate fatigue crack initiation sites. The number of cycles to failure is defined by a 20 pct decrease in the ratio of peak tensile to peak compressive stress, which indicates macroscopic crack initiation [4.16].

Two microstructurally different heats of Alloy 709 were used in this study, with compositions shown in Table 4.2. Both heats were processed at Oak Ridge National Laboratory (ORNL) by hot forging and rolling, then solution annealing at 1100 °C, followed by water quenching. The grain size and uniformity of the as-received microstructures of the two heats were compared by light optical microscopy (LOM). Specimens for LOM were mechanically polished and then electrolytically etched in a solution of 60 pct nitric acid in water with a current density of approximately 10 mA/cm² to delineate the grain boundaries without revealing the annealing twins to the same level of contrast according to a procedure outlined in literature [4.17]. Grain size was determined using the concentric circle intercept method on five random areas according to ASTM E112 [4.18].

The effective length of intergranular cracks was quantified for each condition with a linear intercept method using light optical micrographs of polished specimens. Ten micrographs were taken 1 mm from the surface and ten were taken 2 mm from the surface along the gage

<table>
<thead>
<tr>
<th>Table 4.1 LCF and Creep-Fatigue Test Matrix</th>
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<tbody>
<tr>
<td>Temperature (°C)</td>
</tr>
<tr>
<td>------------------</td>
</tr>
<tr>
<td>650</td>
</tr>
<tr>
<td>650</td>
</tr>
<tr>
<td>550</td>
</tr>
<tr>
<td>550</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 4.2 Compositions of Experimental Heats of Alloy 709 (wt pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat ID</td>
</tr>
<tr>
<td>---------</td>
</tr>
<tr>
<td>011593</td>
</tr>
<tr>
<td>011594</td>
</tr>
</tbody>
</table>
sections of each sample. A random grid of vertical lines was overlaid on images where the cracks were primarily horizontal (normal to the loading axis). The number of crack intersections, $N$, was tabulated and the effective crack length, $L_{eff}$, was determined by

$$L_{eff} = \frac{NA}{l_{grid}} \quad \text{(4.1)}$$

where $A$ is the micrograph area in mm$^2$ and $l_{grid}$ is the total length of gridlines [4.19].

Electron channeling contrast imaging (ECCI) was used to characterize deformation in the resulting microstructures. This technique shows contrast variations as a result of misorientation within the lattice due to boundaries, slip bands, or defects (i.e. dislocations and stacking faults) [12, 20, 21]. ECCI was performed on bulk samples from the gage sections of failed specimens using a JEOL® field emission scanning electron microscope (FESEM) in COMPO mode (signal of two backscatter electron detectors summed) with a scintillator type backscatter electron detector. The bulk nature of this technique allows for a much larger area to be analyzed than in a transmission electron microscope (TEM). Due to the sensitivity of ECC specimens to subsurface deformation from mechanical polishing, specimens were diamond polished to 0.5 µm, followed by electropolishing with a solution of 10 pct perchloric acid in methanol, and finally vibratory polishing with 0.02 µm colloidal silica for up to 8 h.

TEM was used to analyze dislocation structures and precipitation in failed specimens. A Philips® CM12 and a FEI® Talos F200X TEM were used in this analysis, with accelerating voltages of 120 and 200 keV, respectively. TEM specimens were prepared from transverse slices through the gage section away from the fracture surface. 3 mm discs were electropolished on a Fishcione twin-jet electropolisher using a solution of 10 pct perchloric acid in methanol at a temperature of approximately –40 °C.

Dislocation densities of the deformed microstructures were estimated from XRD line broadening. XRD scans were conducted on the same specimens used for ECCI deformation characterization. A PANalytical® XPert diffractometer was used with Cu K$_a$ radiation, a step size of 0.008°, a time per step of 100 s, and a 2θ range from 40 to 100°. A modified Williamson-Hall method was used to determine dislocation density, according to the procedure described by Ungar *et al.* [4.22]. The first five peaks of FCC austenite (111, 200, 220, 113, and 222) were
used in this analysis, and each sample was scanned a total of four times to increase the statistical significance, with sample rotations between each scan.

Vickers microhardness testing was performed on the as-received material and the gage sections of each test specimen conducted to failure on heat 011594 according to ASTM E384 [4.23]. A grid of 50 indents and a 500 gf load was used on each sample.

4.4 Results of Initial Microstructural Characterization

Light optical micrographs of the as-received (solution annealed) microstructures of the two heats of Alloy 709 used for this study are shown in Figure 4.1 [4.14]. Heat 011594, shown in Figure 4.1 (a), has a uniform equiaxed grain structure with an average grain size of approximately 30 µm and an average hardness of 176 HV. Heat 011593, shown in Figure 4.1 (b), has a banded microstructure of non-uniform grain size and shape. This microstructure has bands of relatively large grains elongated in the rolling direction between larger regions with finer grain size and a narrower size distribution. In the uniform regions, the average grain size is approximately 20 µm, while in the bands of large grains the grain size is up to approximately 600 µm. The average hardness of 011593 is 193 HV. Both microstructures contain a significant amount of annealing twins.

![Figure 4.1](image)

Figure 4.1 Light optical micrographs of the as-received microstructures from heats (a) 011594 and (b) 011593 [4.14].

The solution annealed microstructures contain a bi-modal size distribution of Nb(C,N) precipitates observed both intragranularly and intergranularly. Fine spherical precipitates with a
diameter of approximately 100 nm are distributed relatively uniformly throughout the microstructure, while irregularly shaped precipitates ranging in size from approximately 500 nm to 10 µm are present in stringers aligned along the rolling direction. The fine precipitates formed during hot working and subsequent cooling, whereas the coarse precipitates likely formed during casting solidification. Both heats used in this study had similar initial precipitation volume fractions and distributions.

4.5 Results of LCF and Creep-Fatigue

Peak tensile and compressive stress versus cycle number are shown in Figure 4.2 for LCF and creep-fatigue tests at 550 and 650 °C. During LCF at 550 °C, the material cyclically hardens for the entire test, with a higher rate of hardening for approximately the first 200 cycles followed by a slower rate of hardening until the stress drops off as a result of crack formation and propagation. At 650 °C, initial cyclic hardening occurs at a higher rate compared to 550 °C up to a maximum stress after approximately 100 cycles, followed by cyclic softening until failure. With the addition of a 30 min tensile hold (i.e. creep-fatigue), cyclic hardening at 550 °C is extended to approximately 400 cycles to a maximum stress approximately 14 pct greater than in LCF. In creep-fatigue, unlike LCF, cyclic softening occurs after approximately 400 cycles at 550 °C. In creep-fatigue at 650 °C, the cyclic hardening regime is shortened to approximately the first 50 cycles to a maximum stress approximately 5 pct lower than in LCF. Subsequent cyclic softening at 650 °C occurs at a high rate initially, followed by a slower rate until failure.

For both LCF and creep-fatigue tests, the cyclic stress behavior (i.e. hardening and softening) was consistent between the two microstructures, though there was a slight variation in cycles to failure. These results suggest that the homogeneity of the grain structure does not have a significant impact on LCF or creep-fatigue performance under the specific conditions used in this study (i.e. smooth polished specimens machined with the stress axis parallel to the plate rolling direction).

The number of cycles to failure, \(N_f\), for each test is presented in Table 4.3. In LCF, the tests at 550 °C had cyclic lives approximately twice those at 650 °C. In creep-fatigue, however, the cyclic lives were approximately the same at both temperatures. Compared to LCF, the cyclic lives were reduced significantly at both temperatures with the introduction of a 30 min tensile dwell period, which is typical for austenitic stainless steels. The fatigue life reduction factor
(FLRF), defined as $N_f$ in creep-fatigue normalized by $N_f$ in LCF, is a convenient metric to evaluate creep-fatigue lives compared to LCF for a given temperature, strain rate, and strain range [4.5]. At 650 °C, the average FLRF is 0.51, i.e. a life reduction of approximately two when the hold time is imposed. The average FLRF at 550 °C is 0.24, i.e. a creep-fatigue life reduction of approximately four. The significant difference in the creep-fatigue life reduction between the two temperatures indicates that the 30 min tensile hold time is more damaging at 550 °C than 650 °C for Alloy 709.

![Figure 4.2 Peak tensile and compressive stress at each fatigue cycle for (a) LCF and (b) creep-fatigue tests conducted to failure at 550 and 650 °C with $\Delta\varepsilon_T = 1$ pct. Each of the four tests were performed on both heats of material.](image)

<table>
<thead>
<tr>
<th>Test Temperature (°C)</th>
<th>Hold Time (min)</th>
<th>Cycles to Failure, $N_f$ (011593, 011594)</th>
<th>Average FLRF</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>0</td>
<td>4964, 3629</td>
<td>0.24</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>888, 1062</td>
<td></td>
</tr>
<tr>
<td>650</td>
<td>0</td>
<td>1883, 2047</td>
<td>0.51</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>1069, 931</td>
<td></td>
</tr>
</tbody>
</table>

Creep-fatigue hysteresis loops in the hardening regime for both temperatures are shown in Figure 4.3. Serrated yielding is observed in the hysteresis loops at both temperatures, indicating solute pinning effects of DSA on mobile dislocations. The serration shape and frequency are significantly different between the two temperatures, with more frequent load drops at 550 °C and greater magnitude load drops at 650 °C. Additionally, the serrations at
650 °C begin at larger strains with increasing number of cycles until they are almost completely non-existent in the tensile region after approximately 50 cycles. The disappearance of serrations at approximately 50 cycles coincides with the maximum peak tensile stress and the onset of cyclic softening. At 550 °C, serrations are observed up to approximately 400 cycles, which also coincides with a maximum in peak tensile stress. One loop in the softening region, cycle 250, is shown for 650 °C to demonstrate the disappearance of serrated yielding during cyclic softening.

![Stress Relaxation Graph](image)

Figure 4.3 Select cyclic hardening hysteresis loops from creep-fatigue tests on heat 011594 with Δε_T = 1 pct at (a) 550 °C and (b) 650 °C. The curves show differences in plastic strain range (loop width) and disappearance of serrated flow.

Stress relaxation during the tensile hold in a creep-fatigue cycle, which is a decrease in stress due to the conversion of elastic strain into inelastic (i.e. creep) strain, is related to the time-dependent deformation mechanisms and the accumulated damage for each cycle. Figure 4.4 shows stress as a function of time during the 30 min tensile hold of the approximate mid-life cycle (N = 500) at 550 °C and 650 °C. Despite the significantly higher stress at the beginning of the hold at 550 °C compared to 650 °C, the amount of stress relaxation is significantly less for the duration of the hold. Specifically, the stress relaxation at mid-life at 550 °C and 650 °C is approximately 20 and 110 MPa, respectively. The magnitude of stress relaxation is directly related to the amount of inelastic deformation that contributes to the width of the hysteresis loop (see Figure 4.3). Thus, the amount of inelastic strain during the hold period (and the accumulation of inelastic strain throughout the duration of the test) is significantly greater at 650 °C than at 550 °C.
However, the magnitude of inelastic strain cannot always be related to the amount of intergranular damage or number of cycles to failure because the strain rate varies during relaxation and different strain rates correspond to different deformation mechanisms [7, 24]. Depending on the alloy and the testing conditions, initial strain rates during relaxation may be high (>10^{-4} \text{s}^{-1}) and correspond to matrix plasticity rather than diffusion-controlled creep deformation, which occurs at lower strain rates. Analysis of strain rates during stress relaxation provides an understanding of the deformation mechanisms and their contribution to grain boundary damage accumulation. The inelastic strain rate, $\dot{\varepsilon}_p$, can be related to the rate at which stress decreases, $\dot{\sigma}$, by Hooke’s law:

$$\dot{\varepsilon}_p = \frac{\dot{\sigma}}{E}$$

where $E$ is the Young’s modulus at the test temperature. The stress during the hold time is fit by linear regression to a power law relation, which can then be differentiated with respect to time to determine strain rate during the hold. For Alloy 709, the strain rates during stress relaxation at 550 °C are low and vary from 2.5×10^{-7} to 2.0×10^{-8} \text{s}^{-1}. The relaxation strain rates at 650 °C are higher, ranging from 1.5×10^{-5} to 6.0×10^{-8} \text{s}^{-1}. Since the relaxation strain rates at both 550 °C and 650 °C are below those expected for matrix deformation, it is interpreted that all of the inelastic strain accumulated during the hold is due to creep mechanisms. Thus, the larger range and

Figure 4.4 Stress relaxation during the 30 min tensile dwell of cycle 500 (near mid-life) at 550 °C and 650 °C.
greater magnitude of relaxation strain rates at 650 °C corresponds to a greater magnitude of creep deformation during the 30 min hold compared to 550 °C.

4.6 Results of Characterization of Tested Microstructures

Microstructural characterization was performed on the gage sections of specimens from heat 011594 tested at both temperatures in LCF and creep-fatigue. The objective of the microstructural characterization was to correlate microstructural damage, dislocation structures, and precipitation to creep-fatigue behavior and life. The link between microstructure and creep-fatigue performance provides insight into the deformation and damage mechanisms active under accelerated and near-service test conditions.

4.6.1 Macroscopic and Microscopic Damage

Damage was qualitatively characterized for the LCF and creep-fatigue specimens by analysis of secondary crack density and crack mode. For both LCF and creep-fatigue conditions, the specimens tested at 550 °C have significantly fewer surface cracks away from the main fracture (i.e. secondary cracks) in the gage section than the specimens tested at 650 °C. Figure 4.5 shows etched light optical micrographs of secondary cracks in all conditions tested to failure. For both temperatures, the LCF tests result in primarily transgranular cracks that originate at the specimen surface. In creep-fatigue, the fracture mode is primarily intergranular at 550 °C and mixed with transgranular and intergranular fracture at 650 °C.

Internal grain boundary cracks (i.e. away from surface crack tips) are present at end of life for both temperatures tested in creep-fatigue, but not in LCF. Intergranular cracks extend along grain boundaries normal to the loading direction. The morphology of intergranular cracks differs between the two temperatures in acuity and length. Intergranular cracks at 550 °C are sharper compared to the more blunted cracks at 650 °C. Additionally, more short and rounded cracks are present at 650 °C compared to 550 °C, which has cracks that frequently span across several grains.

Intergranular damage at end of life was quantified by the total effective intergranular crack length per area at 1 and 2 mm from the surface; the results are shown for both temperatures in Table 4.4. For both depths from the surface, the 550 °C condition has approximately twice the effective crack length compared to 650 °C. As shown in Table 4.3, the creep-fatigue lives are
Figure 4.5 Light optical micrographs of surface cracks from the gage sections of specimens from heat 011594 tested to failure in LCF at (a) 550 °C and (b) 650 °C and in creep-fatigue with a 30 min tensile hold at (c) 550 °C and (d) 650 °C. Transgranular cracking is typical for both temperatures tested in LCF. In creep-fatigue, fractures are primarily intergranular at 550 °C and mixed transgranular and intergranular at 650 °C. The specimen surface is on the right in all images.
comparable at the two temperatures. Thus, internal damage accumulated during approximately the same number of cycles in creep-fatigue is significantly greater at 550 °C than at 650 °C, which correlates with a greater reduction in creep-fatigue life compared to LCF life at 550 °C.

Table 4.4 Total Effective Intergranular Crack Length for Failed Creep-Fatigue Test Samples (mm/mm²)

<table>
<thead>
<tr>
<th>Test Temperature (°C)</th>
<th>1-2 mm from surface</th>
<th>2-3 mm from surface</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>2.9 ± .8</td>
<td>2.6 ± .7</td>
</tr>
<tr>
<td>650</td>
<td>1.5 ± .4</td>
<td>1.4 ± .3</td>
</tr>
</tbody>
</table>

4.6.2 Cyclically Deformed Dislocation Structures

Dislocation structures were studied in the gage section of specimens tested to failure by both LCF and creep-fatigue tests at both temperatures. Figure 4.6 shows representative ECCI micrographs from each of the conditions tested to failure, revealing significant differences in the deformation structures at each temperature in both LCF and creep-fatigue.

Following the LCF test at 550 °C, the dislocation structures are primarily planar, as shown by the linear slip band traces in the bottom left grain in Figure 4.6 (a). Loosely defined dislocation walls are observed in some grains, adjacent to grain or twin boundaries, as shown in the top grain of Figure 4.6 (a). Dislocation walls are defined here as dense clusters of dislocations between regions of the matrix without significant misorientation. Subgrain boundaries, on the other hand, are well-defined tilt or twist boundaries separating regions of the matrix with distinct misorientation, formed during recovery of dislocation walls. The matrix on either side of the dislocation walls at 550 °C shows little contrast variation, indicating that the walls are not recovered subgrain boundaries.

The resulting microstructure after LCF at 650 °C, as shown in Figure 4.6 (b), has significantly more well-defined dislocation walls and less frequent planar structures compared to LCF at 550 °C. Stark contrast variation across cell boundaries at 650 °C indicates recovered subgrain boundaries, which are not observed at 550 °C. Subgrains on the order of approximately 500 nm are frequently observed adjacent to grain and twin boundaries, as indicated by the arrows in Figure 4.6 (b). The formation of subgrains at 650 °C suggests dislocation rearrangement and annihilation to form well-defined boundaries, which is indicative of extensive dislocation cross-slip and climb [4.25].
Figure 4.6 ECCI micrographs of deformed microstructures from the gage sections of specimens from heat 011594 tested to failure in LCF at (a) 550 °C and (b) 650 °C and in creep-fatigue with a 30 min tensile hold at (c) 550 °C and (d) 650 °C. Slip band traces are indicated by arrows with broken lines in both 550 °C microstructures. Regions with dislocation walls are circled in both LCF microstructures. Subgrains are indicated by arrows in the 650 °C LCF condition. A higher density of subgrains is apparent below the crack in the 650 °C creep-fatigue microstructure.
Representative creep-fatigue deformation structures for 550 and 650 °C are shown in Figure 4.6 (c) and (d), respectively. In creep-fatigue at 550 °C, deformation within the grains consists primarily of planar slip bands, as with LCF. Well-defined dislocation walls are less frequent than in the LCF microstructure. However, subgrains with well-defined boundaries are sometimes present directly adjacent to grain boundaries, indicating more dynamic recovery in creep-fatigue than in the case of LCF. The resulting creep-fatigue microstructure at 650 °C is almost completely void of planar structures and dislocation walls. Instead, well-defined subgrains are adjacent to the majority of grain and twin boundaries and extend deeper into the grains than in LCF, as shown in Figure 4.6 (d). Extensive subgrain formation adjacent to boundaries in the 650 °C creep-fatigue microstructure is indicative of dynamic recovery and reduction of strain energy at the boundaries. It should be noted here that the white spherical precipitates shown in all ECCI micrographs are Nb(C,N) that are present in the solution annealed condition.

Figure 4.7 TEM BF micrographs of resulting creep-fatigue dislocation structures in heat 011594 at (a) 550 °C and (b) 650 °C. Deformation at 550 °C is primarily planar with slip bands impinging on grain and twin boundaries (twin boundaries are shown in the figure). At 650 °C, well-defined subgrains are observed near boundaries (grain boundary on the left).
4.6.3 Precipitation

Precipitation is significantly different in Alloy 709 after LCF and creep-fatigue testing at 550 and 650 °C. In LCF tests, the specimens were exposed to 550 °C for an average of 25 h (approximately 4000 cycles) compared to 12 h at 650 °C (approximately 2000 cycles). In creep-fatigue tests, the specimens were exposed to each temperature for approximately the same time of 500 h (the number of cycles to failure was approximately 1000 for both temperatures). Following all test conditions, grain boundaries contain $M_{23}C_6$ precipitates. However, after creep-fatigue testing at 650 °C, grain boundary precipitates are significantly coarser and discrete compared to the rest of the conditions where precipitates are fine and continuous along the boundaries. The finer continuous grain boundary precipitation during creep-fatigue at 550 °C provides more nucleation sites for grain boundary voids, which likely contributes to the higher density of cracked boundaries compared to 650 °C.

LCF at 650 °C results in fine intragranular carbides clustered on dislocation cell walls and subgrain boundaries, which indicates that the dislocation structures provide heterogeneous nucleation sites for precipitation. Figure 4.8 shows a TEM BF micrograph of approximately 20 nm cuboidal carbides clustered on dislocation walls in the 650 °C LCF condition; the precipitates were determined by selected area diffraction (SAD) to be $M_{23}C_6$. After LCF at 550 °C, intragranular $M_{23}C_6$ carbides are observed less frequently than at 650 °C and are significantly smaller (less than 10 nm).

Creep-fatigue testing results in significant differences in intragranular precipitation between the two test temperatures. At 550 °C, $M_{23}C_6$ carbides are approximately 10 to 20 nm and decorate dislocations uniformly throughout the grains. Figure 4.9 (a) shows a BF TEM image of cuboidal carbides near a $[112]$ zone axis with a SAD inset showing the extra reflections from $M_{23}C_6$; the reflections confirm a cube-cube orientation relationship with the lattice and a lattice parameter approximately three times larger than the matrix. It should be noted here that the tilt conditions from the zone axis resulted in a disappearance of the dislocations in Figure 4.9 (a) to better show the precipitates. After creep-fatigue testing at 650 °C, the $M_{23}C_6$ carbides are significantly coarser (approximately 50 nm) and more uniformly distributed compared to the LCF condition. Figure 4.9 (b) shows the coarse $M_{23}C_6$ particles, along with very fine precipitates revealed by the Moiré fringes on dislocations (indicated by arrows on the figure). The inset SAD
Figure 4.8 TEM BF image of fine $M_{23}C_6$ precipitates after continuous cycling at 650 °C. The fine carbides are densely clustered on dislocation walls and in between the walls in a significantly lower density. This micrograph is taken from the gage section of a specimen from heat 011594.

Figure 4.9 TEM BF images of the resulting microstructure after creep-fatigue testing at (a) 550 °C showing cuboidal $M_{23}C_6$ precipitates and (b) 650 °C showing coarse $M_{23}C_6$ precipitates and a distribution of finer precipitates (arrows). Both micrographs are taken near a [112] zone and have SAD insets down zone that show the $M_{23}C_6$ extra reflections as in (a) and additional reflections (circled in inset SAD) in (b) revealing the fine precipitates to be MX. Both micrographs were taken from gage sections of specimens from heat 011594.
pattern of a [112] zone shows extra reflections (circled), in addition to those from M\textsubscript{23}C\textsubscript{6}, which identify the fine precipitates as MX with a cube-cube orientation relationship to the matrix.

### 4.6.4 Dislocation Density and Hardness

Dislocation densities of the deformed microstructures have been estimated from X-ray line broadening. Peak broadening in an X-ray line profile is due to three factors: instrument broadening, crystallite size, and micro-strain from defects like dislocations. Deconvolution of the broadening into these components can be done using an approach first proposed by Williamson and Hall [4.26] and modified by Ungar et al. [4.22] to account for dislocation strain anisotropy. Since broadening from small crystal (i.e. diffracting domain) size is not a function of the diffraction angle and strain broadening is, a plot of broadening versus angle produces a positive slope that is directly related to the amount of micro-strain or dislocation density in the sample.

Figure 4.10 (a) shows the estimated dislocation density for the four conditions tested to failure, alongside Vickers microhardness measurements of the same microstructures in Figure 4.10 (b). The dislocation density and room temperature hardness values show a similar trend; both values are greater after testing at 550 °C compared to 650 °C for both LCF and creep-fatigue. The higher dislocation density after LCF at 550 °C compared to 650 °C is consistent with the differences in the cyclic stress response between the two test temperatures, i.e. cyclic hardening at 550 °C and cyclic softening at 650 °C. These results also agree with the qualitative TEM and ECCI observations of dislocation density and dislocation structures in the two LCF conditions. The dislocation density after creep-fatigue at 550 °C is approximately the same as the LCF test at the same temperature, within the range of measurement error. Similar dislocation densities after LCF and creep-fatigue tests suggests that no significant recovery occurs during the hold time at 550 °C, which is consistent with the small degree of stress relaxation in creep-fatigue. The slight increase in hardness in the creep-fatigue microstructure over LCF is likely due to the fine intragranular (~10-20 nm) M\textsubscript{23}C\textsubscript{6} precipitates shown in Figure 4.9 (a).

Interestingly, the measured dislocation density after creep-fatigue at 650 °C is greater than that after LCF, despite having a lower peak stress prior to failure than the LCF test. Additionally, more recovery is evident in the deformed microstructures after creep-fatigue testing at 650 °C, which indicates a lower dislocation density in the creep-fatigue condition. The reason for the greater measured dislocation density in this condition may be due to additional
peak broadening from coherency strains at the interface between the matrix and very fine MX precipitates, which are shown in Figure 4.9 (b). Coherency strains can contribute to the peak broadening because there are local regions where the lattice parameter deviates from the bulk of the matrix, thus leading to a range of possible Bragg diffracting conditions [4.27], [4.28]. However, the hardness in the specimen tested in creep-fatigue at 650 °C is not significantly different than the LCF specimen tested at the same temperature, which indicates strength loss from dislocation recovery is compensated by strengthening from fine intragranular precipitates.

![Figure 4.10](image)

Figure 4.10 (a) Estimated dislocation density and (b) Vickers hardness measurements and standard deviations from the gage sections of LCF and creep-fatigue test specimens from heat 011594. The resulting dislocation density and hardness were greater in both test conditions at 550 °C compared to 650 °C.

Dislocation density estimates and hardness measurements from the interrupted and failed microstructures are shown in Figure 4.11. Dislocation density at 650 °C follows approximately the same trend as the peak stress versus cycle shown in Figure 4.2; peak dislocation density occurs after 50 cycles, followed by a significant decrease after cyclic softening occurs. This result supports dynamic recovery being partly responsible for the observed cyclic softening during creep-fatigue testing at 650 °C. At 550 °C, dislocation density is approximately the same as 650 °C after 50 cycles and remains relatively unchanged until failure. This result indicates that the softening observed at 550 °C is due in larger part to grain boundary damage rather than microstructural recovery.
Room temperature hardness results for the interrupted tests are shown in Figure 4.11 (b). At 550 °C, hardness increases significantly after 50 cycles, which is a result of dynamic precipitation of fine carbides intragranularly. At 650 °C, a small decrease in hardness was observed after reaching a maximum at 50 cycles. Hardness does not decrease substantially despite a decrease in dislocation density; carbide formation during creep-fatigue offsets the hardness decrease due to the decrease in dislocation density. These results, combined with the microstructural characterization, support more significant dynamic recovery during creep-fatigue testing at 650 °C compared to 550 °C. A summary of the cyclic behavior and corresponding microstructural observations and measurements is provided in Table 4.5.

Figure 4.11 (a) Estimated dislocation density and (b) Vickers hardness measurements and standard deviations from the gage sections of interrupted creep-fatigue test specimens from heat 011594.

4.7 Discussion

4.7.1 Microstructural Evolution and Mechanical Behavior

The LCF cyclic stress response (i.e. hardening or softening) of austenitic stainless steels depends on many factors including initial microstructure, solute-dislocation interaction, strain amplitude, strain rate, temperature, and hold time. Cyclic hardening is due to increasing dislocation interactions with obstacles such as other dislocations, grain boundaries, and
precipitates, and is common for well-annealed materials. Cyclic softening is attributed to recovery by dislocation annihilation and the formation of low energy dislocation structures (LEDS) such as persistent slip-bands (PSBs), walls/veins, cells, or low angle subgrains [25, 29 - 31]. At sub-creep temperatures (i.e. below \(~0.4T_m\)) the formation of LEDS requires extensive cross-slip of screw dislocations, while at higher temperatures, dislocation climb contributes significantly to dynamic recovery and cyclic softening. Slip behavior in materials where deformation is accommodated by extensive cross-slip is characterized as wavy, and is typical for materials with high stacking fault energy (SFE) where partial dislocations can more readily constrict to unit screw dislocations to cross-slip. Austenitic stainless steels, such as Type 316 and 304, are typically considered to deform by planar slip rather than wavy slip, which is attributed to their low SFE [7, 31]. However, high stresses in the vicinity of grain boundaries, where compatibility must be maintained, can induce cross-slip and the formation of LEDS in otherwise planar slip materials [4.25]. Several investigations have shown that cyclic deformation of such alloys results in the formation of LEDS, such as subgrains, which is associated with the onset of cyclic softening [12, 30]. Therefore, a transition from cyclic hardening to softening indicates a transition in slip behavior from planar to wavy and the onset of recovery by cross-slip and/or climb.

The cyclic stress behavior of Alloy 709 in LCF and creep-fatigue is significantly different at 550 and 650 °C, for a given strain range and strain rate, which results from different deformation behavior at the two temperatures. Cyclic softening after initial cyclic hardening in LCF at 650 °C indicates the onset of dynamic recovery by cross-slip and/or climb, and the formation of LEDS. During LCF at 550 °C, only cyclic hardening occurs, due to primarily planar deformation and a lack of recovery for the duration of the test. Microstructural characterization supports primarily planar deformation at 550 °C and the transition to wavy slip character by cell and subgrain formation at 650 °C. Subgrains in the 650 °C LCF condition are observed adjacent to grain boundaries, where the highest compatibility stresses are expected. Dislocation density measurements further support dynamic recovery at 650 °C, where the resulting dislocation density is less than the LCF condition at 550 °C. Thus, the LCF cyclic stress behavior indicates a transition to wavy slip behavior after initial cyclic hardening at 650 °C, but not at 550 °C.

There are further differences in deformation behavior between creep-fatigue tests at 550 and 650 °C, i.e. with the addition of the 30 min tensile dwell. The creep-fatigue cyclic behavior
Table 4.5 Summary of Microstructural Characterization and Cyclic Behavior for Alloy 709

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Tensile Hold Time (min)</th>
<th>Cyclic Behavior</th>
<th>Microstructural and Fracture Observations and Measurements</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>0</td>
<td>Initial hardening (~200 cycles) followed by a slower rate of hardening</td>
<td>Primarily planar deformation; continuous fine grain boundary M$_{23}$C$_6$; little intragranular precipitation; transgranular fracture</td>
</tr>
<tr>
<td>650</td>
<td>0</td>
<td>Initial hardening (~100 cycles) followed by a slower rate of softening</td>
<td>Planar deformation with cell and subgrain formation near boundaries; grain boundary M$_{23}$C$<em>6$; fine (~30 nm) intragranular M$</em>{23}$C$_6$; transgranular fracture</td>
</tr>
<tr>
<td>550</td>
<td>30</td>
<td>Initial hardening (~400 cycles) to a maximum stress 14 pct greater than in LCF followed by softening; little stress relaxation; serrated flow to ~400 cycles</td>
<td>Primarily planar deformation; infrequent cellular / subgrain structures; constant dislocation density and increasing hardness after 50 cycles; fine continuous grain boundary M$_{23}$C$<em>6$; fine (~20 nm) intragranular M$</em>{23}$C$_6$; intergranular fracture</td>
</tr>
<tr>
<td>650</td>
<td>30</td>
<td>Initial hardening (~50 cycles) to a maximum stress 5 pct lower than in LCF followed by a slower rate of softening; significant stress relaxation; serrated flow to ~50 cycles</td>
<td>Significant subgrain formation; decreasing dislocation density and hardness after 50 cycles; coarse discrete grain boundary M$_{23}$C$<em>6$; fine (~50 nm) intragranular M$</em>{23}$C$_6$ and very fine (~8-10 nm) MX; mixed transgranular and intergranular fracture</td>
</tr>
</tbody>
</table>
at 650 °C is similar to the LCF behavior, except that the transition in deformation character from planar to wavy slip occurs after a smaller number of cycles, as shown by the earlier onset of cyclic softening compared to LCF. A significant decrease in dislocation density after the maximum peak stress at 50 cycles shows that the observed cyclic softening correlates with recovery. After creep-fatigue failure, the subgrain density is higher, boundaries are more well-defined, and subgrains extend further into the grains than in the LCF condition. Since the density of subgrains in the creep-fatigue microstructure is significantly greater than that observed in the LCF microstructure, it is interpreted that the increase in dynamic recovery is a result of the creep-strain during stress relaxation. Therefore, the relaxation strain rates at 650 °C, which are in the range expected for creep deformation rather than matrix plasticity, correspond to dislocation-mediated creep deformation (i.e. climb and glide) rather than diffusion-mediated deformation (i.e. Nabarro-Herring or Coble creep).

In creep-fatigue at 550 °C, a transition from cyclic hardening to softening occurs after approximately 400 cycles, which was not observed in LCF. However, this transition in the cyclic stress behavior is not accompanied by significant subgrain formation or a decrease in dislocation density or hardness, as confirmed by the measurements from interrupted tests. The lack of significant microstructural recovery at failure indicates that the cyclic softening at 550 °C is primarily due to grain boundary cracking rather than dislocation-mediated strain energy minimization (i.e. cross-slip and/or climb). Additionally, creep deformation during the tensile hold is significantly smaller compared to 650 °C, as indicated by the narrow hysteresis loops and the magnitude of stress relaxation. Thus, regardless of the creep mechanism at 550 °C, the small amount of creep deformation is partly responsible for the lack of microstructural recovery and the sustained high tensile stresses at peak tensile strain.

Elevated temperature deformation (planar or wavy) in austenitic stainless steels is affected by the presence of solute atoms [4.32]. The presence of nitrogen is well-known to enhance the planar nature of cyclic deformation at intermediate temperatures and strain rates in austenitic stainless steels, which is attributed to DSA effects that prevent dislocation cross-slip necessary for the formation of LEDS [4.6]. Additionally, microstructural instabilities (i.e. precipitate formation and dissolution) during elevated temperature testing from an initially solution annealed condition may be responsible for changes in strengthening and deformation
mechanisms in austenitic stainless steels. For example, high rates of initial cyclic hardening have been attributed to rapid intragranular precipitation during LCF and creep-fatigue in a high temperature ultra-fine precipitate strengthened (HT-UPS) alloy in which DSA does not occur and cannot account for the hardening [4.30]. In creep-fatigue of HT-UPS, a transition from cyclic hardening to softening occurs, which does not happen in LCF, and is attributed to precipitate bypass by dislocation climb during the hold time. For systems that exhibit both DSA and dynamic precipitation during testing, a delay and eventual disappearance of serrated flow occurs and is accompanied by a change in deformation character. The disappearance of serrated flow is attributed to dynamic precipitation, which depletes the solute concentration in the vicinity of mobile dislocations [33 - 35].

Serrated flow in the creep-fatigue hysteresis loops is present at both 550 and 650 °C for Alloy 709, which indicates active DSA under these conditions. Serrations in the hysteresis loops disappear after approximately 50 cycles at 650 °C and after approximately 400 cycles at 550 °C. The disappearance of serrated flow correlates with the onset of cyclic softening and the transition in slip behavior from planar to wavy at 650 °C. Additionally, the initial rate of cyclic hardening is greater at 650 °C than 550 °C in both LCF and creep-fatigue, which is attributed to a difference in precipitate strengthening early in the test between the two temperatures. After both LCF and creep-fatigue, the volume fraction and size of carbides at 650 °C are significantly greater than at 550 °C, indicating faster nucleation and growth at 650 °C. In creep-fatigue, the intragranular carbides after testing at 550 °C (approximately 500 h) are present in a similar size and morphology to those in the 650 °C LCF condition (after approximately 12 h). The M$_2$C$_6$ carbides in the 650 °C creep-fatigue microstructure are significantly coarser (~50 nm) than the LCF condition and a uniform dispersion of fine (~10 nm) MX carbides are also present. A significantly greater consumption of solute atoms in the formation and growth of carbides at 650 °C is likely responsible for the disappearance of serrated flow after approximately 50 creep-fatigue cycles. The reduced DSA corresponds with enhanced tendency for cross-slip and the formation of LEDS. Significant M$_2$C$_6$ coarsening during cycling at 650 °C may also contribute to the onset of cyclic softening simply due to a loss of strength and easier dislocation bypass. Slower precipitate nucleation at 550 °C is responsible for a slower rate of initial cyclic hardening in LCF and creep-fatigue. The slower growth of the precipitates at 550 °C results in cyclic hardening for a longer duration than at 650 °C since the solute concentration remains high.
enough to maintain strain-aging effects and the precipitates remain small enough to effectively pin dislocations. Thus, the significantly higher tensile stresses achieved in creep-fatigue at 550 °C are attributed to prolonged DSA effects, finer precipitation, and less creep deformation compared to 650 °C. The higher tensile stresses resulting from these factors are responsible for a greater amount of intergranular damage and creep-fatigue life reduction at 550 °C compared to 650 °C.

4.7.2 Microstructural Evolution and Damage Formation During Creep-Fatigue

In most austenitic stainless steels and nickel alloys, the creep-fatigue life (with a tensile dwell) is reduced compared to pure LCF, and is typically accompanied by a shift in failure mode from primarily transgranular to primarily intergranular fracture [7, 30, 36]. Intergranular damage is generally characterized by grain boundary cavitation or wedge cracking from grain boundary sliding due to creep deformation processes (i.e. vacancy diffusion). Several mechanisms for creep cavity nucleation have been suggested including impingement of slip-bands on grain boundaries leading to high stress concentrations, nucleation on second phase particles at grain boundaries, and nucleation at grain boundary triple points as a result of grain boundary sliding [4,7]. The mechanism for void nucleation from slip-band impingement may be due to diffusional flow of vacancies to a region of high tensile stress (i.e. Nabarro-Herring or Coble creep). Alternatively, the stress concentration at the grain boundary may be high enough to form a crack nucleus without significant grain boundary or matrix diffusional flow. Grain boundary precipitates are generally considered beneficial to resist creep deformation by grain boundary sliding, and thus prevent wedge cracking. However, stress concentrations at the matrix/particle interface can lead to void nucleation by decohesion or diffusional processes. Cyclic loading leads to the growth and linkage of grain boundary cavitation damage, which results in fewer numbers of cycles to failure than in the absence of significant creep damage.

At both temperatures tested in this investigation, a significant decrease in the number of cycles to failure in creep-fatigue compared to LCF is accompanied by a change in fracture mode from primarily transgranular to primarily intergranular at 550 °C and mixed transgranular/intergranular at 650 °C. This result is consistent with a higher contribution of creep damage (i.e. grain boundary damage) with the introduction of a tensile hold time. The amount of inelastic creep deformation during the hold period is significantly greater at 650 °C than at
550 °C, which would indicate a greater amount of creep cavitation damage at 650 °C. However, the amount of grain boundary damage is significantly greater at 550 °C, despite the small amount of creep deformation. Therefore, it is hypothesized that differences in microstructural evolution (i.e. deformation structures, dislocation-solute interactions, and precipitation) during creep-fatigue testing at the two temperatures results in different mechanisms for damage formation and propagation.

The creep-fatigue life reduction in austenitic stainless steels containing nitrogen is significantly greater compared to the same alloys with low nitrogen content [8, 9]. The shorter creep-fatigue lives are attributed to enhanced slip planarity and a lack of dynamic recovery in nitrogen-containing alloys, leading to high cyclic stresses and large stress concentrations at grain boundaries from slip-band impingement. In Alloy 709, DSA is initially active at both 550 and 650 °C in creep-fatigue, indicating that the slip is primarily planar. The disappearance of serrated flow after the initial cyclic hardening at 650 °C, which is attributed to a reduction of the strain-ageing effect, is accompanied by the onset of cyclic softening and dynamic recovery by dislocation cross-slip and climb. Consumption of solute atoms responsible for DSA is due to inter- and intragranular carbide precipitation at 650 °C. The formation of subgrains adjacent to grain boundaries at 650 °C significantly reduces stress concentrations from impinging slip bands, and therefore, reduces the driving force for damage formation and propagation. Additionally, the grain boundary precipitates coarsen relatively quickly at 650 °C, which reduces the interfacial surface area and number of void nucleation sites.

At 550 °C, little recovery is observed in the creep-fatigue microstructure and serrated flow and cyclic hardening continue up to approximately 400 cycles, resulting in significantly higher peak tensile stresses compared to 650 °C. Although the magnitude of stress relaxation is small, which results in high stresses at grain boundaries for the entire hold, the associated strain rates are indicative of creep deformation. Additionally, fine continuous grain boundary precipitates provide a high density of potent void nucleation sites. Thus, creep cavitation damage is expected during the tensile holds at 550 °C, though the amount is likely less compared to 650 °C. However, a higher density of grain boundary cracks at failure at 550 °C indicates that the damage is not purely due to creep cavitation. Thus, it is hypothesized that at 550 °C the high grain boundary stresses from impinging planar slip bands and a lack of recovery are responsible for the propagation of grain boundary cavities (formed at fine grain boundary precipitates),
resulting in intergranular cracks extending over several grain diameters. The differences in the morphology of grain boundary cracks at the two temperatures provides support for different mechanisms of damage formation and propagation. Grain boundaries cracks at 650 °C are shorter with blunt crack tips compared to intergranular cracks at 550 °C, which are significantly longer with sharper crack tips. Long sharp cracks at 550 °C indicate less plasticity during crack propagation, e.g. through void nucleation and coalescence.

4.8 Conclusions

1. During the tensile hold at 550 °C, sustained high stresses result in a significantly higher degree of grain boundary damage and a greater reduction of creep-fatigue life (relative to the LCF life) compared to creep-fatigue and LCF tests performed at 650 °C. This result indicates that extrapolation of creep-fatigue behavior and life from accelerated testing at elevated temperatures in this study is not indicative of lower temperature behavior. This finding has implications in using accelerated testing to predict service behavior in sodium-cooled nuclear reactors.

2. During creep-fatigue testing, a greater magnitude in stress relaxation was observed during the tensile hold at 650 °C and corresponds to a higher degree of time-dependent creep deformation compared to 550 °C.

3. The disappearance of serrated yielding in the hysteresis loops corresponds to the onset of cyclic softening, a decrease in dislocation density, and a change in dislocation slip behavior from primarily planar to wavy at 650 °C. This change in slip behavior at 650 °C leads to the formation of LEDS (subgrains) through cross-slip and climb, as the strain aging effect on slip planarity is reduced.

4. Deformation structures and dislocation density measurements indicate that dynamic recovery mechanisms, such as subgrain formation and dislocation annihilation, are more active at 650 °C than at 550 °C. Subgrain formation adjacent to grain boundaries at 650 °C reduces stress concentrations compared to impinging planar slip bands present at 550 °C. Thus, there is a lower degree of grain boundary damage at end of life at 650 °C compared to 550 °C, where no significant recovery is observed.

5. Significant differences in microstructural evolution (i.e. dynamic precipitate nucleation and growth) and the corresponding evolution in strengthening mechanisms have profound
effects on the cyclic hardening rate, maximum tensile stress, and the onset of cyclic softening during LCF and creep-fatigue at 550 and 650 °C. A faster rate of precipitate nucleation and coarsening at 650 °C from an initially annealed condition is responsible for a higher initial rate of cyclic hardening, a reduction of the solute strain aging effect, and the onset of cyclic softening at a smaller number of cycles than at 550 °C.

6. Grain boundary precipitation is finer and more continuous after creep-fatigue testing at 550 °C compared to 650 °C. Since grain boundary precipitates are potent void nucleation sites during creep, a higher density of sites is available at 550 °C, which is partly responsible for the increased grain boundary damage.

7. Variations in the homogeneity of the grain structure in the two conditions evaluated in this study does not have a significant impact on LCF and creep-fatigue behavior or cyclic life under the testing conditions used in this study.

4.9 References


CHAPTER 5
MICROSTRUCTURE EVOLUTION OF ALLOY 709 DURING STATIC AGING AND CREEP-FATIGUE TESTING

5.1 Introduction

Structural materials used in elevated temperature applications, such as fossil and nuclear power plants, often undergo significant microstructural evolution during long-term service conditions. Alloy 709 is a candidate alloy for use in structural components in Gen IV sodium cooled fast spectrum nuclear reactors (FSRs) due to its improved creep-resistance over current code qualified structural alloys, Type 316 stainless steel and Grade 91 [5.1]. Alloy 709 is a 20Cr-25Ni-1.5Mo-Nb-N solid solution and precipitation strengthened austenitic stainless steel developed by Nippon Steel for fossil boiler applications, implemented under ASME Code Case 2581 [5.2]. The improved creep properties of Alloy 709 are attributed to fine Nb-containing precipitates such as Z-phase (CrNbN) and MX (Nb(C,N)) [5.2]–[5.5]. Mechanical properties of precipitation strengthened austenitic stainless steels used in the power generation industry are dependent on stability of the microstructure, specifically the formation, dissolution, and coarsening of precipitates [5.6]. Therefore, understanding the evolution of microstructure and its effects on mechanical properties is important for reliable long-term service design.

Precipitation simulations with CALPHAD (calculations of phase diagrams) software, such as Thermo-Calc® and TC-Prisma®, are often used to predict long-term microstructural evolution in materials subject to high-temperature service [5.7]–[5.10]. However, numerical simulations should be validated experimentally in order to produce reliable predictions. Microstructural evolution of NF709 (similar composition to Alloy 709) has been investigated during aging at 750 and 800 °C for times up to 10000 h [5.5]. Undissolved (residual) precipitates were present in the as-received solution annealed material. During short-term aging (1-200 h), M$_{23}$C$_6$ precipitated on grain boundaries and Z-phase precipitated on dislocations. During long-term aging (2500-10000 h), the microstructure contained Cr$_3$Ni$_2$SiX (M$_6$X or η-carbide) precipitates with similar morphology and location to M$_{23}$C$_6$. Additionally, a higher volume fraction of Z-phase was observed after long aging times, which is suggested to be associated with a decrease in the NbN (undissolved precipitates) concentration.
The expected FSR service temperature is 550°C, which is significantly lower than temperatures where Alloy 709 has been employed previously [5.3]. Several initiatives are underway to study the elevated temperature mechanical properties, such as creep and creep-fatigue, at relevant service temperatures. Initial results show that the mechanical behavior depends, in part, on the evolution of the microstructure during testing. Creep-fatigue behavior is significantly different between 550 and 650 °C, which is believed to be attributed to the difference in evolution of the microstructure at the different temperatures [5.11]. Specifically, M$_{23}$C$_6$ and MX carbides nucleate and grow significantly faster at 650 °C, compared to 550 °C, during low cycle fatigue (LCF) and creep-fatigue testing of solution annealed material. The microstructural evolution leads to a change in deformation behavior and damage mechanisms. Therefore, the rapid evolution of strengthening mechanisms (i.e. precipitate and solid solution strengthening) during accelerated laboratory testing may not be representative of long-term service behavior. Understanding the precipitation sequences and kinetics over a range of temperatures, including those expected during service, and the relation to mechanical properties will allow for reliable extrapolation of laboratory test data to long-term service conditions.

The volume fraction and size of precipitates are necessary for determining the strength contributions and deformation behavior at elevated temperatures and to validate and optimize parameters used in precipitation simulations. However, it is challenging to determine volume fractions and size distributions representative of the material from the small volumes and local regions examined through electron microscopy. Small angle scattering (SAS) with X-rays (SAXS) or neutrons (SANS) may be used for more quantitative characterization of nano-scale precipitates in metallic systems, including size and volume fraction, on a more global scale in the bulk material [5.12]–[5.14]. For systems with small precipitate volume fractions like steels, or low atomic contrast (Z-contrast), SANS is generally more useful than SAXS. In order to determine size, size distribution, and volume fraction of the precipitates, the raw scattering data is fit to a model with a known form factor and contrast in scattering length densities (SLD) between the precipitates and the matrix. The former is correlated with the precipitate morphology and the latter with chemical compositions. Both the form factor and SLD contrast are known a priori, typically determined from electron microscopy techniques. Thus, SANS is a complementary technique, along with electron microscopy, for a more complete precipitate characterization.
The objective of this study is to gain an understanding of the microstructural evolution in Alloy 709 at temperatures relevant to service (550 °C) and accelerated laboratory testing (up to 750°C) and at relatively short aging times (1 to 1000 h), relevant to creep and creep-fatigue test durations. Samples from LCF and creep-fatigue tests conducted at 550 and 650°C are characterized after failure and during several interrupted intervals, and compared to static aged (i.e. no load) solution annealed material. A comparison of the microstructures from static aging and from accelerated high temperature testing provides a deeper understanding of the influence of fatigue and creep-fatigue deformation on precipitate nucleation, morphology, and volume fraction. Finally, numerical precipitation simulations are compared to experimental characterization of precipitates in the static aged material using transmission electron microscopy (TEM) and SANS techniques. The results of this study provide insight into how the microstructural evolution during accelerated laboratory fatigue testing differs from that during static aging, which is more representative of long-term service exposure (i.e. lower temperatures and slower accumulation of deformation). Expected differences in creep-fatigue performance of Alloy 709 between accelerated testing and long-term service are discussed with respect to the differences in precipitation during testing and static aging.

5.2 Materials and Methods

5.2.1 Materials and Aging Conditions

The material used for all static aging conditions in this investigation is from a laboratory produced heat of Alloy 709, labeled heat 011593, with the chemical composition given in Table 5.1. The LCF and creep-fatigue tests conducted to failure were from heat 011593. Specimens for interrupted creep-fatigue tests were produced from heat 011594 with a slightly higher content of both C and N, as shown in Table 5.1. Both heats were produced by vacuum induction melting (VIM) and electro-slag remelting (ESR), followed by hot forging and hot rolling, solution annealing at 1100 °C for 1 h, and water quenching. The resulting plates were nominally 25.4 mm thick.

The as-received solution annealed (SA) alloy was isothermally aged in a furnace under no load (i.e. static aging). The static aging was conducted at 550, 650, and 750 °C for 1, 10, 100, 500, and 1000 h (and 2500 h at 650 °C only). For microstructural comparison to the static aged conditions, samples were taken from the gage sections of LCF and creep-fatigue test specimens
tested at 550 and 650 °C, as described in a previous study [5.11]. Both LCF and creep-fatigue
tests were conducted to failure at each temperature, and the creep-fatigue tests were interrupted
at various cycle intervals.

The static aged samples (~10 x 10 mm) for SANS analysis were mechanically polished to
approximately 350 µm thick to minimize multiple scattering (i.e. more than a single scattering
event per neutron). The LCF and creep-fatigue samples (~7.5 mm diameter) were cut from the
gage section, transverse to the loading direction, and thinned to 350 µm.

Specimens for TEM analysis were mechanically polished to ~1 µm, punched into 3 mm
disks, and electro-polished according to the procedure described in a previous study [5.11].

<table>
<thead>
<tr>
<th>Heat ID</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Ti</th>
<th>Nb</th>
<th>N</th>
<th>S</th>
<th>P</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>011593</td>
<td>0.073</td>
<td>0.90</td>
<td>0.39</td>
<td>24.98</td>
<td>19.84</td>
<td>1.51</td>
<td>&lt;0.01</td>
<td>0.25</td>
<td>0.13</td>
<td>0.0008</td>
<td>&lt;0.005</td>
<td>0.004</td>
</tr>
<tr>
<td>011594</td>
<td>0.078</td>
<td>0.90</td>
<td>0.39</td>
<td>25.01</td>
<td>19.89</td>
<td>1.51</td>
<td>&lt;0.01</td>
<td>0.25</td>
<td>0.14</td>
<td>0.0006</td>
<td>&lt;0.005</td>
<td>0.0037</td>
</tr>
</tbody>
</table>

5.2.2 Electron Microscopy

Carbide and nitride precipitates were studied using scanning electron and transmission
electron microscopy (SEM and TEM, respectively). A JEOL 7000 Field Emission Scanning
Electron Microscope (FESEM) was used for low magnification qualitative characterization of
the as-received and the aged microstructures using samples etched with Aqua Regia for 90 sec.
Energy dispersive spectroscopy (EDS) was used in the FESEM for semi-quantitative analysis of
precipitate chemistry. The crystallographic features of precipitates were further characterized
using selected area electron diffraction patterns (SAEDP) in a Philips® CM12 or FEI® Talos
F200X TEM, operating at 120 and 200 kV, respectively.

M$_{23}$C$_{6}$, MX, and Z-phase can be distinguished from one another by their unique
diffraction patterns and specific orientation relationships (OR) with the matrix. M$_{23}$C$_{6}$ and MX
both have an FCC crystal structure (space group $Fm\overline{3}m$) with the same cube-to-cube OR with the
austenite. However, the lattice parameter of M$_{23}$C$_{6}$ is approximately three times greater
(10.57-10.68 Å) than that of austenite (3.599 Å), whereas the MX lattice parameter (4.24-4.47 Å)
is larger than that of austenite, resulting in significantly different diffraction patterns [5.6]. Z-
phase (CrNbN) is a complex nitride with a tetragonal crystal structure (space group $P4/nmm$),
with $a = 3.037$ Å and $c = 7.391$ Å [5.15]. The OR of Z-phase with austenite is (001)$_z$||(001)$_γ$. 

Table 5.1 Composition of Experimental Heats 011593 and 011594 of Alloy 709 (wt pct)
Simulated SAEDP of each phase in austenite are shown in Figure 5.1 down a [001] zone axis (ZA) in the austenite matrix. It should be noted that three variants for Z-phase are shown in Figure 5.1 (c). The FCC precipitates are easily characterized down any zone axis. When multiple variants are present, Z-phase is distinguished from MX in a 2-beam condition where $g = 020$. All SAEDP shown in this study for Z-phase are taken in this 2-beam condition.

![Simulated selected area electron diffraction patterns (SAEDP) of (a) M$_{23}$C$_6$, (b) MX, and (c) Z-phase oriented down a [001] zone axis (ZA) in austenite.](image)

**Figure 5.1** Simulated selected area electron diffraction patterns (SAEDP) of (a) M$_{23}$C$_6$, (b) MX, and (c) Z-phase oriented down a [001] zone axis (ZA) in austenite.

### 5.2.3 Small Angle Neutron Scattering

Small angle neutron scattering (SANS) was used to characterize the volume fraction and size distribution of precipitates in static aged samples and in samples from gage sections of LCF and creep-fatigue tests. Two different SANS instruments, Quokka [5.17] and Bilby [5.18], at the Australian Nuclear Science and Technology Organisation (ANSTO) were used in this
experiment. For the measurements on the time-of-flight instrument Bilby, a pulsed neutron beam with wavelengths of 4-18 Å was used and the scattering data were collected in a Q-range of approximately 0.0015 to 0.5 Å\(^{-1}\). \(Q\) is the scattering vector defined by

\[
Q = \frac{4\pi}{\lambda} \sin \theta \tag{5.1}
\]

where \(2\theta\) is the scattering angle. For the measurements on the monochromatic SANS instrument, Quokka, a neutron beam with a wavelength of 5 Å and three sample-to-detector distances of 2, 8 and 20 m were used. These configurations gave the scattering data in a Q-range of approximately 0.003 to 0.4 Å\(^{-1}\). The reduced SANS data were corrected on an absolute scale against the direct beam intensity.

Due to the similarity in neutron SLD between the matrix (7.15×10\(^{-6}\) Å\(^{-2}\)) and Nb(C,N) (7.83×10\(^{-6}\) Å\(^{-2}\)) and Z-phase (CrNbN) (5.86×10\(^{-6}\) Å\(^{-2}\)), and the low volume fractions of these phases, these precipitates could not be reliably characterized using SANS. However, the SLD of M\(_{23}\)C\(_6\) (4.40×10\(^{-6}\) Å\(^{-2}\)) results in a large enough contrast to be easily distinguished from the matrix using SANS. Additionally, the larger volume fractions of M\(_{23}\)C\(_6\), compared to the fine MX and Z-phase, allowed for characterization of volume fraction and size using SANS. The composition of M\(_{23}\)C\(_6\) used in the SANS analysis was determined from Thermo-Calc® to be (Cr\(_{0.8}\)Mo\(_{0.115}\)Fe\(_{0.075}\)Ni\(_{0.01}\))\(_{23}\)C\(_6\). The fitting was done assuming a Gaussian particle size distribution and no inter-particle scattering. The scattering intensity from the solution annealed (SA) sample assumed a constant structural background for all of the aged conditions; thus, the raw SA data was subtracted from all conditions prior to fitting. The subtracted intensities, \(I(Q)\), were fitted to the following equation:

\[
I(Q) = (\Delta \rho)^2 \Phi_p \int_0^\infty f(R) V_p(R) F(Q,R)^2 \, dR \tag{5.2}
\]

where \(\Delta \rho\) is the difference in the SLD of the precipitate and the matrix, \(f(R)\) is the precipitate particle size distribution function (approximated by a Gaussian function here), \(F(Q,R)\) is the form factor of the precipitate particles, \(V_p(R)\) is the volume of the precipitate particle with a radius of \(R\), and \(\Phi_p\) is the precipitate volume fraction.
5.2.4 Simulations

Equilibrium second phases in Alloy 709 at temperatures ranging from 500 to 1300 °C, as predicted by Thermo-Calc®, include MX, M$_{23}$C$_6$, CrN, Z-phase, Sigma, and Laves. Sigma and Laves intermetallic phases do not form until long aging times in NF709, and are not expected during the relatively short times of this study (up to 1000 h). Furthermore, Cr$_2$N is rarely reported in stabilized austenitic stainless steels and has not been observed in NF709 [5.6], [5.7]. Therefore, only MX, M$_{23}$C$_6$, and Z-phase were included in the precipitation modeling.

The as-received microstructure contains a significant amount of MX ((Nb,Ti)(C,N)), which formed during casting and subsequent thermo-mechanical processing, as discussed elsewhere [5.11]. Therefore, the composition of the solid solution used in precipitation simulations was adjusted to reflect the elements in existing precipitates. For these simulations, the Nb, Ti, C, and N in the carbo-nitrides present in the as-received microstructure were removed from the matrix composition by assuming an equilibrium volume fraction of MX precipitates at the annealing temperature of 1100 °C, which is 0.37 pct. The assumption of an equilibrium volume fraction of MX is justified with kinetic simulations by Shim et al. [5.7] who showed that MX formed at short times during solution annealing at 1200 °C and precipitation was nearly complete by the end of the 1 h treatment. The modified matrix composition (i.e. without the solute content in MX precipitates) was used in this study to estimate equilibrium volume fractions of M$_{23}$C$_6$ and Z-phase (CrNbN) upon thermal aging of the as-received microstructure at the temperatures of interest in this study (550 to 750 °C).

TC-Prisma® simulates the nucleation and growth of precipitates using a Larger-Schwartz approach and thermodynamic data from Thermo-Calc® databases, detailed elsewhere [5.9], [5.10]. Two important factors are needed to determine critical radius for nucleation: the number of nucleation sites and the interfacial energy of precipitates. The number of nucleation sites in the model was calculated by assuming an initial grain size of 50 µm and a dislocation density of $10^{12}$ m$^{-2}$. An interfacial energy of 0.3 J/m$^2$ for M$_{23}$C$_6$ was used for TC-Prisma® calculations, based on the results of a previous study [5.9]. The interfacial energy for Z-phase used in this study was determined from the available databases. Kinetic precipitation simulations were performed at 550, 650, and 750 °C, assuming M$_{23}$C$_6$ nucleates on grain boundaries and Z-phase nucleates on dislocations, based on a previous aging study of NF709 [5.5]. The thermodynamic and mobility databases used were TCFE7 and MOBFE4, respectively.
5.3 Characterization of Precipitates Using SEM and TEM Results

5.3.1 Solution Annealed Microstructure

The microstructure after solution annealing (1100 °C, 1 h) is shown in the SEM micrographs in Figure 5.2. Coarse undissolved (residual) Nb(C,N) are observed in stringers along the rolling direction and are presumably formed during solidification. Additionally, a relatively uniform distribution of fine (~100-200 nm) spherical Nb(C,N) are present on grain boundaries and within the grains. Since MX is an equilibrium phase at the solution annealing temperature (1100 °C), these finer precipitates likely form during the 1 h annealing treatment. Residual Nb(C,N) has been reported in the solution annealed condition of NF709 [5.5].

![Figure 5.2 SEM micrographs of the solution annealed (SA) microstructure of Alloy 709 showing (a) coarse Nb(C,N) stringers along the rolling direction (RD) and (b) fine (~100-200 nm) Nb(C,N) on grain boundaries and within grains.](image_url)

5.3.2 Static Aging

Figure 5.3 shows a BF TEM image of a sample aged at 550 °C for 1000 h; the only precipitates present are the Nb(C,N) that formed during solution annealing. After 1000 h, M_{23}C_{6} is present occasionally as fine particles (~50 nm diameter) on grain boundaries, as shown in Figure 5.3 (b).
Figure 5.3 BF TEM micrographs after aging at 550 °C for 1000 h showing (a) no additional intragranular precipitation other than residual Nb(C,N) and (b) fine M$_{23}$C$_6$ on a grain boundary.

After aging at 650 °C for only 10 h, M$_{23}$C$_6$ is present on grain boundaries and occasionally on incoherent twin boundaries. After 500 h at 650 °C, M$_{23}$C$_6$ is present intragranularly around pre-existing MX, often growing with a rod-like morphology as shown in Figure 5.4 (a). Nucleation of M$_{23}$C$_6$ on MX particles has been reported previously for NF709 [5.5]. The rod-like morphology of M$_{23}$C$_6$ is common in austenitic steels and is explained by cuboidal particles that nucleate on misfit dislocations, forming long chains, often with branches [5.6]. The rods of M$_{23}$C$_6$ after 500 h at 650 °C are approximately 60 nm in diameter and up to 1-2 μm in length. In addition to M$_{23}$C$_6$, fine MX particles (~5-10 nm diameter) decorate dislocations in this condition, as shown in Figure 5.4 (b). The fine MX precipitates are revealed by the closely spaced Moiré fringes. The extra reflections shown in the SAEDP in Figure 5.4 (c) are the (002) reflections of the MX precipitates in Figure 5.4 (b). Coarse globular grain boundary M$_{23}$C$_6$ particles and plate-like incoherent twin boundary M$_{23}$C$_6$ precipitates are shown in Figure 5.4 (d) and (e), respectively. After 1000 h at 650 °C, Z-phase decorates dislocations, as shown in Figure 5.5 (a) with the corresponding SAEDP in Figure 5.5 (b) showing the (1̅10) and (003) reflections of Z-phase. This result indicates that MX transforms into Z-phase in Alloy 709 after sufficient time at temperature, as suggested in the literature [5.6]. Note that the Z-phase particles are approximately the same size after 1000 h as the MX after 500 h. The M$_{23}$C$_6$ rods are approximately the same size after 1000 h as they are after 500 h.
Figure 5.4 BF TEM micrographs after aging at 650 °C for 500 h showing (a) typical intragranular $M_23C_6$ precipitates growing off of a residual Nb(C,N) particle, (b) very fine MX precipitates on dislocations with SAEDP in (c), (d) globular grain boundary $M_23C_6$ precipitates, and (e) plate-like $M_23C_6$ precipitates on an incoherent twin boundary.
Figure 5.5 BF TEM micrographs after aging at 650 °C for 1000 h showing (a) very fine Z-phase decorating dislocations and (b) SAEDP showing (110) and (003) reflections from the precipitates.

Significant M$_{23}$C$_6$ precipitation occurs on grain boundaries after only 1 h at 750 °C and on incoherent twin boundaries after 10 h. After 100 h at 750 °C, intragranular rod-like M$_{23}$C$_6$ particles and fine Z-phase particles are observed, as shown in Figure 5.6. The M$_{23}$C$_6$ rods in this condition are between approximately 100-175 nm in diameter and 1-2 \( \mu \)m in length. Z-phase, which forms during aging at 750 °C for 100 h, has a larger aspect ratio than aging at 650 °C for up to 1000 h. Z-phase coarsens into rods with a diameter of ~10 nm and an aspect ratio of approximately two. The grain and twin boundary precipitates shown in Figure 5.4 (c) and (d) after 500 h at 650 °C are approximately representative of what is observed after 100 h at 750 °C. After 1000 h at 750 °C, the rod-like morphology of Z-phase is more apparent, as shown in the BF and DF TEM micrograph pair in Figure 5.7. The diameter of the Z-phase rods are ~20 nm after 1000 h. The M$_{23}$C$_6$ rods are approximately the same size after 1000 h as the 100 h condition.

5.3.3 LCF and Creep-Fatigue Microstructures

The morphology of intragranular precipitates and their distribution after elevated temperature LCF and creep-fatigue testing is drastically different compared to the microstructures after static aging at the same temperatures. Additionally, dynamic precipitation of intragranular carbides and nitrides on a relatively high density of dislocations, occurs faster
compared to static aging. The precipitates formed during LCF and creep-fatigue testing at 550 and 650 °C are described in detail in a previous study [5.11]. Figure 5.8 (a) shows a relatively uniform distribution of individual cuboidal $M_23C_6$ carbides (~50 nm) after creep-fatigue testing at 650 °C for approximately 500 h (i.e. after failure). Additionally, a distribution of fine MX (~5-10 nm) exists on dislocations and in the matrix, shown at higher magnification in Figure 5.8 (b). MX is assumed to nucleate on dislocations [5.6]. Therefore, MX particles in the matrix indicate that dislocations bypass the particles by a climb mechanism. Although the morphology of the intragranular $M_23C_6$ carbides in the static aged condition (i.e. chains of cuboidal

Figure 5.6 BF TEM micrographs after aging at 750 °C for 100 h showing intragranular (a) rod-like $M_23C_6$ and fine Z-phase decorating dislocations, which are shown at higher magnification in (b) with corresponding SAEDP in (c).
precipitates) is significantly different than after fatigue testing (*i.e.*, individual cuboidal precipitates), it should be noted that the grain boundary precipitate morphology is not significantly different between the aging and fatigue conditions.

The experimentally determined phases and morphologies of intragranular precipitates are summarized in Table 5.2 for static aging, LCF, and creep-fatigue conditions.

![Figure 5.7 (a) BF and (b) DF TEM micrographs (using 002 and 003 reflections) of Z-phase rods on a dislocation after aging at 750 °C for 1000 h.](image)

**5.4 Small Angle Neutron Scattering Results**

**5.4.1 Static Aging**

The raw scattering data, which is displayed as intensity versus scattering vector, *Q*, for all static aged conditions is shown in Figure 5.9. All of the data shown is from the Bilby instrument, except for the conditions aged for 1000 h at 650 and 750 °C, which are from the Quokka instrument which has a slightly smaller *Q*-range. The SA condition is included for comparison at each aging temperature. Increased scattering intensity over the SA condition indicates the presence of precipitates. At 550 °C, the scattering profile is relatively unchanged until 1000 h, indicating no significant precipitation up to that point. The slight increase in scattering after 1000 h, compared to the SA condition, indicates some precipitation; although the volume fraction and size distribution could not be confidently determined from the data. At 650 °C, there
Figure 5.8 BF TEM micrographs of cuboidal (a) $M_{23}C_6$ and (b) MX precipitates after creep-fatigue failure at 650 °C.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Aging Condition and Time (h)</th>
<th>Precipitate Morphology and Location</th>
<th>Approximate Size (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>Static; 1000</td>
<td>Only residual Nb(C,N)</td>
<td>Residual MX: 100-200</td>
</tr>
<tr>
<td>650</td>
<td>Static; 500</td>
<td>Chains of cuboidal $M_{23}C_6$ adjacent to residual Nb(C,N); cuboidal MX on dislocations</td>
<td>$M_{23}C_6$: 50-100 x 1000-2000; MX: 5-10</td>
</tr>
<tr>
<td></td>
<td>Static; 1000</td>
<td>Chains of cuboidal $M_{23}C_6$ adjacent to residual Nb(C,N); cuboidal Z-phase on dislocations</td>
<td>$M_{23}C_6$: 50-100 x 1000-2000; Z-phase: 5-10</td>
</tr>
<tr>
<td>750</td>
<td>Static; 100</td>
<td>Chains of cuboidal $M_{23}C_6$ adjacent to residual Nb(C,N); rod Z-phase on dislocations</td>
<td>$M_{23}C_6$: 100-175 x 1000-2000; Z-phase: 10 x 20</td>
</tr>
<tr>
<td></td>
<td>Static; 1000</td>
<td>Chains of cuboidal $M_{23}C_6$ adjacent to residual Nb(C,N); rod Z-phase on dislocations</td>
<td>$M_{23}C_6$: 100-175 x 1000-2000; Z-phase: 20 x 40</td>
</tr>
<tr>
<td>550</td>
<td>LCF; ~28</td>
<td>Cuboidal $M_{23}C_6$ on dislocations</td>
<td>$M_{23}C_6$: &lt;10</td>
</tr>
<tr>
<td>550</td>
<td>CF; ~450</td>
<td>Cuboidal $M_{23}C_6$ on dislocations</td>
<td>$M_{23}C_6$: 10-20</td>
</tr>
<tr>
<td>650</td>
<td>LCF; ~12</td>
<td>Cuboidal $M_{23}C_6$ on dislocations</td>
<td>$M_{23}C_6$: 20</td>
</tr>
<tr>
<td>650</td>
<td>CF; ~500</td>
<td>Cuboidal $M_{23}C_6$ and MX on dislocations</td>
<td>$M_{23}C_6$: 50; MX: 5-10</td>
</tr>
</tbody>
</table>

Low cycle fatigue (LCF); Creep-fatigue with 30 min tensile hold (CF)
is a slight increase in scattering over the SA condition after 10 h, which indicates some precipitation. However, significant precipitation does not occur until 100 h at 650 °C, at which point the increased scattering over the SA condition is sufficient to quantify the precipitate volume fraction and size. At times longer than 500 h, the scattering is relatively unchanged, which indicates no further precipitation after that time. At 750 °C, significant precipitation occurs after only 1 h and is approximately complete after 100 h. All conditions, except 1000 h at 650 and 750 °C, were scanned on both instruments with good agreement between them.

The individual aging conditions were fit using two different models: one for the boundary (grain and twin) M23C6 and one for the intragranular M23C6 particles. The grain and twin boundary precipitates were characterized assuming an ellipsoidal shape for both according to the TEM micrographs shown in Figure 5.4 (d) and (e). In reality, the twin boundary particles have a plate morphology, but the ellipsoid model was applied to these precipitates for simplicity. The aspect ratio of the ellipsoid did not have a significant effect on the results, so it was fixed at a value of 2 for all conditions. Thus, the size and volume fraction of the boundary precipitates is an average of both grain and twin boundary particles. The intragranular precipitates were characterized using a cylinder model based on the morphology of the chains of cuboidal precipitates shown in Figure 5.4 (a) and Figure 5.6 (a). Although M23C6 rods were observed up to 1-2 μm in length, the observation limit using SANS is approximately 500 nm. Therefore, the cylinder length in the model was fixed to 500 nm for all conditions.

The resulting precipitate sizes and volume fractions for each of the static aged conditions are summarized in Table 5.3. The total volume fraction and precipitate sizes are generally greater at 750 °C than at 650 °C for a given aging time. For example, the total volume fraction of M23C6 is approximately the same after 500 h at 650 °C and after 100 h at 750 °C. For most aging times, the intragranular volume fraction is greater at 750 °C compared to 650 °C. However, it is interesting to note that the measured intragranular volume fraction is slightly greater at 650 °C than at 750 °C after 500 h. It is also worth noting that the estimated volume fraction of grain boundary (ellipsoid) carbides is greater than the intragranular (cylindrical) carbides at 750 °C for all aging times, but the opposite is true at 650 °C after 500 h.
Figure 5.9 Raw SANS data presented as intensity versus the reciprocal of scattering vector for conditions static aged at (a) 550 °C, (b) 650 °C, and (c) 750 °C for 1, 10, 100, 500, and 1000 h compared to the SA condition (aged 0 h). (b) also includes a condition aged at 2500 h.
5.4.2 LCF and Creep-Fatigue

The raw scattering data for the LCF and creep-fatigue conditions are shown in Figure 5.10 compared to the failed creep-fatigue conditions at each temperature. The increase in scattering intensity compared to the SA conditions in all cases indicates significant precipitation during even the relatively short LCF tests (~28 h at 550 °C and ~10 h at 650 °C). Note that the scattering of the 650 °C LCF condition is similar to the 550 °C creep-fatigue condition, which was at temperature for ~490 h longer, indicating similar precipitation in a much shorter time at 650 °C (Figure 5.10 (a)). In Figure 5.10 (b), the neutron scattering data from the creep-fatigue tests performed at 550 °C and interrupted at 50, 400, and 600 cycles are compared to the test conducted to failure (~888 cycles). Significant precipitation does not occur during creep-fatigue at 550 °C until between 50 and 400 cycles (~25 and 200 h, respectively). In Figure 5.10 (c), the raw scattering data from the creep-fatigue tests performed at 650 °C and interrupted at 25, 50, and 400 cycles are compared to the test conducted to failure (~1000 cycles). Significant precipitation occurs after 25 cycles (~12.5 h) at 650 °C and increases with number of cycles until 400 cycles (~300 h). Note that for creep-fatigue tests at both 550 and 650 °C, the scattering

### Table 5.3 Summary of Average Size and Volume Fraction of M$_2$C$_6$ Precipitates in Static Aged Samples from SANS Analysis

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Aging Time (h)</th>
<th>Boundary Ellipsoid Radius (nm)</th>
<th>Boundary Ellipsoid Vol. Fraction (pct)</th>
<th>Intragrannular Cylinder Radius, Length (nm)</th>
<th>Intragrannular Cylinder Vol. Fraction (pct)</th>
<th>Total Vol. Fraction (pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>1, 10, 100, 500, 1000</td>
<td>-</td>
<td>0</td>
<td>-</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>650</td>
<td>1, 10</td>
<td>-</td>
<td>0</td>
<td>-</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>650</td>
<td>100</td>
<td>36.0</td>
<td>0.20</td>
<td>10.0, 500</td>
<td>0.16</td>
<td>0.36</td>
</tr>
<tr>
<td>650</td>
<td>500</td>
<td>68.0</td>
<td>0.32</td>
<td>22.0, 500</td>
<td>0.55</td>
<td>0.87</td>
</tr>
<tr>
<td>650</td>
<td>1000</td>
<td>76.0</td>
<td>0.49</td>
<td>24.0, 500</td>
<td>0.52</td>
<td>1.01</td>
</tr>
<tr>
<td>650</td>
<td>2500</td>
<td>80.0</td>
<td>0.44</td>
<td>30.0, 500</td>
<td>0.56</td>
<td>1.00</td>
</tr>
<tr>
<td>750</td>
<td>1</td>
<td>21.4</td>
<td>0.09</td>
<td>-</td>
<td>0</td>
<td>0.09</td>
</tr>
<tr>
<td>750</td>
<td>10</td>
<td>61.0</td>
<td>0.37</td>
<td>19.0, 500</td>
<td>0.29</td>
<td>0.66</td>
</tr>
<tr>
<td>750</td>
<td>100</td>
<td>76.6</td>
<td>0.56</td>
<td>20.0, 500</td>
<td>0.31</td>
<td>0.87</td>
</tr>
<tr>
<td>750</td>
<td>500</td>
<td>82.1</td>
<td>0.55</td>
<td>25.5, 500</td>
<td>0.45</td>
<td>1.00</td>
</tr>
<tr>
<td>750</td>
<td>1000</td>
<td>79.0</td>
<td>0.62</td>
<td>33.6, 500</td>
<td>0.6</td>
<td>1.22</td>
</tr>
</tbody>
</table>
Figure 5.10 Raw SANS data for (a) failed LCF and creep-fatigue conditions at 550 and 650 °C, (b) creep-fatigue tests interrupted at 50, 400, and 600 cycles at 550 °C, and (c) creep-fatigue tests interrupted at 25, 50, and 400 cycles at 650 °C. (b) and (c) also include failed creep-fatigue samples at the respective temperatures, which were both approximately 1000 cycles. The data in the SA condition is also included in (a)-(c) for comparison.
intensity (and precipitate volume fraction) is lower at failure than for the longest interrupted tests (600 cycles at 550 °C, 400 cycles at 650 °C). This result indicates that the slightly higher C concentration in heat 011594 used in the interrupted tests results in higher volume fractions of carbides at shorter times.

The models used to fit the data from LCF and creep-fatigue tests were based on the precipitate morphology observed with electron microscopy. The intragranular M$_{23}$C$_6$ precipitates in the fatigue conditions are individual cuboids, rather than rod shapes from cuboids chained together in the static aged condition. Therefore, a cube shape model is used rather than the cylinder model for the intragranular precipitates in the aged condition. However, the grain and twin boundary precipitate morphology is not significantly different than in the static aged condition; thus, the same ellipsoid model is used for boundary precipitates.

The resulting precipitate sizes and volume fractions for the LCF and creep-fatigue conditions are summarized in Table 5.4. At both temperatures, the volume fractions of intragranular carbides are greater than the grain boundary volume fractions in all conditions. Additionally, the total precipitate volume fractions and sizes are significantly greater at 650 °C compared to 550 °C for similar testing times. The average intragranular precipitate sizes after LCF and creep-fatigue failure, determined from the SANS data, correspond well with the sizes reported in a previous study based on TEM measurements [5.11]. For example, the cube radii reported from TEM analysis for the failed 550 and 650 °C creep-fatigue tests are ~5-10 and ~25 nm, respectively. The corresponding radii determined by SANS in this study are 4.7 and 22.3 nm.

5.5 Thermodynamic and Kinetic Simulations Results

The equilibrium phase fractions from the full composition and the modified matrix composition are summarized in Table 5.5 from 550 to 750 °C. Note that MX is not an equilibrium phase below ~980 °C. The volume fraction of M$_{23}$C$_6$ is not affected significantly by the presence of residual Nb(C,N) from solution annealing. However, the equilibrium fraction of Z-phase is approximately an order of magnitude smaller when complete Nb(C,N) precipitation is assumed at the annealing temperature of 1100 °C. The results of the kinetic precipitation simulations are shown in Figure 5.11, which is a partial time-temperature-precipitation (TTP) diagram for M$_{23}$C$_6$ and Z-phase between 550 and 750 °C. The contours in the figure represent
### Table 5.4 Summary of Size and Volume Fractions of M$_{23}$C$_6$ Precipitates in LCF and Creep-Fatigue Samples from SANS Analysis

<table>
<thead>
<tr>
<th>Temperature ($^\circ$C)</th>
<th>Test Condition (condition, cycles, h)</th>
<th>Boundary Ellipsoid Radius (nm)</th>
<th>Boundary Ellipsoid Vol. Fraction (pct)</th>
<th>Intragranular Cuboid Radius (nm)</th>
<th>Intragranular Cuboid Vol. Fraction (pct)</th>
<th>Total Vol. Fraction (pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>LCF, 4964, 28</td>
<td>19.5</td>
<td>0.02</td>
<td>3.7</td>
<td>0.11</td>
<td>0.13</td>
</tr>
<tr>
<td>650</td>
<td>LCF, 1883, 10</td>
<td>31.0</td>
<td>0.08</td>
<td>8.1</td>
<td>0.20</td>
<td>0.28</td>
</tr>
<tr>
<td>550</td>
<td>CF, 50, 25</td>
<td>-</td>
<td>0</td>
<td>-</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>550</td>
<td>CF, 400, 200</td>
<td>30.0</td>
<td>0.06</td>
<td>4.3</td>
<td>0.24</td>
<td>0.30</td>
</tr>
<tr>
<td>550</td>
<td>CF, 600, 300</td>
<td>36.0</td>
<td>0.07</td>
<td>5.3</td>
<td>0.36</td>
<td>0.43</td>
</tr>
<tr>
<td>550</td>
<td>CF, 888, 450</td>
<td>27.6</td>
<td>0.09</td>
<td>4.7</td>
<td>0.16</td>
<td>0.25</td>
</tr>
<tr>
<td>650</td>
<td>CF, 25, 12.5</td>
<td>25.3</td>
<td>0.05</td>
<td>7.7</td>
<td>0.07</td>
<td>0.12</td>
</tr>
<tr>
<td>650</td>
<td>CF, 50, 25</td>
<td>32.0</td>
<td>0.10</td>
<td>11.5</td>
<td>0.22</td>
<td>0.32</td>
</tr>
<tr>
<td>650</td>
<td>CF, 400, 200</td>
<td>32.0</td>
<td>0.27</td>
<td>17.0</td>
<td>0.70</td>
<td>0.97</td>
</tr>
<tr>
<td>650</td>
<td>CF, 1063, 500</td>
<td>46.7</td>
<td>0.40</td>
<td>22.3</td>
<td>0.40</td>
<td>0.80</td>
</tr>
</tbody>
</table>

### Table 5.5 Summary of Equilibrium Precipitate Volume Fractions

<table>
<thead>
<tr>
<th>Temperature ($^\circ$C)</th>
<th>Composition</th>
<th>M$_{23}$C$_6$ (vol. pct)</th>
<th>Z-Phase (vol. pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>550 - 750</td>
<td>Full – No residual MX</td>
<td>1.73 - 1.69</td>
<td>0.460 - 0.468</td>
</tr>
<tr>
<td>550 - 750</td>
<td>Modified – With 0.37 vol. pct residual MX</td>
<td>1.40 - 1.36</td>
<td>0.062 - 0.049</td>
</tr>
</tbody>
</table>

104
10, 50, and 90 pct of the equilibrium volume fraction of the modified matrix composition. M$_{23}$C$_6$ nucleates and grows relatively quickly compared to Z-phase, which is consistent with the literature [5.5], [5.6]. At 750 °C, M$_{23}$C$_6$ reaches 90 pct of the equilibrium volume fraction after only 130 h, compared to 2700 h at 550 °C. Z-phase reaches 90 pct of the equilibrium volume fraction after 200 h at 750 °C and greater than 9000 h at 550 °C. The simulated precipitation results indicate the relative stability of Alloy 709 at 550 °C, which is the expected service temperature for FSRs.

![Figure 5.11 Predicted time-temperature-precipitation (TTP) diagram for M$_{23}$C$_6$ and Z-Phase from 550 to 750 °C. The contours represent 10, 50, and 90 pct of the equilibrium volume fraction at the respective temperatures.](image)

5.6 Discussion

5.6.1 Static and Dynamic Precipitation

The intragranular carbides that form during LCF testing are significantly different in both morphology and volume fraction compared to those that precipitate during static isothermal aging. Specifically, dynamic precipitation results in a relatively uniform distribution of fine cuboidal M$_{23}$C$_6$ particles, while static aging results in long chains of cuboidal particles. Furthermore, the volume fraction of intragranular precipitates is generally greater at shorter times in the LCF and creep-fatigue microstructures compared to the static aged conditions for the same temperatures. The difference in volume fraction between static aging and fatigue testing is especially apparent at 550 °C, where no significant precipitation occurs after 1000 h of static aging.
aging, but a significant volume fraction of fine carbides are present after the approximately 200 h of creep-fatigue testing (400 cycles).

There are several factors that contribute to the differences in dynamic and static precipitation including the number of nucleation sites (i.e. dislocations) and corresponding enhanced solute mobility due to pipe diffusion and a higher vacancy concentration from deformation. Since $M_{23}C_6$ nucleates on dislocations and particle interfaces, the significant increase in dislocation density during fatigue increases the number of sites for carbide nucleation. The increase in dislocation density and vacancy concentration also contributes to enhanced solute diffusion for nucleation and growth of precipitates. Additionally, the presence of serrated yielding during LCF and creep-fatigue testing at 550 and 650 °C indicates dynamic strain aging (DSA) under these conditions [5.11]. DSA is attributed to solute atmospheres that form on arrested dislocations and impart a dragging force on the dislocations once they are mobile. Solute atmospheres around dislocations represent a local increase in solute concentration [5.19], which increases the driving force for precipitate nucleation. DSA in austenitic stainless steels, including Alloy 709, is attributed to both interstitial (C and N) and substitutional (e.g. Cr) atoms [5.20], [5.21]. Therefore, the high local solute concentration at dislocations due to DSA and the large number of nucleation sites (i.e. dislocations) created during LCF are responsible for the higher volume fraction and particle density in the fatigue specimens compared to the static aged material.

5.6.2 Effect of Microstructure Evolution on Mechanical Properties

The differences in precipitate morphology and volume fraction between static and dynamic aging have a significant effect on the strengthening mechanisms and deformation behavior during accelerated testing and long-term service conditions. Particle strengthening for non-shearable precipitates (e.g. carbides and nitrides) can be estimated by either an Orowan bowing mechanism or a threshold stress at temperatures where dislocation climb occurs [5.22]. In either estimation, the increase in shear stress due to precipitates is inversely related to the mean particle spacing, $\lambda$, given by

$$\lambda = r \frac{2\pi}{\sqrt{3fV}}$$ 5.3
where $r$ is the particle radius (assuming a sphere) and $f_i$ is the volume fraction. An increase in particle size for a given volume fraction results in a smaller increase in strength. Additionally, a smaller volume fraction for a given size has the same effect on strength. Therefore, the smaller size of carbides produced during LCF and creep-fatigue provide a greater strength increase compared to the coarse chains of carbides in the static aged condition, even though the volume fractions are comparable. For example, the difference in strength between static and dynamic precipitation of $M_{23}C_6$ carbides is estimated at 650 °C for similar aging times; i.e. the 500 h static aged condition and the failed creep-fatigue condition (~500 h). Although the diameter of the carbide chains is not significantly different than the individual cuboids in the creep-fatigue condition, the volume of a typical carbide chain is nearly two orders of magnitude larger than the individual cuboids. The effective radius for a chain, which is the radius of a sphere with the same volume of a cylindrical chain, is ~89 nm or ~3.75 times larger than the radius of the individual precipitates (~24 nm). Therefore, the strength increase from individual precipitates at 650 °C in the creep-fatigue condition is estimated to be nearly four times greater than from the coarse chains of carbides that form after static aging at the same time and temperature.

The difference in volume fraction of precipitated carbides and nitrides between static and dynamic aging also has a significant influence on solid solution strengthening at elevated temperatures. Solute atmospheres form around dislocations at elevated temperatures and impose a dragging stress on both gliding and climbing dislocations. The dragging stress from solutes has been shown to be directly proportional to the solute concentration in the alloy and to the size misfit of the solute atoms with the matrix atoms squared [5.23]. The relatively large size misfit of C and N in austenite provide significant strength increases, despite relatively small concentrations (compared to substitutional atoms) [5.24]–[5.27]. Furthermore, interstitial solutes have a significant effect on the constriction energy of partial dislocations in FCC alloys, which acts to inhibit dislocation cross-slip, thus promoting planar slip and high rates of strain-hardening [5.19], [5.26], [5.28], [5.29]. Hence, the strain-hardening behavior is also associated with interstitial solute concentration. Precipitation of carbides and nitrides results in a decrease of interstitial solute concentration. Therefore, it is expected that the magnitude of solid solution strengthening and the strain-hardening behavior are functions of the precipitate volume fraction.

During creep-fatigue testing at 650 °C, rapid initial hardening is followed by cyclic softening after approximately 50 cycles, which correlates with dynamic recovery due to increased cross-
slip [5.11]. After approximately 50 cycles (~25 h), the carbide volume fraction (0.32 pct) is approximately the same as after 100 h of static aging at the same temperature. Furthermore, this same volume fraction is not achieved until approximately 400 cycles (~200 h) at 550 °C, which explains the significantly higher strength, prolonged cyclic hardening, and a lack of dynamic recovery compared to creep-fatigue testing at 650 °C. During static aging at 550 °C, a significant volume fraction of carbides does not precipitate until beyond 1000 h. Thus, the high rate of carbide and nitride precipitation during fatigue testing is expected to result in significantly different cyclic behavior in accelerated laboratory tests than after static aging, which may be more representative of long-term service exposure.

The results of this study have significant implications on extrapolating accelerated laboratory test data to long-term service conditions due to the expected differences in strengthening mechanisms and deformation behavior as a result of the differences in microstructure evolution. For example, the microstructure evolution at 550 °C is accelerated during the relatively short-term creep-fatigue test, resulting in a higher volume fraction of fine precipitates compared to static aging for significantly longer times. The strength due to fine precipitates, which form as a result of high dislocation density and DSA during the test, is not representative of the strength from coarse precipitation that form after long times with small amounts of plastic deformation (i.e. during service). Additionally, the effect of solutes on dislocation motion and cross-slip is affected by the precipitate evolution. Thus, the deformation behavior from tests at high temperatures, where the solute concentration rapidly decreases, may not be representative of the deformation at lower service temperatures, where precipitation (and solute consumption) are significantly slower. Accelerated laboratory tests (creep and creep-fatigue) may be necessary to obtain a significant amount of data in a reasonable time. However, extrapolation of test data to long-term service conditions should be done with a strong consideration of the influence of microstructure evolution on the mechanical behavior in Alloy 709.

5.6.3 TTP Validation

The predicted $\text{M}_{23}\text{C}_6$ precipitate volume fractions were compared to the experimentally determined volume fractions from the SANS data. At 550 °C, approximately 10 pct of the equilibrium volume fraction (0.14 pct) is expected after ~900 h. Although the SANS data for the
1000 h condition could not be fit accurately due to the low intensities, there is slightly more scattering than in the SA condition, indicating some amount of additional precipitation. Additionally, fine $M_{23}C_6$ particles are observed on grain boundaries after 1000 h, indicating that this phase does form before 1000 h, as predicted.

At 650 °C, 10 pct of the equilibrium volume fraction is expected after approximately 114 h. After 100 h, the measured volume fraction is approximately 26 pct of equilibrium, indicating that nucleation and initial growth of $M_{23}C_6$ is faster than predicted. 90 pct of the equilibrium volume fraction (1.25 pct) is expected after approximately 350 h at 650 °C. However, only ~63 pct of the equilibrium fraction is measured after 500 h, indicating that the precipitate coarsening is slower than predicted. In fact, after 2500 h of static aging, only approximately 71 pct of the expected equilibrium volume fraction is measured.

At 750 °C, the nucleation and initial growth of $M_{23}C_6$ is significantly faster than predicted. After 10 h of static aging, nearly 50 pct of the equilibrium fraction is measured, which is not predicted until approximately 93 h of aging. Similar to the results at 650 °C, 90 pct of the equilibrium volume fraction (1.22 pct) is not measured until aging times significantly longer than predicted (1000 h at 750 °C compared to approximately 143 h predicted).

Overall, the static aging results show that $M_{23}C_6$ nucleates faster than predicted at temperatures higher than 550 °C, but the expected equilibrium volume fraction is not reached until significantly longer times than expected. The results indicate that parameters used in the precipitation model, like interfacial energy, nucleation sites, and mobility can be optimized for this system. The interfacial energy is assumed to be a constant value, when in reality this value may change with precipitate coarsening. The nucleation sites in this model were assumed to be only on grain boundaries for $M_{23}C_6$. However, nucleation was also observed on incoherent twin boundaries and existing Nb(C,N) particles. Differences in predicted interfacial energy and nucleation sites, versus actual values, may be attributed to the higher than expected volume fractions after short aging times in this study. Furthermore, the mobility of Cr is known to be affected by the presence of N due to the strong affinity for one another [5.27]. The effect of N on reducing the mobility of Cr may be responsible for the lower than expected volume fractions of $M_{23}C_6$ after long aging times.

Although the volume fraction of Nb(C,N) and Z-phase could not be measured from SANS experiments, the absence or presence of the precipitates are discussed qualitatively with
respect to the simulated TTP. Z-phase is identified on dislocations after 100 h at 750 °C, which correlates with the expected time to precipitate approximately 50 pct of the equilibrium volume fraction (0.024 pct) at that temperature. Z-phase precipitates are significantly coarser after 1000 h at 750 °C, where greater than 90 pct of the equilibrium volume fraction (0.044 pct) is expected for the modified composition. However, without measuring the volume fraction, it is unclear whether coarsening occurs by continued precipitation or by a Gibbs-Thompson effect (i.e. particle coarsening at the expense of smaller particles). Z-phase is reported to grow at the expense of residual NbN in NF709 at times longer than 200 h of aging, due to the fact that MX is not the equilibrium nitride at temperatures below ~980 °C [5.5]. Therefore, the true equilibrium volume fraction of Z-phase, which is determined from the full alloy composition in Table 5.5, is expected to eventually be achieved by the complete dissolution of NbN. While a numerical simulation of this reaction (NbN → Z-phase) is outside the scope of this investigation, the presence of Z-phase after only 100 h at 750 °C indicates relatively fast nucleation and growth at this temperature.

At 650 °C, Z-phase is also predicted to be at approximately 50 pct of the equilibrium volume fraction after 500 h. However, TEM SAEDPs suggest the fine precipitates on dislocations are MX, not Z-phase. MX is also present on dislocations and in the matrix after creep-fatigue testing at 650 °C for approximately 500 h. After 1000 h, diffraction from the precipitates reveals (003) reflections belonging to Z-phase. This result indicates that MX is an intermediate metastable phase and a precursor to Z-phase formation in Alloy 709, as suggested in literature for other 20Cr-25Ni alloys [5.6].

### 5.7 Conclusions

1. The morphology and volume fraction of precipitates that form during static aging are different than those that form during LCF and creep-fatigue testing. Precipitates form significantly faster during fatigue testing, as a result of higher dislocation density and higher solute mobility. The fine cuboidal carbides that form on dislocations during fatigue testing are expected to provide nearly four times the strength compared to coarse chains of M\textsubscript{23}C\textsubscript{6} carbides that form during static aging.

2. Precipitation of carbides and nitrides reduces the interstitial solute concentration, which is expected to reduce the solution strengthening and the friction stress that promotes high
strain-hardening rates in Alloy 709. Therefore, the more rapid precipitation during cyclic testing results in a faster evolution of strengthening and deformation mechanisms compared to static aging, which is more representative of long-term exposure.

3. Precipitation also occurs significantly faster at temperatures greater than the expected FSR service temperature of 550 °C. Therefore, accelerated laboratory testing with solution annealed material, at temperatures and strains higher than expected during service, results in accelerated microstructural evolution compared to long-term service at lower temperatures and strains. The results of this study indicate the significance of the microstructural evolution on extrapolation of mechanical properties during laboratory tests to expected service lifetimes.

4. The volume fractions of $M_23C_6$ carbides, measured from SANS, during static aging are higher than expected from TC-Prisma® calculations at short aging times, indicating faster nucleation than predicted. At long aging times, the volume fractions are smaller than predicted, indicating slower growth of the carbides than expected. These results indicate that modelling parameters such as interfacial energy, nucleation sites, and mobility can be improved for this system.

5. Z-phase is present after 100 h at 750 °C and after 1000 h at 650 °C, as predicted from the kinetic simulations. No intragranular precipitates are observed after 1000 h at 550 °C, which is also expected from the modeling results.

5.8 References


[5.5] T. Sourmail and H. K. D. H. Bhadeshia, “Microstructural evolution in two variants of


CHAPTER 6
DEFORMATION AND DAMAGE MECHANISMS DURING CREEP-FATIGUE IN
ALLOY 709, PART 1: TEMPERATURE AND HOLD TIME EFFECTS
ON SOLUTION ANNEALED MATERIAL

6.1 Introduction

Alloy 709 is a precipitation and solution strengthened 20Cr-25Ni-1.5Mo-Nb-N austenitic stainless steel that is a candidate alloy for structural components in sodium cooled fast spectrum nuclear reactors (FSRs), where creep-fatigue performance is critical. Thermal fatigue arises from power plant start-up and shut-down cycles. Steady state operation at elevated temperature results in time-dependent (i.e. creep) deformation. The synergistic effect of cyclic and creep deformation generally results in a greater accumulation of damage, and hence a shorter life, compared to low cycle fatigue (LCF) or constant stress creep alone. Austenitic stainless steels are primarily used for elevated temperature power plant structural applications due to their high creep strength, good corrosion resistance, and relative low cost compared to nickel-based superalloys. Creep-fatigue tests are simulated in a laboratory using strain-controlled cycling with a prescribed dwell time at a constant strain for each cycle. Hold periods at peak tensile strain are well-known to be the most damaging for austenitic alloys, so this condition is typically studied for design purposes. Results from accelerated laboratory testing (both creep and LCF), where temperatures and stresses or strains are typically greater than expected during service, are extrapolated to less severe service conditions. However, extrapolation of accelerated test data can result in unreliable life predictions if the deformation and damage mechanisms are not consistent across all conditions. Hence, understanding creep-fatigue failure across a range of test conditions and microstructures as a function of deformation and damage mechanisms is necessary for producing reliable life prediction models, and ultimately, component design.

Austenitic stainless steels are well-known for their high strength and ductility combination, which is attributed to high strain-hardening rates during deformation. High rates of work-hardening are due to a lack of dynamic recovery by cross-slip of screw dislocations. In FCC crystal structures, partial dislocations must constrict to a unit screw dislocation prior to cross-slip. Stacking fault energy (SFE), which is the energy associated with the distance between partial dislocations, generally dictates the relative ease for which dislocations can cross-slip.
Austenitic stainless steels typically have a low SFE, which restricts dislocation motion by cross-slip due to the relatively large distance between partials, resulting in deformation that is characterized as primarily planar. In high SFE materials, like aluminum alloys, constriction of partial dislocations requires less energy and cross-slip readily occurs. Deformation in these materials is characterized by wavy slip and strain-hardening rates are relatively low. Thus, the high strain-hardening behavior in austenitic steels is due to minimal dynamic recovery by cross-slip as a result of their low SFE.

However, several FCC metals with high SFE exhibit high work-hardening rates and planar deformation, including Mn austenitic stainless steels, nickel alloys, and others [6.1]–[6.4]. Solute atoms are sometimes attributed to the increase of slip planarity and the rate of strain-hardening in alloys where the SFE is relatively high. Kocks showed that the degree of strain-hardening and planar deformation in Ni, which typically deforms in a wavy manner as a pure metal, is enhanced with the addition of small amounts of C. The lack of significant cross-slip in this binary alloy cannot be attributed to a change in the SFE, which is negligible with the addition of the interstitial solutes [6.2]. Likewise, in Mn-containing austenitic steels which have a high SFE, deformation is primarily planar as a result of the high concentration of interstitial C [6.1]. Hong and Laird proposed that the mechanism for the transition from wavy to planar slip in FCC alloys (where SFE does not change significantly) is primarily due to the elastic interaction of the solutes and the edge components of the partial screw dislocations [6.3]. The strain energy of edge dislocations, which contain components of hydrostatic stress, is decreased by the presence of solute atoms around the core which form so-called Cottrell atmospheres. Solute atmospheres around dislocation cores increase the frictional stress for dislocation movement. Thus, besides the stacking fault width, an additional friction stress produced by solute atmospheres at the edge components of partial screw dislocations needs to be overcome in order for constriction of the partials and cross-slip to occur.

Dynamic strain aging (DSA) occurs in a wide variety of alloys in the range of temperatures and/or strain-rates where solute mobility is approximately equal to the dislocation velocity, which results in the dynamic formation of atmospheres around dislocations that are temporarily arrested (e.g. at precipitates, boundaries, dislocation forests, etc.). The repeated pinning and unpinning of dislocations in this manner typically manifests macroscopically as stress drops or serrations in the stress-strain curve, due to the formation of localized deformation.
bands [6.5]. DSA generally results in an increase in work-hardening and a decrease in ductility. The occurrence of DSA with the addition of N in austenitic steels, such as 304L(N) and 316L(N), has been shown to enhance the planarity of slip, leading to an increase in strength through high work-hardening rates [6.6]–[6.10]. In LCF and creep-fatigue, DSA typically results in prolonged cyclic hardening and a decrease in number of cycles to failure [6.5], [6.10]–[6.14], although some have reported an increase in LCF performance [6.15]. The mechanism for increased work-hardening in these alloys is debated in literature. Many have attributed enhanced planarity to short range ordering (SRO) in the matrix due to the high affinity of N and Cr. SRO leads to glide plane softening and heterogeneous deformation in dense slip bands [6.16]–[6.18]. Others attribute a decrease in SFE with the addition of N to the strong planar nature of the deformation [6.19], although the change in SFE has also been argued as being negligible [6.2], [6.3], [6.10]. In any case, there is a strong correlation between interstitial concentration, strain-hardening, and planar slip in intermediate temperatures and strain rates where DSA is observed.

In addition to solution strengthening, austenitic stainless steels used at elevated temperatures also frequently utilize dispersion strengthening through intragranular carbides, nitrides, intermetallic precipitates, and oxides which provide strong barriers to dislocation motion [6.20]. Intragranular precipitates can be formed during thermal-mechanical processing prior to service (e.g. cold rolling and isothermal aging), or they may nucleate and grow during service. Grades which are stabilized by Ti and/or Nb take advantage of strengthening from fine MX carbides (where M is Ti or Nb and X is C or N) and typically prevent $\text{M}_{23}\text{C}_6$ (where M is predominantly Cr) precipitation, which leads to a loss of corrosion resistance through depletion of Cr. Nano-scale MX carbo-nitrides, which precipitate dynamically on dislocations, contribute significantly to the work-hardening of austenitic steels at elevated temperatures during both constant load creep [6.21]–[6.24] and low cycle fatigue (LCF) [6.25]–[6.27].

This work originates from a previous study, which examined the LCF and creep-fatigue tests of Alloy 709 at the expected FSR service temperature of 550 °C and a higher accelerated test temperature of 650 °C [6.28]. It was shown that with strain rate, strain range, and tensile hold time being held constant at $10^{-3}$ s$^{-1}$, 1 pct, and 30 min, respectively, the creep-fatigue cyclic stress behavior and life reduction relative to the LCF life are significantly different between the two temperatures. Microstructural evolution by dynamic precipitation has a significant effect on the cyclic stress behavior (i.e. hardening and softening), deformation mechanisms, internal
damage, and creep-fatigue life when testing an initially solution annealed microstructure. A high initial rate of cyclic hardening at 650 °C is attributed to both DSA and dynamic precipitation of fine intragranular carbides. The onset of cyclic softening is attributed to the disappearance of serrated flow and precipitate coarsening, resulting in increased cross-slip and subgrain formation. Sustained DSA and a slower rate of precipitation at 550 °C result in prolonged cyclic hardening and high tensile stresses due to planar deformation and a lack of dynamic recovery. High tensile stresses due to a lack of recovery lead to significant intergranular cracking at 550 °C. Dynamic recovery and a decrease in the maximum tensile stress at 650 °C are responsible for less intragranular damage and better creep-fatigue performance compared to 550 °C. It was proposed that a change in slip behavior from planar to wavy at 650 °C is due to a reduction of the dynamic strain aging effect on slip planarity, which is supported by a disappearance of serrated flow and a reduction of dislocation density at the onset of cyclic softening. The disappearance of DSA, and the subsequent onset of recovery, was hypothesized to be due to a significant consumption of solutes in dynamically precipitating carbides and nitrides.

The present work seeks to provide better understanding of and support for the proposed mechanisms of slip mode transition from planar to wavy and the relation to dynamic strain aging, precipitation, damage, and creep-fatigue performance of Alloy 709. The temperature range for creep-fatigue testing is expanded to study the evolution of strengthening mechanisms (i.e. precipitation, DSA, and Taylor hardening) at temperatures below (500 °C), in-between (600 °C), and above (700 °C) previous test temperatures, where precipitation is expected to occur at different rates during testing. Also, the effect of hold time on microstructure evolution is evaluated with shorter hold times (5 and 15 min) at peak tensile strain compared to previous results with 30 min hold times. Mechanisms of both cyclic and time-dependent deformation are discussed with respect to microstructure evolution and damage mechanisms. An assessment is made on the applicability of accelerated creep-fatigue testing of Alloy 709 in relation to expected service conditions in nuclear structural applications.
6.2 Materials and Methods

6.2.1 Materials

Several different heats of solution annealed (SA) Alloy 709 have been used in this study with compositions and processing details given in Chapter 3. The as-received SA microstructure is discussed in Chapter 4.

6.2.2 LCF and Creep-Fatigue Testing

LCF and creep-fatigue testing and specimen details are given in Chapter 3. In this study, tests have been conducted at temperatures ranging from 500 to 700 °C and with tensile hold times, \( t_h \), ranging from 0 to 30 min, as shown in the experimental matrix summarized in Table 6.1. In all tests the total strain amplitude, \( \varepsilon_{at} \), was held constant at 1 pct.

6.2.3 Microstructure Characterization

Deformation structures in tested specimens were characterized from channeling contrast in backscatter electron (BSE) images using a field emission scanning electron microscope (FESEM). Precipitates have been characterized using bright field (BF) and dark field (DF) techniques with a transmission electron microscope (TEM). Internal damage was characterized with light optical micrographs (LOM). Experimental details of characterization techniques used in this study including light optical and electron microscopy, dislocation density analysis, and Vickers micro-hardness are discussed in Chapter 4.

6.2.4 Stress Partitioning, Strain Hardening, and Plastic Strain Analysis

The evolution of cyclic flow stress was investigated for all test conditions by partitioning the total stress amplitude, \( \sigma_a \), into the friction and back stress components, \( \sigma_f \) and \( \sigma_b \), respectively. The method for stress partitioning of a fully reversed hysteresis loop used in this investigation, originally proposed by Cottrell [6.29] and extended by Kuhlmann-Wilsdorf and Laird [6.30], is a widely recognized approach for understanding dislocation behavior in fatigued metals. Friction stress (or effective stress) is considered to be an isotropic and thermal component of flow stress that represents the stress to move a dislocation through the matrix in
the presence of obstacles, such as solute atoms, precipitates, and other dislocations. Based on the stress partitioning method, the friction stress is essentially the cyclic yield stress. Back stress (or internal stress) is the athermal component of flow stress associated with long range dislocation pile-ups. Back stress is associated with the increase in shear stress from strain-hardening. Thus, partitioning the total flow stress into the two components can provide an understanding of the mechanisms responsible for the evolution of stress and strain during cyclic deformation. The schematic hysteresis loop in Figure 6.1 demonstrates how the individual stress components are determined. Complete details of the stress partitioning method used in this study are given in Chapter 3.

Strain-hardening behavior in materials is indicative of the deformation behavior and the strengthening mechanisms. The strain-hardening rates were analyzed for the tensile half of each hysteresis loop. The strain-hardening analysis is detailed in Chapter 3.

The amplitude of plastic strain, $\varepsilon_{ap}$, for each creep-fatigue cycle is composed of two components: the plastic deformation during cycling (i.e. loading and unloading strain), $\varepsilon_{ap,cyc}$, and the time-dependent (i.e. creep) deformation, $\varepsilon_{cr}$, that occurs during the constant strain hold period as a result of the conversion of elastic strain into inelastic strain. It is important to distinguish between the different components of $\varepsilon_{ap}$ since each component contributes differently to the accumulation and propagation of damage. Complete details of the method used for

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Tensile Hold Time, $t_h$ (min)</th>
<th>Heat ID</th>
<th>Test End Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>700</td>
<td>30</td>
<td>58776</td>
<td>To Failure</td>
</tr>
<tr>
<td>650</td>
<td>0</td>
<td>011593, 011594</td>
<td>To Failure</td>
</tr>
<tr>
<td>650</td>
<td>5</td>
<td>58776</td>
<td>To Failure</td>
</tr>
<tr>
<td>650</td>
<td>15</td>
<td>58776</td>
<td>To Failure</td>
</tr>
<tr>
<td>650</td>
<td>30</td>
<td>011593, 011594</td>
<td>To Failure</td>
</tr>
<tr>
<td>650</td>
<td>30</td>
<td>011594</td>
<td>Interrupted at various intervals</td>
</tr>
<tr>
<td>600</td>
<td>30</td>
<td>58776</td>
<td>To Failure</td>
</tr>
<tr>
<td>550</td>
<td>0</td>
<td>011593, 011594</td>
<td>To Failure</td>
</tr>
<tr>
<td>550</td>
<td>5</td>
<td>58776</td>
<td>To Failure</td>
</tr>
<tr>
<td>550</td>
<td>15</td>
<td>58776</td>
<td>To Failure</td>
</tr>
<tr>
<td>550</td>
<td>30</td>
<td>011593, 011594</td>
<td>To Failure</td>
</tr>
<tr>
<td>550</td>
<td>30</td>
<td>011594</td>
<td>Interrupted at various intervals</td>
</tr>
<tr>
<td>500</td>
<td>30</td>
<td>58776</td>
<td>To Failure</td>
</tr>
</tbody>
</table>

Table 6.1 Creep-Fatigue Testing Matrix
partitioning the total plastic strain amplitude for each cycle into the two different components are given in Chapter 3.

![Figure 6.1 Schematic hysteresis loop illustrating the partitioning of maximum stress, σ_{max}, into friction and back stress components, σ_f and σ_b, respectively. Adapted from [6.30].](image)

6.3 Creep-Fatigue Cyclic Stress and Life Results

6.3.1 Temperature Effect

The peak tensile stress versus cycle number is shown in Figure 6.2 for all temperatures tested with a 30 min tensile hold time. Note that peak compressive stress is not shown for clarity, but the behavior is approximately the same in compression for all temperatures. Cycles to failure for all creep-fatigue tests are shown in Table 6.2. For all test temperatures greater than 500 °C, initial cyclic hardening is followed by softening after a maximum in peak tensile stress, which occurs at a smaller number of cycles for higher temperatures. At the lowest test temperature of 500 °C, cyclic hardening for approximately 500 cycles is followed by a relatively stable peak stress until failure after approximately 1800 cycles. The maximum tensile stress is nominally the same as 550 °C. At higher temperatures, the maximum stress decreases with increasing temperature.

The cyclic stress behavior at the highest test temperature of 700 °C is similar to that at 650 °C, where initial cyclic hardening is followed by cyclic softening until failure. At both 650 and 700 °C, cyclic softening occurs in two stages; a high initial rate of softening precedes a
slower more stable softening rate until failure. The onset of softening at 650 °C has been attributed to a change in slip mode from planar to wavy and to the formation of low energy dislocation structures through cross-slip and climb [6.28]. At 700 °C, where the thermal contribution to dislocation motion and obstacle bypass is greater, a lower maximum stress prior to cyclic softening is expected. At the intermediate temperature of 600 °C, cyclic softening begins at a cycle count and a maximum tensile stress between the 550 and 650 °C tests. The cyclic softening at 600 °C occurs at a rate greater than that at 550 °C, but it does not exhibit the two-stage softening observed at higher temperatures. Additionally, the number of cycles to failure at 600 °C is the shortest of any of the test temperatures, which suggests that creep-fatigue life is not a monotonic function of temperature for these test conditions.

### 6.3.2 Tensile Hold Time Effect

The effect of tensile hold time on creep-fatigue cyclic stress behavior and life is shown in Figure 6.3 for test temperatures of 550 and 650 °C and hold times of 0, 5, 15, and 30 min. At 550 °C, the introduction of even the shortest hold time of 5 min results in a maximum in cyclic stress followed by softening, though the rate of softening is slower at 5 min compared to 15 or 30 min. The maximum tensile stress for all creep-fatigue tests at 550 °C is approximately the same and is significantly greater than the maximum stress achieved during LCF (i.e. no hold). Additionally, the introduction of all hold times at 550 °C reduces the fatigue life significantly.
Figure 6.3 Peak tensile stress versus cycle number for creep-fatigue tests at (a) 550 and (b) 650 °C with $\varepsilon_{aT} = 1$ pct and tensile hold times of 0, 5, 15, and 30 min.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Hold Time, $t_h$ (min)</th>
<th>Cycles to Failure, $N_f$</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>30</td>
<td>1842</td>
</tr>
<tr>
<td>550</td>
<td>30</td>
<td>974*</td>
</tr>
<tr>
<td>600</td>
<td>30</td>
<td>307</td>
</tr>
<tr>
<td>650</td>
<td>30</td>
<td>1000*</td>
</tr>
<tr>
<td>700</td>
<td>30</td>
<td>648</td>
</tr>
<tr>
<td>550</td>
<td>5</td>
<td>1438</td>
</tr>
<tr>
<td>550</td>
<td>15</td>
<td>1074</td>
</tr>
<tr>
<td>650</td>
<td>5</td>
<td>491</td>
</tr>
<tr>
<td>650</td>
<td>15</td>
<td>398</td>
</tr>
</tbody>
</table>

* - Average of two tests is reported.
compared to the LCF life and the shortest life occurs with the longest hold time (30 min). At 650 °C, on the other hand, the shorter hold times result in fewer numbers of cycles to failure compared to the 30 min hold. Furthermore, in the 5 and 15 min hold time tests, the peak tensile stresses are consistently greater than in the 30 min hold time test. The maximum stress with a 5 min hold occurs after approximately twice the number of cycles compared to the 30 min hold and the two-stage softening that is observed with the 30 min hold does not occur in the shortest hold time test. Instead, a relatively high rate of cyclic softening occurs after the maximum in peak tensile stress is reached.

6.4 Hysteresis Loop Analysis Results

Quantitative analysis of hysteresis loops for all LCF and creep-fatigue tests provides further understanding of the differences in cyclic behavior and cycles to failure under various testing conditions. For each test, every hysteresis loop available has been partitioned into friction and back stress components (on the half loop following peak compression strain), and the components of plastic strain from loading/unloading (i.e. cyclic plastic strain) and from stress relaxation during the tensile hold (i.e. creep strain). Additionally, the tensile strain-hardening rate has been analyzed. Qualitatively, the presence of serrated flow (and the disappearance of serrations with increasing cycle count, if applicable) has also been extracted from the hysteresis loops.

6.4.1 Serrated Flow in Hysteresis Loops

Serrated flow is present in both the tensile and compressive halves of hysteresis loops, at least initially, for all conditions studied. Previous results showed that serrated flow is initially present during creep-fatigue testing at both 550 and 650 °C, and disappears after approximately the same number of cycles where the peak tensile stress reaches a maximum [6.28]. This result is consistent for the disappearance of serrations in tension among the range of temperatures tested in this study with a 30 min tensile hold. In many of the conditions, the disappearance of serrated flow in compression occurs at a greater number of cycles compared to tension. For shorter hold times, including LCF, serrated flow generally persists for a greater number of cycles compared to the 30 min hold time tests. A summary of observed serrated flow behavior is presented in Table 6.3, which includes a relative comparison of the critical strain to the onset of serrations in tension.
and compression, $\varepsilon_{c,tens}$ and $\varepsilon_{c,comp}$, and the approximate number of cycles where serrations completely disappear from the hysteresis loops in each half of the loop. DND (“Do Not Disappear”) is used to denote where serrations are sustained for the entire test.

<table>
<thead>
<tr>
<th>Temperature ($°C$)</th>
<th>Hold Time, $t_h$ (min)</th>
<th>$\varepsilon_{c,tens}$ vs. $\varepsilon_{c,comp}$</th>
<th>Cycles to Disappearance of Serrated Flow in Tension</th>
<th>Cycles to Disappearance of Serrated Flow in Compression</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>30</td>
<td>$\varepsilon_{c,tens} &gt; \varepsilon_{c,comp}$</td>
<td>400</td>
<td>1300</td>
</tr>
<tr>
<td>550</td>
<td>30</td>
<td>$\varepsilon_{c,tens} &gt; \varepsilon_{c,comp}$</td>
<td>350</td>
<td>400</td>
</tr>
<tr>
<td>600</td>
<td>30</td>
<td>$\varepsilon_{c,tens} &gt; \varepsilon_{c,comp}$</td>
<td>100</td>
<td>200</td>
</tr>
<tr>
<td>650</td>
<td>30</td>
<td>$\varepsilon_{c,tens} &gt; \varepsilon_{c,comp}$</td>
<td>50</td>
<td>100</td>
</tr>
<tr>
<td>700</td>
<td>30</td>
<td>$\varepsilon_{c,tens} \approx \varepsilon_{c,comp}$</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>550</td>
<td>0</td>
<td>$\varepsilon_{c,tens} \approx \varepsilon_{c,comp}$</td>
<td>DND</td>
<td>DND</td>
</tr>
<tr>
<td>550</td>
<td>5</td>
<td>$\varepsilon_{c,tens} &gt; \varepsilon_{c,comp}$</td>
<td>1100</td>
<td>DND</td>
</tr>
<tr>
<td>550</td>
<td>15</td>
<td>$\varepsilon_{c,tens} &gt; \varepsilon_{c,comp}$</td>
<td>500</td>
<td>800</td>
</tr>
<tr>
<td>650</td>
<td>0</td>
<td>$\varepsilon_{c,tens} \approx \varepsilon_{c,comp}$</td>
<td>DND</td>
<td>DND</td>
</tr>
<tr>
<td>650</td>
<td>5</td>
<td>$\varepsilon_{c,tens} &gt; \varepsilon_{c,comp}$</td>
<td>70</td>
<td>DND</td>
</tr>
<tr>
<td>650</td>
<td>15</td>
<td>$\varepsilon_{c,tens} &gt; \varepsilon_{c,comp}$</td>
<td>40</td>
<td>DND</td>
</tr>
</tbody>
</table>

DND – Does not disappear.

### 6.4.2 Friction and Back Stress

The friction and back stress components of the flow stress are shown in Figure 6.4 at all test temperatures with 30 min hold times. The evolution of the friction stress with cycle number follows closely the evolution of peak tensile stress (see Figure 6.2). The friction stress initially increases, followed by a decrease at all temperatures (except at 500 °C) where cyclic softening is observed. The back stress, on the other hand, is either relatively constant (650 and 700 °C) or generally increases throughout the test at lower temperatures. Therefore, it seems the evolution of friction stress is primarily responsible for the cyclic hardening and softening response. The relative magnitude of friction stress across the test temperatures is somewhat different than the peak tensile stress. For example, the friction stress at 700 °C is larger compared to 500 and 550 °C up to approximately 100 cycles, despite having a significantly lower peak tensile stress. Furthermore, the highest magnitude of friction stress occurs at 600 °C, which has a lower maximum tensile stress compared to 500 and 550 °C. Conversely, the magnitude of back stress is
generally inversely proportional to temperature (i.e. the highest temperature has the lowest back stress).

Figure 6.4 (a) Friction stress, $\sigma_f$, and (b) back stress, $\sigma_b$, for all temperatures tested in creep-fatigue with $\varepsilon_{AT} = 1$ pct and $t_h = 30$ min.

The friction and back stress components are shown for all hold times at 550 and 650 °C in Figure 6.5. The evolution of friction stress is similar for all creep-fatigue tests at 550 °C (i.e. with a hold time), except that the decrease in magnitude occurs at a greater number of cycles for the shorter hold time tests. The LCF friction stress at 550 °C is significantly lower than in the creep-fatigue tests and only increases in magnitude throughout the test, similar to the peak tensile stress behavior. The evolution of back stress is also similar for all hold time tests at 550 °C, with the 5 min hold having a slightly lower maximum than the longer hold times after approximately 300 cycles. The LCF back stress is relatively stable and significantly lower than all of the hold time tests at 550 °C. At 650 °C, the friction stress is greater in the short hold time tests compared to the 30 min hold for the entire test. For both the 5 and 15 min hold tests, the friction stress increases with cycle number to a maximum that is approximately the same as the LCF test. The friction stress begins to decrease with fewer cycles for longer hold times at 650 °C, similar to the trend at 550 °C. The evolution of back stress with cycle number, on the other hand, is not strongly affected by the duration of the hold time at 650 °C.
Figure 6.5 (a) and (c) friction stress, $\sigma_f$, and (b) and (d) back stress, $\sigma_b$, at 550 and 650 °C, respectively, for creep-fatigue tests with hold times of 0, 5, 15, and 30 min and $\varepsilon_{aT} = 1$ pct.
6.4.3 Strain-Hardening Behavior

The strain-hardening rates were evaluated for all of the conditions as a function of cycle number. Similar to the evolution of the back stress, the strain-hardening rates increase with cycle number for all temperatures to approximately the number of cycles where cyclic softening is observed. Figure 6.6 shows the mid-life tensile strain-hardening rates, along with the tensile stress amplitude versus strain amplitude for all temperatures tested with a 30 min hold. In general, the strain-hardening rates increase with decreasing temperature. However, the rate of strain-hardening at 550 °C is consistently greater than at 500 °C. It is worth mentioning that the strain-hardening rate at 600 °C is approximately the same as the lower temperatures up to 150 cycles (i.e. the mid-life cycle at 600 °C). However, the strain-hardening rates continue to increase at 500 and 550 °C beyond 150 cycles, which results in higher mid-life rates compared to 600 °C. An increase in long-range dislocation interactions is responsible for both high strain-hardening rates and high back stresses; note the similar trend in strain-hardening rate and back stress with temperature (see Figure 6.4(b)).

Figure 6.6 Mid-life strain-hardening rate and tensile stress amplitude versus strain amplitude for all temperatures tested in the SA condition with 30 min tensile holds and \( \varepsilon_{\alpha T} = 1 \) pct.
6.4.4 Plastic Strain and Stress Relaxation

The evolution of cyclic plastic strain amplitude, $\varepsilon_{ap,cyc}$, during a creep-fatigue test is shown in Figure 6.7 for all creep-fatigue test conditions. For all tests, $\varepsilon_{ap,cyc}$ decreases initially as the material work hardens and the hysteresis loops become narrower. A comparison of $\varepsilon_{ap,cyc}$ at different test temperatures tested with a 30 min tensile hold is shown in Figure 6.7 (a). For conditions where cyclic softening is observed in the test, $\varepsilon_{ap,cyc}$ reaches a minimum and then either plateaus or increases at approximately the same cycle count as the onset of softening. At 650 and 700 °C, $\varepsilon_{ap,cyc}$ increases after approximately the same number of cycles where friction stress reaches a maximum. The large $\varepsilon_{ap,cyc}$ after the short initial hardening stages at 650 and 700 °C is due to a relatively low friction stress and back stress, which correlate to a lower cyclic yield stress and more plasticity during fatigue. The cyclic plastic strain at 500 °C is larger than at 700 °C up to approximately 80 cycles, which is also where the friction stress at 500 °C surpasses that of 700 °C. $\varepsilon_{ap,cyc}$ is the smallest at 550 and 600 °C, corresponding to high values of friction stress and back stress. The amplitude of cyclic plastic strain at 500 °C is consistently larger than at 550 or 600 °C, indicating more plastic flow for a similar applied stress at the lowest test temperature, which is typical in the temperature range where DSA is active.

The effect of tensile hold time on cyclic plastic strain is shown in Figure 6.7 (b). At 650 °C, $\varepsilon_{ap,cyc}$ is the smallest with the 5 min hold, compared to the 15 and 30 min hold tests, which corresponds to higher friction stress values. Note that the cyclic plastic strain is only slightly larger in the 15 min hold test compared to the 5 min hold. At 550 °C, however, the shortest hold time (i.e. 5 min) results in the greatest amount of cyclic plastic strain, which indicates that cyclic plastic deformation becomes more difficult with increasing hold time. At 650 °C, the longer hold times result in more plastic deformation during cycling. Thus, a difference in microstructural evolution is likely responsible for the opposite trends in cyclic plastic strain at 550 and 650 °C with increasing hold time.

All test conditions in this study exhibit relaxation strain rates below $10^{-4}$ s$^{-1}$ during the tensile hold; therefore, the relaxation strain will be referred to as creep strain, $\varepsilon_{cr}$. The evolution of $\varepsilon_{cr}$ during tensile hold is shown in Figure 6.8 for all of the conditions tested in creep-fatigue. Figure 6.8 (a) shows that for a constant tensile hold time of 30 min, the magnitude of creep strain per cycle increases with temperature above 550 °C. $\varepsilon_{cr}$ is the lowest and approximately the same
at 500 and 550 °C, which indicates significant creep deformation does not occur until temperatures above 550 °C despite large initial tensile stresses at the beginning of the hold at these lower temperatures.

Figure 6.7 Cyclic plastic strain amplitude, $\varepsilon_{ap,cyc}$, as a function of cycle number for (a) all temperatures tested with a 30 min tensile hold and (b) all hold times tested at 550 and 650 °C. $\varepsilon_{aT} = 1$ pct for all tests.

Figure 6.8 Creep strain, $\varepsilon_{cr}$, as a function of cycle number for (a) all temperatures tested with a 30 min tensile hold and (b) all hold times tested at 550 and 650 °C. $\varepsilon_{aT} = 1$ pct for all tests.
The creep strain during different hold times is compared at 550 and 650 °C in Figure 6.8 (b). At 550 °C, the magnitude of creep strain is relatively unaffected by the hold time, which is due to the low creep strain rates at this temperature (e.g. $2.5 \times 10^{-7}$ to $2.0 \times 10^{-8}$ s$^{-1}$ during the 30 min hold at 550 °C). At 650 °C, where the relaxation strain rates are greater, especially at the beginning of the hold, shorter hold times generally result in smaller magnitudes of creep strain per cycle. This result is particularly interesting because it shows that less creep deformation occurs, and presumably less creep damage accumulates, during the shortest hold time tests which also have the shortest creep-fatigue lives.

6.5 Microstructural Characterization Results

6.5.1 Internal Damage and Deformation

In a previous study, it was found that the microstructures after LCF and creep-fatigue failure were significantly different at 550 and 650 °C for the same hold time, strain range, and strain rate [6.28]. At 550 °C, a high density of long sharp grain boundary cracks is attributed to rapid propagation of damage due to high tensile stresses at boundaries, which are not relieved by stress relaxation during the hold. At 650 °C, a recovered microstructure with well-defined subgrains near damaged boundaries lowers the strain energy for crack propagation. Additionally, the significant stress relaxation at 650 °C lowers the tensile stresses at grain boundaries, resulting in a lower density of cracks that are shorter and more blunted compared to 550 °C.

Channeling contrast in the BSE micrographs in Figure 6.9 shows representative deformation structures near damaged grain boundaries in the gage sections of specimens tested in creep-fatigue with 30 min holds at 500, 600, and 700 °C. At both 500 and 600 °C, deformation is primarily planar in all grains and adjacent to boundaries, as shown by the highlighted slip traces in Figure 6.9 (a) and (b). Creep-fatigue testing at 550 °C results in a similar deformation structure [6.28]. Additionally, the resulting grain boundary cracks at 500 and 600 °C are similar in length and acuity to those at 550 °C; i.e. sharp cracks that frequently span several grain diameters. At 500 °C, a small region of fine subgrains is shown directly ahead of the grain boundary crack, indicating a higher degree of dynamic recovery compared to 550 and 600 °C, where no subgrains are observed.

The microstructure after creep-fatigue testing at 700 °C is significantly different compared to temperatures of 600 °C and below. Figure 6.9 (c) shows well-defined subgrains near
grain boundaries, indicating significant recovery in the vicinity of boundaries. Additionally, the grain boundary damage at 700 °C is typically composed of individual voids or short blunted cracks, compared to the long sharp grain boundary cracks at 600 °C and below. The resulting microstructure at 700 °C is similar to 650 °C, although the density of both subgrains and grain boundary voids are generally greater at the higher temperature. The higher density of grain boundary voids at 700 °C is attributed to the larger magnitude of accumulated creep strain compared to 650 °C. The higher density of subgrains at 700 °C indicates more dislocation cross-slip, which correlates with a lower friction stress and strain-hardening rates compared to 650 °C.

The internal grain boundary damage was measured after failure at 500, 600, and 700 °C using the same technique described in the previous study [6.28]. The effective intergranular crack lengths, $L_{\text{eff}}$, for all temperatures tested to failure with a 30 min hold are summarized in Table 6.4. At 600 °C, where the creep-fatigue life is the shortest, the microstructure has the highest density of grain boundary cracks. Similarly, the longest creep-fatigue life at 500 °C results in the lowest density of intergranular cracks. Interestingly, the test at 700 °C has a significantly lower density of internal cracks compared to 550 °C, despite a shorter life. The smaller effective crack length at 700 °C reflects the slower propagation of grain boundary voids. Note that only cracks were measured in this study (i.e. voids were not assessed).

### 6.5.2 Fracture Mode

Fracture surfaces from the largest primary crack at test temperatures of 500, 600, and 700 °C with 30 min tensile holds are shown in Figure 6.10. Intergranular fracture is present at all test temperatures, which is indicated by the short arrows pointing to grain boundary triple points. Regions of flat transgranular fracture are observed at both the highest and the lowest test temperatures, as indicated by the broken lines that are approximately perpendicular to fatigue striations on the surface. In general, 700 °C has a higher degree of transgranular fracture than at 500 °C. The fracture mode at 650 °C is also mixed with regions of both transgranular and intergranular cracking at 650 °C. At 600 °C, where the number of cycles to failure was the lowest, the fracture mode is primarily intergranular. Similarly, the fracture mode at 550 °C is primarily intergranular. The fracture mode in the LCF tests at 550 and 650 °C are transgranular, with little evidence of intergranular cracking. The addition of a tensile hold time in LCF tests typically changes the fracture mode from transgranular to intergranular in austenitic steels, since
the hold time is typically accompanied by intergranular creep void formation [6.31]. However, at 650 and 700 °C, where the accumulated creep strain is the highest, greater amounts of transgranular fracture are observed. At 550 and 600 °C, where the creep strain is small for each cycle, the degree of transgranular fracture is small. Therefore, the accumulation of creep strain alone, cannot explain the fracture mode or the number of cycles to failure in creep-fatigue under the conditions tested in this study.

Figure 6.9 BSE micrographs of deformed microstructures from the gage sections of creep-fatigue tests at (a) 500 °C, (b) 600 °C, and (c) 700 °C. Deformation is primarily planar at 500 and 600 °C, highlighted by the arrows indicating slip band traces. A high density of well-defined subgrains indicates that the slip mode is primarily wavy near grain boundaries at 700 °C, shown in (c).
Table 6.4 Total Effective Intergranular Crack Length for Failed Creep-Fatigue Tests with 30 min Holds

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Effective Intergranular Crack Length, $L_{eff}$ (mm/mm$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>1.3 ± 0.5</td>
</tr>
<tr>
<td>550</td>
<td>2.9 ± 0.8</td>
</tr>
<tr>
<td>600</td>
<td>3.8 ± 0.4</td>
</tr>
<tr>
<td>650</td>
<td>1.5 ± 0.4</td>
</tr>
<tr>
<td>700</td>
<td>1.7 ± 0.6</td>
</tr>
</tbody>
</table>

Figure 6.10 Fracture surfaces observed in the SEM at (a) 500 °C, (b) 600 °C, and (c) 700 °C. Short arrows point to grain boundary triple points and broken lines are perpendicular to and span the length of striated regions.
6.5.3 Dislocation Density and Hardness

Dislocation densities in the gage sections of failed specimens have been estimated from peak broadening of XRD line profiles. Figure 6.11 shows the estimated dislocation densities and measured Vickers hardness values for each temperature tested in creep-fatigue with 30 min hold times. The test temperatures of 550 and 600 °C have the highest dislocation densities at failure. Above 600 °C, dislocation density decreases significantly with increasing temperature due to dynamic recovery and the formation of LEDs (i.e. subgrains) by cross-slip and/or climb. The dislocation density at 500 °C is slightly lower than at 550 and 600 °C, despite a similar room temperature hardness to the sample tested at 550 °C. Above 600 °C, the room temperature hardness trend follows the dislocation density values. The deviation between the trends in hardness and dislocation density below 600 °C indicates that additional strengthening from precipitation and/or solid solution are significant after testing at the lower temperatures. In other words, at the higher testing temperatures (600 to 700 °C), where the hardness trend correlates well with dislocation density, the most significant strengthening mechanism is Taylor hardening (i.e. dislocation-dislocation interactions). At the low test temperatures, where the hardness trend increases for a similar or even lower dislocation density, fine precipitates and/or significant solution strengthening contribute to the high hardness at room temperature.

Figure 6.11 Dislocation density (left axis) and Vickers hardness (right axis) after creep-fatigue testing at all temperatures with 30 min holds and SA starting microstructure.
6.5.4 Precipitation

Precipitates, which form intragranularly and on grain boundaries during testing, have been characterized using conventional TEM bright field (BF) and dark field (DF) techniques in creep-fatigue specimens after failure at all temperatures tested with 30 min holds and interrupted cycle intervals at 550 and 650 °C. Previously published results showed that intragranular precipitation of carbides and nitrides is significantly different at the end of creep-fatigue testing at 550 and 650 °C [6.28]. Specifically, cuboidal $M_{23}C_6$ carbides are more than twice the size at 650 °C (~50 nm) compared to 550 °C (~10-20 nm) for a similar number of cycles to failure and, correspondingly, time at temperature. Additionally, fine Nb(C,N) (<10 nm) particles are observed after testing at 650 °C, but not at 550 °C. The earlier disappearance of serrated flow and the onset of cyclic softening at 650 °C is attributed to more significant solute consumption by precipitate nucleation and growth compared to 550 °C.

Creep-fatigue tests with 30 min holds at 550 °C were interrupted after 50, 400, and 600 cycles, which correlate to the cyclic hardening, peak hardening, and cyclic softening portions of the fatigue life, respectively. At 650 °C, tests were interrupted after 25, 50, and 400 cycles, corresponding to the same parts of the fatigue life. At 550 °C, intragranular precipitates are not observed after 50 cycles. However, after 400 cycles, which corresponds with a maximum in tensile stress, intragranular $M_{23}C_6$ carbides (~10 nm) are present on dislocations. After 600 cycles at 550 °C, the intragranular $M_{23}C_6$ carbides are slightly coarser (~10-20 nm), as shown in the BF/DF pair in Figure 6.12. At 650 °C, intragranular $M_{23}C_6$ carbides are observed after every interrupted interval. The approximate sizes of $M_{23}C_6$ cuboids are <10 nm, ~30-40 nm, and ~50 nm after 25, 50, and 400 cycles, respectively. The BF/DF pair in Figure 6.13 shows the $M_{23}C_6$ carbides after 400 cycles at 650 °C at a similar magnification to the micrographs in Figure 6.12. Note the Moiré fringes in Figure 6.13 (a) indicating a lattice misfit between $M_{23}C_6$ and the matrix. Additionally, fine Nb(C,N) precipitates (<10 nm) are present after 400 cycles at 650 °C, which are highlighted in the BF micrograph in Figure 6.13 (a).

After creep-fatigue failure at 500 °C, fine intragranular $M_{23}C_6$ carbides (~10 nm) are present on planar dislocation structures, as shown in the DF TEM micrograph in Figure 6.14 (a). The carbides are finer and have a smaller volume fraction compared to those present after failure at 550 °C. Note that the test at 500 °C was at temperature for approximately twice the time (~1000 h) compared to the 550 °C tests (~500 h). At 600 °C, the $M_{23}C_6$ carbides present after
Figure 6.12 (a) BF and (b) DF TEM micrographs showing $\mathrm{M}_2\mathrm{C}_6$ carbides ~10-20 nm in size decorating planar dislocation structures after 600 cycles in creep-fatigue at 550 °C with 30 min tensile holds. DF imaging with the 400 reflection from the $\mathrm{M}_2\mathrm{C}_6$ carbides.

Figure 6.13 (a) BF and (b) DF TEM micrographs showing $\mathrm{M}_2\mathrm{C}_6$ carbides ~50 nm in size decorating dislocations after 400 cycles in creep-fatigue at 650 °C with 30 min tensile holds. Fine MX precipitates <10 nm in size are indicated in the BF image in (a). DF imaging with the $\overline{4}04$ reflection from the $\mathrm{M}_2\mathrm{C}_6$ carbides.
failure are coarser (~30-40 nm) compared to 550 °C, as shown in the DF TEM micrograph in
Figure 6.14 (b). The test duration at 600 °C was approximately 150 h. It should be noted that
dark field imaging was used to reveal the fine carbides without dense dislocation structures.
Figure 6.14 (c) shows a BF TEM micrograph of the microstructure after creep-fatigue failure at
700 °C. At 700 °C, intragranular M_{23}C_{6} carbides are significantly coarser (~100-250 nm) than
those at lower temperatures, despite a relatively short time at temperature (~325 h). Fine
Nb(C,N) (~10-20 nm) are also distributed uniformly on dislocations and in the matrix, similar to
those observed at 650 °C, although slightly coarser. MX precipitates are not observed at
temperatures below 650 °C. Dynamic precipitation of carbides and nitrides is a strong function
of temperature in Alloy 709 in the range of temperatures studied here. Therefore, the test
temperature dictates the rate at which solutes are consumed from solution when starting from the
same SA microstructure. The decrease of friction stress, disappearance of serrated flow, and the
onset of dynamic recovery correlate with the rate of dynamic precipitation, which occurs faster at
higher test temperatures.

6.6 Discussion

6.6.1 Strengthening Mechanisms

Solid solution strengthening, precipitate strengthening, and dislocation strengthening
(Taylor hardening) are all relevant mechanisms in Alloy 709 at elevated temperatures.
Depending on the test temperature and time at temperature, the relative magnitude of
strengthening from each mechanism is different. Furthermore, the evolution of one mechanism is
dependent on the other two; that is, there is a strong inter-dependence of the strengthening
mechanisms during elevated temperature testing.

Solid solution strengthening in Alloy 709 is expected to be the most significant early in
testing at elevated temperatures; i.e. when solutes are still in solution. A recent investigation of
the magnitude of strengthening from various solute species (W, Nb, Cu, C, and N) in a
23Cr25NiWCuCo austenitic alloy, Sanicro 25, has been conducted through first principle
calculations based on density functional theory (DFT) [6.32]. The results indicate that W
provides the most significant increase in flow strength and that interstitial elements, C and N,
provide only a small strength increase in strength, primarily due to the small concentration.
Using a Taylor factor of 3.06, Heczko et al. determined that the increase in resolved shear stress
from solid solution strengthening, $\tau_{ss}$, in Sanicro 25 is approximately 9 MPa [6.26]. Mo is responsible for the solid solution strengthening in Alloy 709 [6.33]. The maximum contribution to solution strengthening from Mo has been shown to be less than W in austenitic steels [6.34]. Therefore, an increase in shear stress in Alloy 709 from Mo is expected to be less than 10 MPa.

Figure 6.14 DF TEM micrographs (using the $\bar{4}04$ reflection from $\text{M}_{23}\text{C}_6$) showing $\text{M}_{23}\text{C}_6$ carbides on dislocations at testing temperatures of (a) 500 °C (~10 nm) and (b) 600 °C (~30-40 nm). (c) BF TEM image after testing at 700 °C shows both coarse $\text{M}_{23}\text{C}_6$ carbides and fine MX (~10-20 nm) intragranularly. All images are taken from gage sections in failed creep-fatigue tests with 30 min tensile holds.
Alloy 709 is also strengthened by fine intragranular precipitates (M$_2$3C$_6$, Nb(C,N), and Z-phase), which precipitate dynamically during creep-fatigue testing. Fine carbides and nitrides nucleate on dislocations within the grains, which is common for Nb and Ti containing austenitic steels [6.10], [6.25]–[6.27]. Significant cyclic strengthening at elevated temperatures in HT-UPS and Sanicro 25 is attributed to intragranular MX particles on the order of less than 10 nm. The increase in resolved shear stress, $\tau_{or}$, from hard particles may be estimated by the Orowan bowing mechanism

$$\tau_{or} = 0.84 \frac{G b}{\lambda_s - d}$$

where $G$ is the shear modulus, $\lambda_s$ is the particle spacing, and $d$ is particle diameter [6.35]. However, at temperatures above ~0.4$T_m$, dislocation climb becomes relevant in the bypass of particles. To estimate the increase in strength from fine particles when dislocation climb is significant, a threshold stress, $\tau_d$, has been introduced by Atrz et al. [6.35] which takes the form

$$\tau_d = \frac{G b}{2\lambda_s} \sqrt{1 - k^2}$$

where $k$ is an attraction parameter which varies from 0 (maximum attraction) to 1 (no attraction). Depending on the interfacial energy of the particle (e.g. misfit between particle and matrix), the segment of dislocation line that is climbing around the particle may have a lower energy at the interface and, thus, will feel an attractive force which will need to be overcome for the dislocation to complete the bypass. Only a modest amount of attraction ($k = 0.94$) is needed for the dislocation detachment to become the strength-controlling mechanism. The large positive size misfit between MX and austenite, which varies from ~22 pct [6.26] to ~28 pct [6.25], results in a significant attractive force. Heczko et al. [6.26] have calculated $\tau_d$ in Sanicro 25 for NbC particles ($d = 5$ nm) to be 20 MPa for minimum attraction ($k = 0.94$) and up to 50 MPa for maximum attraction ($k = 0$). The predicted equilibrium volume fraction of MX in Sanicro 25 is approximately twice that in Alloy 709 at 700 °C [6.36]. Thus, an estimated increase in resolved shear stress from MX at 700 °C in Alloy 709 is approximately 4.4 MPa ($k = 0.94$) and up to 13 MPa ($k = 0$), assuming $d = 10$ nm and half the volume fraction of Sanicro 25 (0.017 pct).
Although $\text{M}_2\text{C}_6$ is significantly coarser compared to MX, the volume fraction is greater than MX at all temperatures. As discussed in Chapter 5, volume fractions of $\text{M}_2\text{C}_6$ carbides were estimated from small angle neutron scattering (SANS) after creep-fatigue at 550 and 650 °C. At 550 and 650 °C, the volume fractions are approximately 0.25 pct and 0.80 pct, respectively. Assuming a precipitate diameter of 10 nm at 550 °C, the shear strength increase varies from approximately 18 MPa ($k = 0.94$) to 52 MPa ($k = 0$). Assuming a precipitate diameter of 50 nm at 650 °C, the shear strength increase varies from approximately 6.1 MPa ($k = 0.94$) to 18 MPa ($k = 0$). At 550 °C, the small amount of creep deformation indicates that dislocation climb is slow; therefore, the maximum interaction strength ($k = 0$) may be a reasonable estimate. Conversely, a smaller interaction strength is assumed at 650 °C, where climb is significant. The relatively wide spacing of the Moiré fringes in the $\text{M}_2\text{C}_6$ particles (see Figure 6.13 (a)) indicates a smaller misfit with the matrix compared to MX, which results in a smaller interaction strength.

The final, and most significant, contribution to the resolved shear stress in Alloy 709 is due to dislocation forest strengthening, or Taylor hardening. The increase in strength from dislocation-dislocation interactions, $\tau_{\text{Taylor}}$, is estimated from

$$\tau_{\text{Taylor}} = \alpha G b \sqrt{\rho}$$

where $\alpha$ is a constant between 0.2-0.4 for polycrystals and $\rho$ is the dislocation density. Heczko et al. [6.26] have measured the dislocation densities after LCF testing at 700 °C with strain amplitudes of 0.2 and 0.7 pct, to be $9 \times 10^{13}$ m$^{-2}$ and $4.5 \times 10^{14}$ m$^{-2}$, respectively, which results in strength increases of 60 and 130 MPa, respectively. Thus, dislocation forests provide the largest magnitude of strength increase compared to solid solution and precipitate strengthening for Sanicro 25 at 700 °C. In Alloy 709, dislocation density estimates result in $\tau_{\text{Taylor}}$ of approximately 55 ± 12 MPa at 550 °C, compared to 24 ± 4 MPa at 700 °C.

The estimates of strength increase from solutes, precipitates, and dislocation forests indicate that Taylor hardening is the dominant strengthening mechanism under the conditions studied here, followed by precipitates, and finally solution strengthening. However, the calculated back stress is lower than the friction stress in all cases, which is not expected if long-range dislocation strengthening dominates. Part of this discrepancy is due to the assumptions made in the partitioning method, namely, that the magnitude of back stress at peak strain acts to
aid deformation upon reversal. At elevated temperatures, where dislocations can easily rearrange and annihilate, this simple assumption that planar dislocation pile-ups completely aid dislocation motion in the opposite direction results in an over estimation of the friction stress. Also, the determination of yielding from a strain offset inherently results in an over estimation of the friction stress. Finally, Kuhlmann-Wilsorf and Laird argued that a large component of the friction stress determined by Cottrell’s method is equal to, and has the same physical cause, as the back stress [6.30]. All conditions in this study have a friction stress that is more than two times the back stress, which means that without the dislocation forest strengthening component (i.e. the back stress), the friction stress is still larger than the back stress. The relatively high values of friction stress, compared to back stress, indicates that the influence from solutes and precipitates on dislocation motion is larger than predicted by the simple estimates above.

6.6.2 Interaction and Evolution of Strengthening Mechanisms

A comparison of the relative values of friction stress and back stress, and their evolution, among the different conditions studied here provides an understanding of the interaction and evolution of strengthening mechanisms. The back stress is the smallest at the highest test temperatures for a given hold time. The low back stress correlates with recovered dislocation structures, low dislocation densities, and low strain-hardening rates. On the other hand, the friction stress is the greatest at the highest temperatures at low cycle counts, indicating a higher resistance to dislocation motion compared to the lower temperatures where the friction stress is initially low and increases steadily with cycle number. An increase in friction stress with temperature in austenitic stainless steels and nickel-based alloys is attributed to DSA [6.11], [6.37], [6.38]. Based on the stress partitioning method used in this study, intragranular precipitates that impede dislocation motion also contribute to the measured friction stress. Therefore, faster precipitation at the higher temperatures also contributes to the high friction stress early in the tests. However, the friction stress begins to decrease at lower cycle counts at higher temperatures. The decrease in friction stress, which corresponds to cyclic softening, is interpreted to be due to a decrease in both solution and precipitate strengthening as precipitates coarsen and consume solute atoms. At the intermediate temperatures, the friction stress continues to rise for a larger number of cycles due to slower precipitation and sustained DSA. At the lowest
temperature, where precipitation and solute diffusion is relatively slow, the friction stress increases at the slowest rate.

The evolution of friction and back stress at different hold times reinforces the temperature effect on the evolution of strengthening mechanisms. At 550 °C, the introduction of a tensile hold time increases both the friction and back stresses significantly compared to LCF without a hold. Although part of the increase in the friction stress may be due to precipitates, dynamic precipitation is relatively slow at 550 °C, indicating that the increase is primarily due to solute effects. The significant increase in back stress and strain-hardening rates with even a small hold time (e.g. 5 min) is attributed to an increase in dislocation-solute interactions (i.e. DSA). It is proposed that solute atmospheres form at lower test temperatures while dislocations are arrested during the tensile hold, resulting in high strain-hardening rates. At 650 °C, the introduction of a hold time has a different effect on friction stress. Dynamic precipitation occurs relatively rapidly at 650 °C, which is partly responsible for the rapid rise in friction stress in all conditions. As precipitates coarsen and consume solutes friction stress decreases. For the short hold times (0 and 5 min), the friction stress remains high for a larger number of cycles due to smaller amounts of precipitation (and solute consumption), simply because the time per cycle is shorter. Increasing the hold time at 650 °C results in lower friction stress values and an earlier decrease in terms of cycle count compared to the shorter hold times since precipitates form during the dwell period and consume solutes.

Hong and Laird [6.3] proposed a model for the increase in frictional force for partial dislocations to constrict and cross-slip, which is a linear function of the local concentration of solutes at the dislocation core. There is a maximum local concentration, and therefore frictional force, which occurs from DSA at a specific temperature and strain rate combination [6.39]. Andrews et al. [6.40] built upon this model and determined computationally the dependence of constriction energy of partial dislocations on solute concentration, misfit strains (size and modulus), dislocation line tension, and stacking fault energy. The results of the simulations show that the misfit strain dominates the strengthening contributions and that constriction energy increases with solute concentration in all cases. These models suggest that a critical solute concentration exists where the applied stress is insufficient to overcome the friction force, resulting in a transition from wavy to planar deformation.
Dynamic strain aging indicates a high driving force for solute atmosphere formation around dislocations. Serrated flow is initially present in hysteresis loops for all conditions studied here. However, serrations disappear at some number of cycles for most conditions (see Table 6.3). Specifically, the number of cycles and time required for the disappearance of serrations is inversely related to test temperature. The relationship between test time and the disappearance of serrated flow is reinforced with the short hold time tests; serrations disappear at a greater number of cycles for shorter hold times. The rate of dynamic precipitation is also strongly related to test temperature and time at temperature; precipitate nucleation and growth occurs faster at the higher test temperatures in this study. Thus, a correlation exists between the time to precipitate a significant volume fraction of carbides and nitrides and the cycle count where serrated yielding disappears.

The reduction of DSA, which is interpreted to be a result of the decrease in interstitial solute content, also corresponds to a transition in the deformation behavior. For example, the microstructure in the creep-fatigue test at 650 °C with a 30 min hold is significantly different at 50 cycles compared to 400 cycles, where serrated flow is not present and the friction stress is lower. The microstructure has well-defined subgrains after 400 cycles, compared to 50 cycles, where deformation is primarily planar, as shown in Figure 6.15. The dislocation density after 400 cycles is also lower compared to 50 cycles [6.28]. Additionally, the volume fraction of carbides is approximately three times greater after 400 cycles compared to 50 cycles. Since cross-slip of screw dislocations is considered the principal dislocation mechanism for dynamic recovery in FCC crystals [6.41], it is interpreted that the solute atoms responsible for impeding cross-slip early on in the test are reduced to below a critical concentration at 650 °C between 50 and 400 cycles, at which point cross-slip becomes an active deformation mechanism. This critical solute concentration for the transition in slip behavior from planar to wavy is achieved earlier at higher temperatures (e.g. after 15 cycles at 700 °C). Similarly, the transition in slip mode occurs later for shorter hold times at 650 °C. As the solutes are consumed and the DSA effects are reduced, the energy for partial dislocations to constrict and cross-slip can be overcome by the applied stress, resulting in wavy slip and subgrain formation. At 550 and 600 °C, however, a disappearance of DSA and a decrease in friction stress do not correlate with significant recovery. The lack of dynamic recovery at these temperatures indicates that although
the solute concentration decreases during the test, the critical concentration for partial dislocation constriction and subsequent cross-slip is not achieved.

![Micrographs](image)

Figure 6.15 BSE micrographs of deformed microstructures from the gage sections of creep-fatigue tests at 650 °C interrupted at (a) 50 cycles and (b) 400 cycles. Deformation is primarily planar after 50 cycles, highlighted by the arrows indicating slip band traces. After 400 cycles, subgrain formation is observed adjacent to grain boundaries and ahead of crack tips (shown in (b)).

In Alloy 709, the evolution and interaction of solute and precipitate strengthening mechanisms during elevated temperature testing (and service) results in a transition in slip behavior, a decrease in work-hardening rate and dislocation density, and ultimately, a decrease in strength from dislocation forests (i.e. Taylor hardening). A decrease in friction stress as interstitial solutes are consumed in carbides and nitrides leads to lower work-hardening rates due to dynamic recovery. Therefore, the most significant impact of dynamic precipitation of carbides and nitrides during LCF and creep-fatigue tests is due to the increase of dislocation cross-slip and the resulting low work-hardening rate and decreased dislocation density.

### 6.6.3 Deformation Behavior and Damage Accumulation

When a tensile hold time is added in LCF testing of austenitic steels, the fracture mode typically changes from transgranular to some degree of intergranular cracking [6.31]. The increased fraction of intergranular fracture is attributed to grain boundary cavitation as a result of creep deformation during the hold. Thus, propagation of surface initiated transgranular fatigue cracks can be assisted by creep void formation. Alternatively, cyclic loading can propagate and
link together creep damage nucleated at grain boundaries, resulting in long grain boundary cracks. The latter scenario is potentially the most damaging and results in the greatest reduction in creep-fatigue performance due to the brittle nature of the fracture. The mechanisms for damage formation and propagation in Alloy 709 are discussed here with respect to plastic deformation, both cyclic and time-dependent (i.e. creep strain).

Failure in creep-fatigue is related to both inelastic creep strain and plastic strain during cycling. For the following comparison, the components of inelastic strain are averaged over the entire test by the number of cycles; specifically, the average cyclic plastic strain amplitude, $\bar{\varepsilon}_{ap,cyc}$, and the average creep strain, $\bar{\varepsilon}_{cr}$, per cycle are provided. Figure 6.16 shows the number of cycles to failure, $N_f$, $\bar{\varepsilon}_{ap,cyc}$, and $\bar{\varepsilon}_{cr}$ as a function of test temperature for creep-fatigue tests performed with a 30 min tensile hold. The cyclic plastic strain amplitude decreases somewhat from 500 °C to a minimum at 550 °C, after which it increases again for higher temperatures. Increased cyclic plastic strain is a result of relatively low strain-hardening rates and is associated with wavy slip due to dislocation cross-slip. Significant subgrain formation, which indicates cross-slip, is observed in failed specimens tested at 650 and 700 °C. High strain-hardening rates during cycling, as a result of the high friction stress from DSA, lead to low cyclic plasticity at 550 and 600 °C. At 500 °C, the solute concentration is expected to be the highest, due to the relatively slow precipitation. However, the slightly larger amount of cyclic plastic strain compared to 550 and 600 °C indicates that the effects of DSA are not as strong at the lower temperature, as indicated by the lower average friction stress.

The creep strain, on the other hand, is smallest at low temperatures and increases significantly at temperatures greater than 550 °C as a result of increasing stress relaxation during the hold periods. Since dislocation climb (which is the mechanism attributed to creep deformation at the stresses, strain rates, and temperatures in this study) is a thermally activated process, the increase in creep strain with temperature is expected.

Figure 6.16 is partitioned into four different temperature regimes, each with different relative contributions of deformation. In region 1, where creep deformation is at a minimum and the cyclic deformation is greater than in region 2, the number of cycles to failure is the greatest. Small creep strain results in less grain boundary cavitation. The deformation is primarily planar, although some recovery is observed directly ahead of grain boundary cracks, indicating some amount of cross-slip at low temperatures. The fracture surface in region 1 has components of
both transgranular and intergranular fracture (see Figure 6.10 (a)). In region 2, where creep deformation begins to increase and cyclic plasticity is at a minimum, the number of cycles to failure is also at a minimum. A greater amount of creep cavitation is expected in this region, compared to lower temperatures, as the average creep strain per cycle increases with temperature. The lack of plasticity during cycling, which is attributed to difficulty of cross-slip, results in high work-hardening rates and tensile stresses. Thus, creep cavitation damage propagates rapidly in the presence of high tensile stresses, resulting in long sharp grain boundary cracks and a fracture mode that is primarily intergranular (see Figure 6.10 (b)). An increase in cycles to failure occurs in region 3. In this region, cyclic plasticity increases significantly due to a reduction of friction stress from region 2, leading to an increase in dislocation cross-slip and subgrain formation. Creep deformation also increases in region 3. However, the onset of wavy slip and higher degree of recovery in this region acts to lower the elastic strain energy, and the tensile stresses, around damaged boundaries. Increasing plasticity around grain boundary cavitation retards propagation of intergranular damage, resulting in blunted grain boundary cracks and a mixed mode fracture. The cycles to failure decreases again in region 4 where creep deformation and cyclic plasticity are at a maximum. A highly recovered microstructure exists in this region due to the relative ease of cross-slip. However, significant creep cavitation decreases
the cyclic life as transgranular fatigue cracks are linked by a relatively high fraction of creep voids (see Figure 6.10 (c)).

The results from the short hold time tests provide support for the relative influence of cyclic and creep deformation on creep-fatigue life. The 5 min hold time test at 550 °C, where the creep strain per cycle is approximately the same as the longer hold time tests, results in a higher number of cycles to failure compared to longer hold times due to slightly more cyclic plasticity (see Figure 6.7 (b)). More cyclic plasticity with the shortest time at 550 °C may indicate a hold time effect on solute atmosphere formation and friction stress at relatively low temperatures. That is, the atmosphere formed during the short hold times may not be as dense compared to longer holds, leading to a lower friction stress that approaches that of the LCF test (i.e. no hold time available for atmosphere formation). At 650 °C, the 5 and 15 min hold time tests have significantly shorter lives compared to the 30 min hold time tests. The 5 min hold time has the smallest amount of creep strain at 650 °C, but also the smallest cyclic plastic strain (see Figure 6.7 (b)). The small cyclic plastic strain during the 5 min hold time test is attributed to higher interstitial concentration as a result of slower precipitation (in terms of number of cycles) compared to the longer hold times. Although the cyclic plastic strain is slightly larger in the 15 min hold test, compared to 5 min, the creep strain is significantly larger during the 15 min hold, which results in the shortest creep-fatigue life at 650 °C. Hence, the creep damage, which is a result of creep strain, is propagated more rapidly in the shortest hold time tests at 650 °C as a result of low cyclic plastic strain and high friction stress. Note that the maximum friction stress in LCF at 650 °C is similar to that of the 5 and 15 min hold tests, but the lack of creep strain in LCF results in the highest number of cycles to failure.

From these results, the creep-fatigue life of Alloy 709 can be maximized if (1) the creep strain is minimized and (2) a moderate amount of plasticity occurs during cycling to retard propagation of grain boundary cavitation damage. Cyclic plasticity is a strong function of microstructure (i.e. solute content and precipitation), which evolves differently under different conditions. Therefore, evolution of the microstructure should be a consideration in the extrapolation of accelerated testing performance to long-term service conditions.
6.7 Conclusions

1. Microstructural evolution of Alloy 709, by dynamic precipitation of carbides and nitrides (M$_{23}$C$_6$ and Nb(C,N)), during creep-fatigue testing of a solution annealed microstructure is a strong function of temperature. Solute atoms responsible for DSA promote planar deformation and high strain-hardening rates due to the friction stress imposed on dissociated partial dislocations, thus resisting cross-slip and recovery. A decrease in solute content, as a result of dynamic precipitation reduces the friction stress and leads to a transition in slip character from planar to wavy.

2. Enhanced cyclic deformation by dislocation cross-slip results in the formation of low energy dislocation structures (i.e. subgrains) adjacent to grain boundaries. Subgrain formation lowers the strain energy at grain boundaries and the driving force for propagation of intergranular damage (i.e. voids and cracks). Where wavy slip occurs, grain boundary damage is in the form of voids and short blunted cracks. The resulting fracture is mixed transgranular and intergranular. Conversely, where planar deformation dominates, long sharp grain boundary cracks form and the resulting fracture is intergranular.

3. The degree of creep damage, which occurs during the tensile hold period, is also a strong function of temperature as more stress relaxation occurs at higher temperatures.

4. It is proposed that the strain-controlled creep-fatigue life of Alloy 709 can be maximized if the creep deformation is kept at a minimum and the cyclic deformation is enhanced through dislocation cross-slip, thus reducing the formation and the propagation of intergranular damage.

6.8 References


2019.


CHAPTER 7
DEFORMATION AND DAMAGE MECHANISMS DURING CREEP-FATIGUE IN ALLOY 709, PART 2: EFFECT OF INITIAL MICROSTRUCTURE

7.1 Introduction

In part 1 of this study (Chapter 6) it was shown that the deformation behavior of Alloy 709 in creep-fatigue changes as a function of test temperature and time at temperature, as a result of microstructure evolution. Specifically, the slip character transitions from planar to wavy during testing from a solution annealed (SA) condition as carbides and nitrides dynamically precipitate and coarsen, drawing solute out of solution. A decrease in solute concentration corresponds to a reduction of dynamic strain aging (DSA), which is typically associated with enhanced slip planarity. The transition in slip character is attributed to significant dislocation cross-slip and subsequent recovery of the microstructure, leading to lower tensile stresses and more plastic deformation ahead of grain boundary cracks. In the conditions where wavy slip occurs, well-defined subgrains are formed adjacent to grain boundaries and the resulting intergranular damage consists of short blunted cracks. In the conditions where deformation is primarily planar, slip band impingement and high tensile stresses at grain boundaries propagate intergranular damage, resulting in long sharp grain boundary cracks. The transition to wavy slip and dynamic recovery, which is believed to slow the propagation of internal damage, occurs faster at higher temperatures due to a higher rate of solute consumption through faster precipitation. However, the formation of creep damage (i.e. intergranular cavitation), which occurs during the tensile hold time in creep-fatigue, also occurs faster at higher temperatures. Hence, it was proposed that creep-fatigue performance could be optimized if both the nucleation and propagation of grain boundary creep damage is minimized by promoting dynamic recovery during cycling, while keeping the creep deformation during the hold at a minimum.

The objective of the present study is to understand the effect of initial microstructure on creep-fatigue performance. Specifically, a microstructure that is aged to produce a stable precipitate population (i.e. near equilibrium) is tested under the same conditions as the solution annealed material at 550 and 650 °C to investigate the effect of interstitial solute concentration and precipitate population on DSA, slip character, damage propagation, and creep-fatigue life.
Mechanisms of both cyclic and time-dependent deformation are discussed with respect to microstructure evolution and damage mechanisms.

7.2 Materials and Methods

The materials, testing procedure, microstructural characterization techniques, and fatigue data analysis used in this study were discussed in detail in Chapter 6. Material from heat 58776 was used in this work. The creep-fatigue testing conditions with the aged specimens are identical to those described in an earlier study with the SA microstructure (550 and 650 °C; tensile hold time, $t_h=30$ min; total strain amplitude, $\varepsilon_{aT} = 1$ pct; strain rate, $\dot{\varepsilon} = 10^{-3}$ s$^{-1}$) [7.1].

7.2.1 Isothermal Static Aging

The thermodynamic and kinetic precipitate simulations to determine the aging condition predicted to produce a significant volume fraction of carbides and nitrides, $M_{23}C_6$ and Z-phase (CrNbN), were discussed in Chapter 5. Based on the simulations, the isothermal aging condition chosen for this study was 100 h at 750 °C. This aging condition is expected to produce approximately 90 pct of the equilibrium volume fraction of $M_{23}C_6$ and approximately 50 pct of the equilibrium volume fraction of Z-phase.

Prior to aging, specimens machined from a solution annealed plate were vacuum encapsulated in quartz tubes containing Ti slugs to prevent oxidation of the specimens. Following aging, specimen gage sections were re-polished along the axial direction to the same original finish produced by the machining vendor.

7.2.2 Determination of Critical Strain to the Onset of Serrated Flow, $\varepsilon_c$

It has been shown that the critical plastic strain, $\varepsilon_c$, at the onset of serrated flow in monotonic tensile tests is related to the temperature, strain-rate, activation energy of the diffusing species, and the concentration of the solutes that are responsible for the strain aging effect [7.2]–[7.4]. $\varepsilon_c$ is compared for the different initial microstructures tested under the same conditions of temperature and strain rate to determine the relative solute concentration causing the serrated flow in each condition. In monotonic deformation, $\varepsilon_c$ is simply defined as the plastic strain where the first load drop occurs. In this study, $\varepsilon_c$ is the amplitude of plastic strain where the first significant load drop (greater than 0.5 MPa) occurs in the tensile half of the hysteresis loop. The
cyclic plastic strain amplitude, $\varepsilon_{ap, cyc}$, is determined first, according to the procedure described in Chapter 6. The region of serrated flow from the first load drop up to peak tensile strain is then subtracted from $\varepsilon_{ap, cyc}$ to determine $\varepsilon_c$.

### 7.3 Aged Microstructure Results

Precipitates in the aged microstructure (100 h at 750 °C) are shown in the SEM and bright field (BF) TEM micrographs in Figure 3.5. A distribution of coarse $M_{23}C_6$ is found on grain boundaries, twin boundaries, and intragranularly. Inside the grains, cuboidal $M_{23}C_6$ precipitates are typically observed linked together in continuous chains, forming the needle shapes shown in Figure 3.5 (a). This morphology of intragranular $M_{23}C_6$ is common in austenitic steels [7.5]. These chains of cuboidal $M_{23}C_6$ often nucleate and grow from pre-existing Nb(C,N) particles. The chains of intragranular $M_{23}C_6$ carbides are approximately 150 nm wide and can be several microns in length. Figure 3.5 (a) also shows a grain boundary decorated with coarse globular $M_{23}C_6$ particles. After aging, dislocations are decorated with fine Z-phase precipitates, shown in Figure 3.5 (b). Z-phase precipitates are shown at higher magnification in Figure 3.5 (c) with an inset selected area diffraction pattern (SADP) confirming the tetragonal crystal structure and the orientation relationship ($(001)_Z|| (001)_\gamma$, $[1\bar{1}0]_Z||[100]_\gamma$) described by Robinson and Jack [7.6]. The fine Z-phase precipitates are rod shaped approximately 10 nm in diameter and 20 nm long. It has been shown that for aging times longer than 200 h at 750 °C, an increase in the amount of Z-phase occurs at the expense of the residual (i.e. non-equilibrium) MX particles [7.7], [7.8]. It should be noted that the spherical carbo-nitrides existing prior to aging did not shrink a noticeable amount in this relatively short thermal treatment.

### 7.4 Creep-Fatigue Cyclic Stress and Life Results

Peak tensile stress versus cycle number for the creep-fatigue tests at 550 and 650 °C with aged specimens are shown in Figure 7.2, compared to the results from the SA specimens tested under the same conditions. The cyclic stress behavior, maximum stress, and number of cycles to failure is significantly different between the two initial microstructures for each test temperature. At 550 °C, the aged microstructure hardens to a maximum tensile stress approximately 25 pct lower than the SA microstructure. The maximum stress is achieved after only 50 cycles, whereas the maximum stress is not reached until 400 cycles in the SA condition. Similarly, at 650 °C, the
Figure 7.1 (a) SEM and (b), (c) TEM BF micrographs of the microstructure of Alloy 709 after aging at 750 °C for 100 h. M23C6 carbides are shown aligned intragranularly (needle shape) and on a grain boundary in (a). M23C6 carbides are shown in higher magnification in (b), along with fine Z-phase precipitates decorating dislocations. Higher magnification of the Z-phase precipitates with an inset selected area diffraction pattern shown in (c).
maximum stress in the aged microstructure is approximately 23 pct lower than in the SA condition and is achieved after only 10 cycles, compared to 50 cycles with an SA microstructure. Unlike the creep-fatigue testing of the SA material, the cyclic stress response of the aged microstructures after the initial cyclic hardening stage at both test temperatures is relatively stable (i.e. no significant hardening or softening) until failure. The stable cyclic stress response in the aged condition indicates that microstructural hardening is balanced with softening for each cycle. The most significant difference in creep-fatigue performance between the two microstructures is the increase in number of cycles to failure, $N_f$, in the aged conditions. At 550 °C, the number of cycles to failure in the aged condition (3337 cycles) is more than three times greater than the SA condition (average of 974 cycles from two tests). The number of cycles to failure at 650 °C is also increased substantially from an average of 1000 cycles in the SA condition to 1438 cycles in the aged condition. The number of cycles to failure for each microstructure at both test temperatures is summarized in Table 7.1.

Figure 7.2 Peak tensile stress versus cycle number for creep-fatigue tests with $\varepsilon_{aT} = 1$ pct at (a) 550 and (b) 650 °C with specimens that were tested in the solution annealed condition and those that were aged at 750 °C for 100 h.
7.5 Hysteresis Loop Analysis Results

7.5.1 Friction and Back Stress

A remarkable difference in the evolution of both friction and back stress during creep-fatigue tests between the aged and SA microstructures is shown in Figure 7.3 at both 550 and 650 °C with the same 30 min tensile hold. With the aged microstructure, the friction stress at both temperatures is stable and approximately the same value, and is significantly lower than for the SA condition at either temperature. The back stress is also lower for the aged condition at both temperatures, although a greater magnitude is observed at 550 °C for both microstructures. The stable friction stress in the aged condition indicates that the significantly higher values of friction stress in the SA condition (and their evolution) are attributed to the evolution of the microstructure during testing.

7.5.2 Strain-Hardening Behavior

A comparison of the tensile strain-hardening rates at the mid-life cycle between the different microstructural conditions is shown in Figure 7.4. Pre-aging the microstructure decreases the strain-hardening rates significantly at 550 °C. At 650 °C, however, the strain-hardening rates are approximately the same at the mid-life cycle for both the SA and the aged microstructures. It should be noted that significant recovery (i.e. subgrain formation) was observed prior to the mid-life cycle in the SA condition at 650 °C, as discussed in Chapter 6. The similar strain-hardening behavior between the two microstructures at 650 °C indicates that the size and distribution of precipitates does not significantly change the deformation mechanism responsible for strain-hardening.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Hold Time (min)</th>
<th>Initial Microstructure</th>
<th>Cycles to Failure, $N_f$</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>30</td>
<td>SA</td>
<td>974*</td>
</tr>
<tr>
<td>650</td>
<td>30</td>
<td>SA</td>
<td>1000*</td>
</tr>
<tr>
<td>550</td>
<td>30</td>
<td>Aged 750 °C, 100 h</td>
<td>3337</td>
</tr>
<tr>
<td>650</td>
<td>30</td>
<td>Aged 750 °C, 100 h</td>
<td>1438</td>
</tr>
</tbody>
</table>

* - Average of two tests is reported.
Figure 7.3 (a) Friction stress, $\sigma_f$, and (b) back stress, $\sigma_b$, for both microstructures tested at 550 and 650 °C.

Figure 7.4 Mid-life strain-hardening rate and tensile stress amplitude versus strain amplitude at 550 and 650 °C with the aged and SA microstructures.
7.5.3 Plastic Strain and Stress Relaxation

The evolution of cyclic plastic strain amplitude, $\varepsilon_{ap,cyc}$, and creep strain, $\varepsilon_{cr}$, during creep-fatigue tests at 550 and 650 °C is shown in Figure 7.5 for both the aged and the SA conditions. At both 550 and 650 °C, the stable aged microstructure has significantly more cyclic plastic strain per cycle compared to the SA microstructure. The lower friction stress, back stress, and strain-hardening rates in the aged condition correlate with more plastic deformation during cycling compared to the SA condition. Although the friction stress is approximately the same for each temperature for the aged condition, the higher back stress and higher strain-hardening rates at 550 °C are responsible for the slightly lower cyclic plastic strain compared to 650 °C.

![Figure 7.5 (a) Cyclic plastic strain, $\varepsilon_{ap,cyc}$, and (b) creep strain, $\varepsilon_{cr}$, for both microstructures tested at 550 and 650 °C.](image)

During the tensile hold period, stress relaxes as elastic strain is converted to inelastic strain, which is characterized as creep strain below a certain relaxation rate [7.9], [7.10]. Despite the significant increase in cyclic deformation in the aged condition, the amount of creep strain per cycle is relatively unchanged at both test temperatures by aging the microstructure prior to testing, as shown in Figure 7.5 (b). At both temperatures, the inelastic strain rates during the hold period are also approximately the same for both microstructures. The accumulated creep strain ($N_{f,cr}$) throughout the test in the aged microstructure is significantly greater than in the SA...
condition, which is summarized in Table 7.2. Since the amount of creep strain per cycle is approximately the same in both microstructures, the larger accumulated creep strain in the aged condition is primarily due to the increase in cycle count at both temperatures.

It should be noted that, since the aged condition tests begin the hold time at lower tensile stresses at both temperatures (~25 pct lower at 550 °C and ~23 pct lower at 650 °C), the relative magnitude of stress relaxation is greater in the aged condition. In other words, the same magnitude of creep strain at a lower initial stress indicates a lower creep resistance in the aged condition compared to the SA microstructure.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Initial Microstructure</th>
<th>( N_f \varepsilon_{cr} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>SA</td>
<td>0.163</td>
</tr>
<tr>
<td>650</td>
<td>SA</td>
<td>0.745</td>
</tr>
<tr>
<td>550</td>
<td>Aged 750 °C, 100 h</td>
<td>0.677</td>
</tr>
<tr>
<td>650</td>
<td>Aged 750 °C, 100 h</td>
<td>1.049</td>
</tr>
</tbody>
</table>

### 7.5.4 Serrated Flow in Hysteresis Loops

Serrated flow is initially present in the hysteresis loops at both test temperatures with the aged microstructure, as was the case for the SA condition. The serrated flow also disappears with an increase in cycle count with both microstructures. However, the number of cycles before the serrations disappear from the hysteresis loops is significantly different between the two microstructures. At 550 °C, serrations in the tensile half of the cycle disappear after approximately 50 cycles in the aged condition, compared to 350 cycles in the SA condition. At 650 °C, serrated flow disappears from the tensile half of the loop after approximately 700 cycles in the aged condition, compared to 50 cycles in the SA condition.

The significant difference in the presence of serrated flow between the two microstructures is better understood through a comparison of the critical plastic strain to the onset of serrations, \( \varepsilon_c \), which is summarized in Table 7.3. In both microstructures, \( \varepsilon_c \) increases slowly with cycle number; the increase is most significant at 650 °C in the SA condition. At the same time, the cyclic plastic strain decreases as the material hardens initially and the hysteresis loops become narrower, as shown in Figure 7.5 (a), particularly in the SA condition tested at 550 °C. Once the critical strain for serrated flow (which increases) becomes equal to the cyclic plastic
strain (which decreases), serrations are no longer observed. At both temperatures, $\varepsilon_c$ is larger in the aged microstructure compared to the SA condition for a given cycle number. For example, $\varepsilon_c$ is shown in the fifth cycle hysteresis loops for both microstructures and test temperatures in Figure 7.6. In the SA microstructure, $\varepsilon_c$ increases slowly at 550 °C (~0.001 in 350 cycles) compared to 650 °C (~0.0036 in 50 cycles). In the aged condition, the increase in $\varepsilon_c$ is initially faster compared to the SA condition (in the first 10 cycles or so), after which the increase is slower until the serrations disappear. The reason for the larger number of cycles where serrations are observed in the aged condition at 650 °C is due to the larger cyclic plastic strain, or the width of the hysteresis loops) compared to the SA condition. At 550 °C, serrated flow is observed over a greater number of cycles in the SA condition due to smaller values and a slower increase in $\varepsilon_c$ compared to the aged condition.

Table 7.3 Summary of Critical Strain, $\varepsilon_c$, for Both Microstructures and Test Temperatures

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Initial Microstructure</th>
<th>Range of $\varepsilon_c$ ($10^{-3}$)</th>
<th>Cycle Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>SA</td>
<td>3.0 – 4.0</td>
<td>1 – 350</td>
</tr>
<tr>
<td>650</td>
<td>SA</td>
<td>1.4 – 5.0</td>
<td>1 – 50</td>
</tr>
<tr>
<td>550</td>
<td>Aged 750 °C, 100 h</td>
<td>4.0 – 5.7</td>
<td>1 – 50</td>
</tr>
<tr>
<td>650</td>
<td>Aged 750 °C, 100 h</td>
<td>2.1 – 6.0</td>
<td>1 – 700</td>
</tr>
</tbody>
</table>

Figure 7.6 Hysteresis loops for the fifth creep-fatigue cycle in the SA and aged conditions at (a) 550 °C and (b) 650 °C showing the critical strain, $\varepsilon_c$, for each condition. The onset of serrated flow occurs at a greater plastic strain in the aged condition at both temperatures.
7.6 Microstructural Characterization Results

7.6.1 Internal Damage and Deformation

The deformation structures after creep-fatigue testing at 550 and 650 °C with aged specimens are shown in Figure 7.7. At 550 °C, a high density of well-defined subgrains is observed adjacent to most grains boundaries, indicating significant recovery. The presence of subgrains after creep-fatigue failure in the aged microstructure is in stark contrast to the observations after testing under the same conditions with a SA microstructure, where no significant recovery is observed [7.1]. Additionally, where the internal damage in the SA microstructure primarily consists of long sharp cracks along grain boundaries, the intergranular damage in the aged condition is primarily round voids and short blunted cracks. Similarly, the aged microstructure tested at 650 °C has a significantly higher density of subgrains at failure compared to the SA microstructure. Since the grain boundary damage in the aged conditions consists primarily of voids, rather than long cracks, the area density of grain boundary voids is determined for each aged condition following failure from scanning electron micrographs. The density of intergranular voids after testing the aged microstructure is greater at 650 °C than at 550 °C, which is summarized in Table 7.4. A higher density of grain boundary voids at 650 °C is likely due to more accumulated creep strain throughout the test at 650 °C (1.05) compared to 550 °C (0.68), despite less than half the number of cycles to failure. Thus, a smaller amount of intergranular damage at 550 °C in the aged condition is due to less accumulated creep strain per cycle and slow propagation of damage due to dynamic recovery at the grain boundaries.

7.6.2 Fracture Mode

The primary fracture surfaces from the aged microstructures tested at 550 and 650 °C are shown in Figure 7.8. The mode of fracture in creep-fatigue with the aged condition is primarily transgranular at both 550 and 650 °C, as indicated by the large striated regions. Note that there is more microvoid associated damage on the fracture surface at 650 °C, which likely occurs along grain boundaries due to creep damage; the arrows in Figure 7.8 (b) indicate grain boundary associated damage. In the SA condition, the creep-fatigue fracture surfaces are primarily intergranular at 550 °C and mixed with primarily intergranular and a small fraction of transgranular components at 650 °C [7.1]. Primarily transgranular fracture in the aged condition
Figure 7.7 BSE micrographs of deformed microstructures from the gage sections of creep-fatigue tests on aged microstructures tested at (a) 550 °C and (b) 650 °C.

Table 7.4 Grain Boundary Void Density after Creep-Fatigue Failure

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Grain Boundary Void Density (10^4 voids/µm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>3.2 ± 0.7</td>
</tr>
<tr>
<td>650</td>
<td>11 ± 2.7</td>
</tr>
</tbody>
</table>

Figure 7.8 Fracture surfaces observed in the SEM for the pre-aged microstructures creep-fatigue tested at (a) 550 and (b) 650 °C. Fracture is primarily transgranular with flat striated regions. Short arrows point to grain boundary associated damage at 650 °C.
creep-fatigue tests is similar to the LCF fracture surfaces in the SA condition at both 550 and 650 °C. Note that no creep strain is accumulated in the SA condition LCF tests. Thus, although the total accumulated creep strain (and presumably, creep damage) in the aged condition creep-fatigue tests is significantly greater compared to the SA microstructure, the primarily transgranular fracture indicates a higher resistance to propagation of grain boundary damage in the aged microstructure.

7.6.3 Dislocation Density and Hardness

After creep-fatigue testing at both 550 and 650 °C, the aged microstructures have significantly lower dislocation density and room temperature hardness values compared to the SA microstructures tested under the same conditions (Figure 7.9). The lower dislocation density after testing the aged microstructure is due to a significantly higher degree of dynamic recovery compared to the SA microstructure tested at identical temperatures and hold times. Both dislocation density and hardness values are slightly higher at 550 °C compared to 650 °C, which is also true of the SA condition. For the aged conditions, the magnitudes of precipitate and solution strengthening should be comparable at both temperatures, due to the stability of the aged microstructure. Therefore, the higher hardness at 550 °C compared to 650 °C is attributed primarily to a higher dislocation density, as a result of less dynamic recovery (see the microstructures in Figure 7.7).

Figure 7.9 Comparison of (a) dislocation density and (b) Vickers hardness for pre-aged and SA microstructures after creep-fatigue testing at 550 and 650 °C.
7.6.4 Precipitation

The objective of this study is to understand how the evolution of a thermodynamically unstable microstructure (*i.e.*, the solution annealed condition) affects deformation and damage mechanisms that ultimately lead to failure in creep-fatigue. Hence, aging the microstructure prior to testing was intended to produce a microstructure that is stable and does not have increased precipitation during elevated temperature testing. It was verified that dynamic precipitation during creep-fatigue testing is not significant in the aged microstructures after testing at both 550 and 650 °C. Even though the testing times with the aged microstructures are significantly longer compared to the tests in the SA condition, fine Nb(C,N) (MX) precipitates are not observed after testing. The formation of Z-phase (CrNbN) has been proposed to be preceded by NbN [7.5], [7.7], which is likely the reason MX is observed after testing at 650 and 700 °C in the SA condition, discussed in Chapter 6. In contrast, the kinetics are favorable for Z-phase nucleation during aging at 750 °C for 100 h. However, the equilibrium volume fraction of Z-phase is not expected to be achieved after the relatively short thermal treatment. Thus, during testing in the aged condition, Z-phase likely coarsens preferentially to the formation of non-equilibrium MX. The BF TEM micrograph in Figure 7.10 (a) shows a representative distribution of coarse M23C6 and fine Z-phase after testing at 650 °C, which has a significantly larger spacing than the precipitates that form during testing from the SA microstructure. Additionally, the Z-phase particles are slightly coarser after testing at both temperatures, as shown in Figure 7.10 (b) for comparison with the same precipitates in the untested microstructure shown in Figure 3.5 (c).

7.7 Discussion

7.7.1 Serrated Flow

In Chapter 6, serrated flow was shown to disappear at some number of cycles at every temperature studied with the same tensile hold time. Furthermore, the disappearance of serrations occurred at a greater number of cycles as the test temperature decreased. It was concluded that the consumption of solutes through precipitation of carbides and nitrides is responsible for the reduced DSA effects and the disappearance of serrated flow. Precipitation occurs significantly faster at the higher test temperatures, which explains the earlier disappearance of serrated flow in terms of cycle number. Thus, it should be expected that an aging treatment to precipitate out a
significant volume fraction of carbides and nitrides might result in a complete absence of DSA during similar creep-fatigue testing conditions.

![TEM BF micrographs of precipitates in the aged microstructures after creep-fatigue testing at 650 °C showing (a) the relatively large spacing of M\textsubscript{23}C\textsubscript{6} (coarse) and Z-phase (fine) precipitates, which are shown at higher magnification in (b).](image)

Figure 7.10

In this study, serrated flow existed from the beginning of testing at both temperatures. However, the onset of serrations was delayed significantly to higher strain values in the aged microstructure, compared to the SA condition. Nakada and Keh [7.3] showed that for Ni-C alloys, the critical strain for serrated flow in monotonic tests is a function of interstitial content for a given temperature and strain rate. Specifically, a higher C-content in the Ni-C alloys results in a smaller \(\varepsilon_c\) for the same temperature and strain rate. For same alloy (i.e. same C-content) and strain rate, an increase in temperature also results in a smaller \(\varepsilon_c\). Recently, Alomari et al. [7.11] confirmed this relationship between temperature and \(\varepsilon_c\) in Alloy 709 during tensile testing in the range of temperatures and strain rates where serrated flow occurs. Although serrations are observed in the aged condition during creep-fatigue testing in this study, the larger critical strain values at each temperature, compared to the SA condition, suggests a smaller solute concentration responsible for DSA. Furthermore, the larger \(\varepsilon_c\) at 550 °C compared to 650 °C is consistent in both microstructures, and with the literature where composition is constant [7.3], [7.4], [7.11].
The rate at which $\varepsilon_c$ increases may be indicative of the rate of microstructural evolution. In the SA condition, $\varepsilon_c$ increases at a rate of $7 \times 10^{-5}$ cycle$^{-1}$ until the disappearance of serrations at 650 °C and at a slower rate of $3 \times 10^{-6}$ cycle$^{-1}$ at 550 °C. The significantly higher rate at which $\varepsilon_c$ increases at 650 °C is attributed to the faster precipitation of $M_{23}C_6$ and Nb(C,N). In the aged condition, $\varepsilon_c$ also increases for both temperatures even though significant precipitation is not observed. McCormick showed that, in addition to the effect of concentration and temperature, critical strain also increases with increasing mobile dislocation density [7.4]. An increase in dislocation density may offer an explanation for the critical strain increase observed in the aged condition tests. At 550 °C in the aged condition, $\varepsilon_c$ increases at $3 \times 10^{-5}$ cycle$^{-1}$, a rate significantly faster compared to the SA condition. During the short cyclic hardening stage where the increase in critical strain is rapid, the friction and back stress are larger in the aged condition than in the SA condition. The larger number of obstacles in the matrix (i.e. the fine Z-phase that precipitated on dislocations during the aging treatment) are likely responsible for a more rapid increase in dislocation density in the aged microstructure, compared to the SA condition. Note that the strain-hardening rate is greater in the aged condition tests in the hysteresis loops shown in Figure 7.6. Similarly, at 650 °C, $\varepsilon_c$ increases rapidly in ~10 cycles in the aged condition followed by a slower rate of increase until the eventual disappearance at ~700 cycles. The tensile stress is greater in the aged condition for the first ~10 cycles, which supports the increase of dislocations as an explanation for the increase in critical strain.

7.7.2 Solute Concentration and Cyclic Plastic Deformation

The concentration of interstitial solutes has a significant effect on deformation behavior in alloys where DSA occurs [7.3], [7.12]–[7.15]. Kocks et al. showed that lower concentrations of C in Ni-C alloys resulted in lower strain-hardening rates and an earlier onset of dynamic recovery (i.e. at lower stresses) in monotonic tensile tests at elevated temperatures where DSA occurs [7.12]. The effect of solute concentration on strain-hardening is primarily attributed to the rate of dislocation rearrangement (i.e. recovery). Flow stress is also concentration dependent, as both friction stress and dislocation interaction strength increase with solute concentration [7.12]. Hong and Laird proposed that interstitial solutes in FCC inhibit dislocation cross-slip through elastic interactions with the dissociated partials (i.e. by an increase in friction stress), which is the reason for delayed recovery and high strain-hardening rates in conditions where DSA occurs.
Hence, under certain conditions there exists a solute concentration dependence on dynamic recovery by dislocation cross-slip.

In the aged Alloy 709 microstructure, the relatively low interstitial solute content results in significant dynamic recovery during creep-fatigue testing at 550 and 650 °C. The stable cyclic stress response, well-defined subgrains, and relatively low dislocation densities indicate that strain-hardening is balanced by dynamic recovery, which is typical for low-interstitial austenitic stainless steels that exhibit wavy slip in LCF (e.g. 304L and 316L) [7.17]–[7.19]. Dynamic recovery in the aged microstructure is attributed to an increase in dislocation cross-slip, rather than an increase in dislocation climb, which is supported by the relatively unchanged amount of creep deformation during the hold at both temperatures, compared to the SA condition. Enhanced dislocation cross-slip is primarily responsible for the lower strain-hardening rates and larger magnitude of cyclic plasticity in the aged condition, compared to SA condition. Note that the mid-life strain-hardening rates were approximately the same in both microstructures at 650 °C because significant cross-slip already occurs at the mid-life cycle in the SA condition.

As discussed in Chapter 6, the friction stress calculated here is simply a measure of the cyclic yield stress. A lower yield stress is also partially responsible for more cyclic plasticity in the aged microstructure; the lower yield strength is due to coarser precipitates, lower solute concentration, and lower dislocation density compared to the SA condition. Between the two microstructures, the significantly larger increase in cyclic plasticity at 550 °C, compared to 650 °C, is attributed to both a decrease in strain-hardening rates and a decrease in the yield stress. At 650 °C, where the strain-hardening behavior is relatively unchanged with aging, the increase in cyclic plasticity is primarily due to the decrease in yield strength. In the SA condition, the high yield strength is due to a combination of fine precipitates, relatively high solute concentration, and a high dislocation density. In the aged condition, coarse M$_2$3C$_6$ precipitates and a low volume fraction of widely spaced fine Z-phase particles result in a lower yield strength and strain-hardening rate. Additionally, the lower solute concentration lowers the true friction stress on dislocations, leading to enhanced cross-slip and a reduction of dislocation density, which also lowers both the strain-hardening rates and yield strength.

The presence of serrations in the aged microstructure indicates that although there is still a high enough concentration of solutes for DSA effects to be observed, the concentration is low enough that the applied stress can overcome the friction stress restricting cross-slip. Therefore,
the presence of DSA does not always correlate with enhanced slip planarity. Furthermore, cross-slip and dynamic recovery are impeded only if the solute concentration is above a critical value (for a given applied stress).

Since significant cross-slip occurs at both test temperatures in this study, an estimate of the solute concentration in the aged microstructure indicates where the slip mode transition from planar to wavy occurs in Alloy 709 under these testing conditions. In Chapter 5, small angle neutron scattering (SANS) is used to characterize the volume fraction of $\text{M}_{23}\text{C}_6$ carbides for several conditions of static aging and creep-fatigue testing. Due to the small volume fraction of $Z$-phase and the similarity of scattering contrast between that phase and the matrix, accurate estimates were not obtained for this phase. Therefore, the volume fraction of $Z$-phase determined by TC-Prisma® precipitation simulations is used to estimate the concentration of N consumed during aging, which is ~50 pct of the equilibrium volume fraction (0.024 pct) after 100 h at 750 °C. The measured volume fraction of $\text{M}_{23}\text{C}_6$ carbides after aging at 750 °C for 100 h is approximately 0.88 pct, which is somewhat lower than the equilibrium concentration (1.36 pct). Accounting for the $Z$-phase and $\text{M}_{23}\text{C}_6$ precipitation, approximately 0.21 at pct C and 0.01 at pct N are consumed by precipitation during aging. The modified SA composition, accounting for MX precipitates that are not solutionized, has 0.29 at pct C (0.063 wt pct C) and 0.44 at pct N (0.11 wt pct N). Therefore, the aged microstructure has approximately 0.08 at pct C (0.017 wt pct C) and 0.43 at pct N (0.108 wt pct N) in solution.

In the conditions studied here, a transition from planar to wavy slip occurs in Alloy 709 where the solute concentrations of C and N are as low as 0.08 at pct and 0.43 at pct, respectively. Although the effects of interstitial N are often attributed to the enhanced slip planarity during deformation in austenitic stainless steels [7.15], [7.19], [7.20], similar effects from interstitial C have been reported in Ni alloys and stainless steels which do not contain significant N [7.3], [7.12], [7.13], [7.21]. In fact, due to the similarity in the diffusion of C and N in austenite, the interstitials are typically grouped together (C + N) in activation energy calculations for serrated flow [7.11], [7.14]. Additionally, the size misfit parameter is similar between the two interstitials (although N is somewhat larger than C [7.22]), such that the increase in friction stress is also best understood by the collective concentration (i.e. C + N). In this study, there is a significant reduction of interstitial C from the aging treatment, but the N is relatively unchanged due to the small volume fraction of $Z$-phase. During creep-fatigue testing at 550 °C, the transition in
deformation behavior from planar in the SA condition to wavy slip in the aged condition is, therefore, due to a reduction of the total interstitial concentration.

It should be noted that the SANS measured volume fractions of $\text{M}_2\text{C}_6$ carbides after creep-fatigue testing in the SA condition at 550 and 650 °C are approximately 0.25 pct and 0.80 pct, respectively. The C consumed by dynamic precipitation in the SA condition during creep-fatigue testing at 650 °C is similar to that consumed during the aging treatment, which explains the similar deformation behavior between the two microstructures at 650 °C.

### 7.7.3 Time-Dependent Deformation

The magnitude of time-dependent creep deformation during the 30 min tensile hold is approximately the same for both microstructures. However, the lower stresses at the beginning of the tensile hold in the aged condition compared to the SA condition (~25 pct lower at 550 °C and ~23 pct lower at 650 °C) indicate that the creep resistance is affected by the aging treatment. This result suggests that the relatively uniform distribution of fine intragranular precipitates in the SA condition is responsible for the high creep resistance due to the additional threshold stress imparted on climbing dislocations. Additionally, the drag stress from solute atmospheres on climbing dislocations is assumed to be higher in the SA condition [7.23]. Aging the microstructure lowers the concentrations of interstitial solutes (C and N), which decreases the resistance of dislocation climb and promotes dynamic recovery by dislocation cross-slip. In strain-controlled creep-fatigue conditions, a decrease in flow strength and enhanced recovery leads to longer cyclic lives. However, it should be noted that the reduction of creep strength and enhanced dynamic recovery may be detrimental for constant stress creep conditions.

### 7.7.4 Interaction of Deformation and Damage Mechanisms

The effect of initial microstructure of Alloy 709 on plastic deformation and number of cycles to failure is shown in Figure 7.11. This figure illustrates the significance of cyclic plasticity and inelastic creep deformation on creep-fatigue performance under the conditions studied here. Aging the microstructure results in a significant increase in the amplitude of cyclic plastic deformation during creep-fatigue at both 550 and 650 °C. The lower friction stress and flow stress in the aged condition compared to the SA condition are responsible for the increase in dislocation cross-slip and enhanced plastic deformation during cycling. However, the magnitude
of creep deformation per cycle at both temperatures is practically unaffected by aging the microstructure. Therefore, a higher amount of cyclic plasticity for a given amount of creep damage results in an increase in creep-fatigue life.

![Figure 7.11 Number of cycles to failure, $N_f$, average cyclic plastic strain amplitude, $\varepsilon_{ap,cyc}$, and average creep strain, $\varepsilon_{pcr}$, for each cycle as a function of test temperature for creep-fatigue tests with 30 min tensile holds.](image)

The increase in cycles to failure with increasing cyclic plasticity, as shown in Figure 7.11, is accompanied by a significant change in failure mechanisms and damage propagation. The change in failure mechanisms is most obvious at 550 °C. At this temperature, the fracture mode changes from intergranular in the SA condition to transgranular with the aged microstructure, despite an accumulation of creep strain in the aged condition that is more than three times that of the SA condition. The importance of this result lies in the presumption that creep damage (i.e. grain boundary void nucleation and growth) is related to the magnitude of creep deformation at strain rates below which dislocation glide accommodates the strain [7.9]. In other words, the total accumulated creep damage at the end of life is estimated to be approximately three times greater in the aged condition. Additionally, the morphology of grain boundary damage is different in the two microstructures; a high density of long sharp intragranular cracks form in the SA condition compared to grain boundary (triple point) voids and short blunted cracks in the aged condition. The difference in the fracture mode between the two microstructural conditions at 550 °C is attributed to the propagation of internal grain
boundary damage, as a result of the difference in cyclic plasticity. In the aged condition, damaged boundaries are surrounded by well-defined subgrains, indicating significant localized plastic deformation. Hence, the significant increase in plastic deformation by dislocation cross-slip in the vicinity of grain boundary voids (or cracks) increases the local grain boundary toughness. Furthermore, the decrease in strain-hardening as a result of pre-aging the microstructure results in lower tensile stresses to propagate the cracks. Creep-fatigue testing at 550 °C from the SA condition results in high tensile stresses and limited plastic strain, which leads to rapid propagation of grain boundary damage and brittle intergranular fracture.

The importance of creep strain is emphasized for the 650 °C test temperature. Although there is an increase in the creep-fatigue life in the aged microstructure at 650 °C, compared to the SA condition, the increase is less than at that achieved at 550 °C. The smaller increase in cycles to failure at 650 °C by pre-aging the microstructure is attributed to the relatively high average creep strain per cycle and the total accumulated creep strain compared to 550 °C, as shown in Figure 7.11. Although the flow strength is lower and the cyclic plastic strain is higher in the aged condition than the SA condition, resulting in a higher density of subgrains in the aged condition, the creep damage is more significant compared to the lower temperature. This result emphasizes the detrimental effect of creep strain, which occurs during the hold at peak tensile strain, on creep-fatigue life. Thus, when there is an adequate amount of cyclic deformation to reduce the tensile stresses and increase the grain boundary toughness, the creep-fatigue performance is best where the creep damage accumulation is minimized.

The results of this research have profound effects on the extrapolation of creep-fatigue behavior and performance from accelerated laboratory tests to long term service conditions. As the microstructure evolves from the solution annealed condition during long times at elevated temperatures in service, the deformation and damage mechanisms also change significantly. The initially potent solid solution strengthening promotes planar deformation, thereby inhibiting dynamic recovery, which is desirable for constant stress creep conditions. However, as carbides and nitrides form during long times at temperature, the tendency for dislocation cross-slip increases, leading to dynamic recovery, which is desirable for retarding the propagation of internal damage in strain-controlled LCF conditions. Since dynamic recovery is not desirable for creep strength, the results of this study suggest that there is a trade-off between constant load creep and strain-controlled creep-fatigue performance. Furthermore, the microstructural
evolution that leads to the transition in deformation behavior \(i.e.\) planar to wavy slip) and the improved creep-fatigue life occurs slowly at the expected service temperature (550 °C) compared to higher testing temperatures \(e.g.\) 650 °C). This result is supported by the static aging study and the creep-fatigue tests with the SA microstructure in Chapters 4, 5, and 6. The difference in microstructural evolution among different test temperatures, and the associated transition in deformation and damage mechanisms, indicates that accelerated laboratory testing of the solution annealed microstructure is not representative of the long term creep-fatigue performance in service. Therefore, accelerated testing with a stable pre-aged microstructure should be considered to be more representative of longer term creep-fatigue performance of Alloy 709.

7.8 Conclusions

1. Creep-fatigue performance is optimized in Alloy 709 by promoting wavy deformation during cycling and minimizing the amount of creep strain during the hold period. Cross-slip promotes dynamic recovery in the vicinity of grain boundaries, reducing the strain energy for the propagation of internal creep damage, which results in higher grain boundary toughness, transgranular fracture, and longer creep-fatigue lives.

2. A reduction of the total interstitial concentration \((C + N)\) during precipitation of carbides and nitrides from exposure at elevated temperatures results in a transition in cyclic deformation character from planar to wavy. In this study, interstitial C was reduced significantly more than N during the pre-aging heat treatment. Therefore, the high friction stress that results in the inhibition of dislocation cross-slip is not due to interstitial N alone.

3. Pre-aging results in a reduction in solution and precipitate strengthening, due to the coarse nature of the carbides that form during static aging compared to those that precipitate dynamically. Dynamic recovery, as a result of lower solution and precipitate strengthening, lowers the dislocation density. Therefore, a decrease in Taylor hardening also occurs as a result of pre-aging. Relatively low flow stress and strain-hardening rates result in enhanced cyclic plasticity, low peak tensile stresses, and slow propagation of creep-fatigue damage.
4. Creep resistance is reduced somewhat in Alloy 709 as a result of the aging treatment used in this study. Although the creep strain per cycle did not change significantly in creep-fatigue testing, this result has important implications for constant stress creep conditions.

7.9 References


The objective of this research was to develop an understanding of the deformation and damage mechanisms responsible for creep-fatigue failure of Alloy 709 across a range of accelerated testing conditions. A mechanistic understanding of LCF and creep-fatigue behavior provides a basis for reliable extrapolation of short-term test data to long-term service conditions relevant to nuclear applications. Specifically, the effects of microstructural evolution during accelerated laboratory testing were investigated with respect to creep-fatigue behavior and performance, and compared to the effects of long-term microstructural evolution during service. The conclusions of this study are addressed here with respect to the specific research questions posed in Chapter 1.

**How do the cyclic and time-dependent deformation mechanisms at 550 °C, compared to 650 °C, affect the cyclic stress-strain response, internal damage formation and propagation, and reduction in creep-fatigue life compared to pure LCF?**

This question was primarily addressed through tests on the solution annealed condition. Cyclic plasticity and time-dependent creep deformation have different effects on the formation and propagation of damage that lead to creep-fatigue failure. Specifically, creep deformation is responsible for grain boundary cavitation and void formation. At both temperatures the accumulation of creep strain correlates with creep damage (*i.e.* grain boundary voids). On the other hand, cyclic plastic deformation reduces the strain energy available for propagation of grain boundary creep damage.

The amount of time-dependent creep strain accumulated during creep-fatigue at 550 °C is a small fraction of that accumulated at 650 °C, due to less stress relaxation during the tensile hold. Despite a greater resistance to creep deformation at 550 °C, the creep-fatigue life is reduced significantly more at 550 °C compared to 650 °C, relative to LCF.

Therefore, the significant difference in creep-fatigue behavior and life at 550 and 650 °C is attributed to differences in cyclic deformation mechanisms when testing from a solution annealed condition. Specifically, at 550 °C, slip is primarily planar for the duration of the test due to a strong inhibition of dislocation cross-slip, which prevents recovery and leads to high stresses during cycling. The lack of dynamic recovery results in propagation of grain boundary damage and failure by intergranular fracture. At 650 °C, the deformation character changes
during creep-fatigue testing from planar to wavy, which is associated with cyclic softening and subgrain formation. Recovery during creep-fatigue testing results in lower stresses, blunting of internal damage, and mixed mode fracture propagation (transgranular and intergranular). Although the accumulated creep damage is greater in creep-fatigue at 650 °C, compared to 550 °C, a greater amount of plastic deformation during cyclic loading retards the propagation of the grain boundary damage.

The shortest creep-fatigue lives occur under conditions of minimal cyclic plasticity, while the best creep-fatigue performance is realized with a moderate amount of cyclic deformation and a minimal amount of creep deformation.

**How does initial microstructure and microstructural evolution (specifically precipitation) of Alloy 709 affect LCF and creep-fatigue behavior in accelerated laboratory testing compared to the behavior expected during long-term service exposure?**

The microstructural instability of solution annealed Alloy 709 results in an evolution of precipitate and solid solution strengthening during short-term LCF and creep-fatigue testing at elevated temperatures. Intragranular precipitation of carbides and nitrides (M$_2$3C$_6$ and Nb(C,N)) increases the strength initially, followed by a decrease as precipitates coarsen. Additionally, dynamic precipitation leads to a reduction in solid solution strengthening as solute concentration decreases. The microstructural evolution, and thus the evolution of strengthening mechanisms, is a strong function of test temperature. The higher strength of Alloy 709 at the lower testing temperatures in this study is a result of finer precipitates and higher solute content compared to higher temperatures.

Solute atoms responsible for dynamic strain aging (DSA) promote planar deformation and strain-hardening through an increase in friction stress on partial dislocations, which inhibits cross-slip and recovery. Dynamic precipitation decreases the solute content, lowers the friction stress, and leads to a transition in slip character from planar to wavy, in turn promoting recovery. Dynamic recovery correlates with lower strain-hardening rates and lower dislocation densities. Therefore, a reduction of the solute content also leads to a lower magnitude of Taylor hardening. The reduction in strength and the transition of slip character as a result of dynamic aging results in more cyclic plasticity and lower stress at peak strain.

Static aging of Alloy 709 under conditions which promote significant carbide and nitride precipitation prior to creep-fatigue testing results in a substantial increase in the creep-fatigue life.
at 550 and 650 °C, compared to the solution annealed condition. A reduction of the total interstitial concentration (C + N) from aging promotes cross-slip and dynamic recovery at lower stresses than in the solution annealed condition. Aging prior to testing also results in a reduction in precipitate strengthening, due to the coarse nature of the $M_{23}C_6$ carbides that form during static aging compared to those that precipitate dynamically. Although fine Z-phase also forms during static aging, the small volume fraction does not result in significant strengthening. The enhanced cyclic plasticity and relatively low peak tensile stresses after static aging result in transgranular fracture in creep-fatigue under the same test conditions where failure occurs by intergranular fracture in the solution annealed condition.

The significant improvement in creep-fatigue life at 550 °C with the pre-aged microstructure is due to more cyclic plasticity with approximately the same amount of creep strain per cycle compared to the solution annealed condition. Although the cyclic plasticity is also greater at 650 °C in the aged condition, a smaller increase in creep-fatigue life over the solution annealed condition is due to the relatively high creep strain per cycle compared to the test at 550 °C. These results support the hypothesis that creep-fatigue performance of Alloy 709 is optimized by enhancing the cyclic plasticity and minimizing the creep strain.

Creep resistance is reduced somewhat in Alloy 709 as a result of the aging treatment used in this study. For the creep-fatigue conditions studied, the creep strain per cycle is approximately the same in both the aged and the solution annealed microstructures, although the stresses are significantly lower in the aged condition. A lower creep resistance in the aged condition has important implications for constant stress creep conditions.

The results of this study indicate that the creep-fatigue performance during accelerated testing from a solution annealed condition is not representative of the performance during long-term service. Although precipitation during static aging of Alloy 709 at the expected service temperature of 550 °C is relatively slow compared to the higher temperatures studied, significant precipitation is expected in a short time compared to the expected service life. Therefore, the deformation behavior in the aged microstructure at the expected service temperature of 550 °C, which results in slow propagation of intergranular creep damage, is more indicative of the behavior during long-term nuclear service.
CHAPTER 9
FUTURE WORK

This chapter provides several possible pathways for continued understanding and optimization of the creep-fatigue performance in Alloy 709.

9.1 LCF Tests with Aged Microstructures

A significant improvement in the creep-fatigue performance at the expected FSR service temperature is achieved by thermal aging Alloy 709 at 750 °C for 100 h prior to testing. However, it is unclear what the effect of aging is on LCF performance. Based on the design methodology and life-prediction method used by ASME for nuclear structural materials code, the fatigue life reduction under creep-fatigue conditions is necessary. In other words, the damage diagram used for component design requires a normalization of creep-fatigue life by the LCF life. The damage diagram is useful for comparison to other code qualified materials (i.e. Type 316 stainless steel). Therefore, a direct comparison of the lives under LCF and creep-fatigue should be made with the same microstructure. Furthermore, LCF testing with the aged microstructure would provide an understanding of the fatigue damage mechanisms in a stable long-term microstructure, independent of the interaction with creep damage.

9.2 The Effect of Strain Range and Strain Rate on Creep-Fatigue Behavior

The scope of the current research was limited to a testing with a single strain rate and strain range. While the conditions tested in this study are typical for accelerated testing in order to collect a significant amount of data in a timely manner, they are not representative of those expected during service. Specifically, the creep-fatigue behavior should be studied with smaller strain ranges and slower cyclic strain rates. Since dynamic strain aging is a time-dependent phenomenon, slower strain rates may be more detrimental to the creep-fatigue life if dynamic recovery is inhibited. The magnitude of stress relaxation, and the associated creep strain rates, are smaller with smaller strain ranges. Although the accumulation of creep strain is expected to be slower with smaller strain ranges, the formation of creep damage needs to be understood at lower stresses and creep strain rates.
9.3 Heat Treatment Optimization

The effect of static thermal aging of Alloy 709 prior to creep-fatigue testing was investigated in detail for one aging condition (750 °C for 100 h). A second aging condition (650 °C for 500 h) was also tested under the same conditions. The results are briefly discussed in Appendix C, but microstructural characterization was not carried out on the second aged microstructure after testing due to time constraint. The two aging treatments result in slightly different microstructures, namely the presence of Z-phase. The slight difference in initial microstructure results in a different creep-fatigue response and life at both temperatures tested. These results indicate that the creep-fatigue performance of this alloy is highly sensitive to the starting microstructure. An optimal heat treatment prior to service may be obtained by minimizing creep deformation and maximizing cyclic plasticity through a balance of precipitation and solution strengthening.

9.4 Effect of Sigma Phase on Creep-Fatigue

Sigma phase forms after long times at temperature and is generally considered detrimental to mechanical properties in stainless steels. At the expected FSR service temperature, Sigma is an equilibrium phase that is predicted to form during component life-times. Therefore, the effect of Sigma phase on the creep-fatigue properties of Alloy 709 should be investigated. A long time aging treatment designed to precipitate Sigma on grain boundaries should be conducted prior to creep-fatigue testing at the expected service temperature.

9.5 Characterization of Interstitial Solute Content with Internal Friction and XRD

The results of this study indicate that the interstitial solute concentration has a significant effect on the cyclic deformation mechanisms and creep-fatigue failure. Solute atoms effect dislocation motion by both cross-slip and climb. In strain-controlled creep-fatigue, it seems there is a trade-off between the benefits of cross-slip to enhance cyclic plasticity and the drawbacks of climb which promotes cavitation damage (negative climb). In order to achieve an optimal microstructure for creep cavitation damage in this material, a further understanding of the effects both C and N have on elevated temperature deformation should be developed. Interstitial solute content should be varied systematically and correlated with both cyclic and time-
dependent deformation. Interstitial solute content could be characterized with internal friction (IF) measurements of high resolution X-ray diffraction, where a shift in lattice parameter can be related to solute concentration. Additionally, atom probe tomography may be used to investigate the nature of the solute atmospheres around dislocations.
APPENDIX A

DAMAGE DIAGRAM CONSTRUCTION

As discussed in Chapter 2, the damage accumulation under creep-fatigue conditions, according to the ASME Boiler and Pressure Vessel Code, is to be evaluated through a linear summation of fatigue and creep damage according to the criterion given by

\[
\sum_j \left( \frac{n}{N_d} \right)_j + \sum_k \left( \frac{\Delta t}{T_d} \right)_k \leq D
\]

where \( D \) is the allowable combined damage fraction, \( n \) and \( N_d \) are the number of cycles of type \( j \) and allowable number of cycles of the same type, respectively, and \( \Delta t \) and \( T_d \) are the actual time at stress level \( k \) and the allowable time at that stress level, respectively. The fatigue and creep damage terms (the left and right terms on the left side of Equation A.1, respectively) are evaluated in an uncoupled manner, and the interaction is accounted for empirically by the \( D \) term through strain-controlled creep-fatigue test data at various temperatures, strain ranges, and hold times.

The fatigue damage portion of the interaction is relatively straightforward and is defined as the ratio of cycles to failure under creep-fatigue conditions, \( n \), to the number of cycles to failure under continuous cycling conditions, \( N_d \), for the same strain rate, total strain range, and temperature.

The time-dependent component of the damage calculation is more involved and requires knowledge of the creep-rupture time for a range of stresses since \( \Delta t \) is the time at a given stress and \( T_d \) is the time to rupture at that given stress. Essentially, the time spent at a given stress is normalized by the rupture time at that stress and summed over the entire hold time to account for accumulated creep damage per cycle. Therefore, the instantaneous stress during relaxation and the time to rupture for any stress at a given temperature are needed for the creep damage analysis.

As described in Section 3.8.3 in Chapter 3, the stress during relaxation is determined as a function of relaxation time according to Equations 3.16 and 3.17. Equation 3.18 is a simplified version of Equation 3.17, in which stress is a function of time and three empirical constants: \( a \), \( b \), and \( c \). The stress relaxation data is fit to the relation in Equation 3.18 using a non-linear solver,
as described in Section 3.8.3 in Chapter 3, to determine the instantaneous stress during relaxation.

Evaluation of the creep-rupture time at a given stress and temperature is commonly done by extrapolating creep-rupture data using a Larson-Miller relationship, which is linear in semi-log space. The Larson-Miller parameter (LMP) is defined as

\[ LMP = T(C + \log_{10}(t)) \]  

where \( T \) is temperature in Kelvin, \( C \) is the Larson-Miller constant, and \( t \) is rupture time in hours. LMP is generally represented as a polynomial function of \( \log \) stress of degree \( n \) less than 3. When \( n \) equals 1, a linear equation takes the form

\[ LMP = a_0 + a_1 \log_{10} \sigma \]  

where \( a_0 \) and \( a_1 \) are fitting parameters and \( \sigma \) is stress in MPa. Combining Equations A.2 and A.3 gives

\[ \log_{10} t = -C + \frac{a_o}{T} + \frac{a_1}{T} \log_{10} \sigma \]  

The fitting parameters, \( a_0 \) and \( a_1 \), and the Larson-Miller constant, \( C \), can then be obtained using a linear regression analysis of the empirical creep-rupture data. This method allows the use of all creep-rupture data generated for a given material to be used to extrapolate to conditions that are not frequently tested such as low temperature and low stress.

Simplifying Equation A.4 gives time to rupture, \( T_d \), as a function of stress for a given temperature from

\[ T_d = A \sigma^m \]  

where \( A \) and \( m \) are fitting constants specific to test temperature. The stress during relaxation, determined from Equation 3.18, can be plugged into Equation A.5. Finally, the accumulated creep damage for a given cycle (typically the mid-life cycle is chosen to be representative of all
cycles), $D_k$, is determined by integrating the time spent at a given stress divided by the rupture time at that stress according to the following equation:

$$D_k = \int_0^{t_h} \frac{1}{T_d} dt = \frac{a^{-m}}{A(1 - cm)} \left( (t_h + b)^{1-cm} - (b)^{1-cm} \right)$$

where $t_h$ is the hold time in seconds. The damage for the mid-life cycle is multiplied by the number of cycles in the test to estimate the accumulated creep damage during a test, $D_c$.

Therefore, when LCF and creep-fatigue test data are available at a constant temperature, strain amplitude, and strain rate, the accumulated creep and fatigue damage can be computed from the above analysis. Finally, a damage diagram can be constructed for the material which is necessary for qualification in nuclear reactor construction.

The individual damage components have been evaluated from LCF and creep-fatigue tests (with 30 min tensile holds) on solution annealed Alloy 709 (heats 011593 and 011594) at 550 and 650 °C. The approximate mid-life cycle stress relaxation data was from each creep-fatigue test was fit to Equation 3.18; the resulting constants are given in Table A.1. The Larson-Miller plot shown in Chapter 2 for NF709 was used to determine rupture time as a function of stress and temperature; the resulting constants in Equations A.2, A.3, and A.5 are given in Table A.2. The tabulated damage components, $D_k$, $D_c$, and $D_f$, where $D_f$ is the fatigue component, are given in Table A.3. Finally, the damage diagram is shown in Figure 3.9, along with the design curves for other nuclear grade steels for reference.

### Table A.1 Constants for Mid-Life Stress Relaxation Function

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Heat ID</th>
<th>Approximate Mid-Life Cycle</th>
<th>$a$</th>
<th>$b$</th>
<th>$c$</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>011594</td>
<td>500</td>
<td>435.38</td>
<td>167.04</td>
<td>-0.0165</td>
</tr>
<tr>
<td>550</td>
<td>011593</td>
<td>500</td>
<td>434.50</td>
<td>62.11</td>
<td>-0.0147</td>
</tr>
<tr>
<td>650</td>
<td>011594</td>
<td>500</td>
<td>362.37</td>
<td>13.29</td>
<td>-0.0778</td>
</tr>
<tr>
<td>650</td>
<td>011593</td>
<td>500</td>
<td>364.03</td>
<td>9.78</td>
<td>-0.0782</td>
</tr>
</tbody>
</table>

### Table A.2 Larson Miller Constants

<table>
<thead>
<tr>
<th>$C$</th>
<th>$a_0$</th>
<th>$a_1$</th>
<th>$m$</th>
<th>$A$ At 550 °C</th>
<th>$A$ At 650 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>16.41</td>
<td>32721.95</td>
<td>-6164.12</td>
<td>-7.49</td>
<td>8.02×10^{26}</td>
<td>3.95×10^{22}</td>
</tr>
</tbody>
</table>
Table A.3 Calculated Fatigue and Creep Damage Components

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Heat ID</th>
<th>Cycles to Failure</th>
<th>$D_b \times 10^{-5}$</th>
<th>$D_c$</th>
<th>$D_f$</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>011594</td>
<td>n 1062 N 3629</td>
<td>5.641</td>
<td>0.060</td>
<td>0.247</td>
</tr>
<tr>
<td>550</td>
<td>011593</td>
<td>n 888 N 4964</td>
<td>6.208</td>
<td>0.055</td>
<td>0.207</td>
</tr>
<tr>
<td>650</td>
<td>011594</td>
<td>n 931 N 2047</td>
<td>21.64</td>
<td>0.201</td>
<td>0.474</td>
</tr>
<tr>
<td>650</td>
<td>011593</td>
<td>n 1069 N 1883</td>
<td>22.25</td>
<td>0.238</td>
<td>0.544</td>
</tr>
</tbody>
</table>

Figure A.1 Damage diagram for solution annealed Alloy 709 at 550 and 650 °C. Design curves for Type 304 and 316 stainless steels, 2.25Cr-1Mo, and 800H are shown for reference.
APPENDIX B
MISCELLANEOUS CREEP-FATIGUE TESTS

B.1 Small Strain Amplitude (0.4 pct) Creep-Fatigue Tests

A creep-fatigue test with a 0.4 pct total strain amplitude and a 30 min tensile hold time was initiated at 550 °C with the intention of running to failure, but was unintentionally interrupted after approximately 3000 cycles. This test revealed that at 550 °C, only cyclic hardening occurred within the first 3000 cycles, as shown in Figure B.1.

![Figure B.1](image)

Figure B.1 Peak tensile stress versus cycle number for a creep-fatigue test at 550 °C with $\varepsilon_{a,T} = 0.4$ pct and $t_h = 30$ min that was interrupted after approximately 3000 cycles.

Based on the differences in cyclic hardening/softening behavior between the 550 and 650 °C creep-fatigue tests (30 min hold) with a strain amplitude of 1 pct and a hold time of 30 min, creep-fatigue tests were initiated at both 550 and 650 °C with a total strain amplitude of 0.4 pct to identify behavioral differences (hardening/softening) between the two temperatures. Both tests were initiated at the same time and once cyclic softening was observed in the 650 °C condition, both tests were interrupted. Figure B.2 shows the peak tensile stress as a function of cycle number for the small strain amplitude tests, which were interrupted at approximately 850 cycles. As expected, based on the test that ran for over 3000 cycles, only cyclic hardening is observed at 550 °C. However, after approximately 250 cycles, there is a maximum peak tensile stress at 650 °C, followed by slight softening until the test was interrupted. The cyclic softening
at 650 °C indicates that the cyclic behavior is markedly different between the two temperatures not only at large strain ranges, but also at smaller strains more relevant to actual service conditions. The onset of cyclic softening in the small strain amplitude test occurs at a cycle count approximately five times larger (~250 cycles) than in the 1 pct strain amplitude tests (~50 cycles).

B.2 Creep-Fatigue Tests with a Microstructure Aged at 650 °C for 500 h

Test specimens were aged at 650 °C for 500 h and creep-fatigue tested at 550 and 650 °C with 30 min holds. The aged microstructure was detailed in Chapter 5. At 550 °C, the number of cycles to failure was 3958. At 650 °C, the number of cycles to failure was 889. At both test temperatures, the peak tensile stress is greater than for the microstructure aged at 750 °C for 100 h and less than the SA microstructure, as shown in Figure 3.13. At 550 °C, the cyclic stress response is stable for the condition aged at 650 °C and failure occurs at a slightly higher number of cycles compared to the condition aged at 750 °C (3337 cycles). At 650 °C, the initial cyclic hardening in the condition aged at 650 °C is followed by cyclic softening at a rate similar to the SA condition until failure at a lower cycle count compared to the condition aged at 750 °C (1438 cycles).
Figure B.3 Peak tensile stress versus cycle number for creep-fatigue tests at (a) 550 and (b) 650 °C with specimens that were static aged at 650 °C for 500 h and at 750 °C for 100 h, compared to the solution annealed (SA) condition.

The evolution of friction and back stress during creep-fatigue tests for both aged microstructures and the SA condition are shown in Figure B.4. At 550 °C, the friction stress in the 650 °C aged condition is greater than in the condition aged at 750 °C, but well below the friction stress in the SA condition after the initial hardening period. In both aged conditions, the friction stress is relatively stable at 550 °C compared to the SA condition. At 650 °C, the friction stress in the microstructure aged at 650 °C is significantly higher than in the condition aged at 750 °C, and approximately the same as the SA microstructure after 100 cycles. The back stress at 550 °C is approximately the same in both aged conditions. At 650 °C, the back stress in the condition aged at 650 °C is approximately the same as the SA microstructure, and slightly higher than the condition aged at 750 °C.

The cyclic plastic strain and creep strain per cycle are shown in Figure B.5 for both aged microstructures and the SA condition. For both test temperatures, the cyclic plastic strain in the condition aged at 650 °C is smaller than in the condition aged at 750 °C, but larger than in the SA condition. The magnitude of creep strain is approximately the same in all conditions tested at 550 °C. At 650 °C, the magnitude of creep strain for each cycle is slightly higher in the condition aged at 650 °C than in the condition aged at 750 °C or the SA condition.
As discussed in Chapter 5, the volume fraction of carbides in the microstructure aged at 650 °C for 500 h is approximately the same as in the microstructure aged at 750 °C for 100 h. Therefore, the interstitial C is also expected to be approximately the same in both conditions. However, a significant dispersion of Z-phase particles is present after aging at 750 °C for 100 h, which are not observed after aging at 650 °C for 500 h. Instead, smaller MX precipitates are occasionally observed after aging at 650 °C. Since the volume fractions of Z-phase and MX could not be determined from the SANS experiment, it is unclear what the differences are in interstitial N between the two aged conditions. The hardness in the 750 °C, 100 h aged condition (197.7 ± 4.2 HV) is significantly greater than in the 650 °C, 500 h aged condition (183.0 ± 5.7 HV). The higher hardness in the 750 °C, 100 h condition indicates a higher volume fraction of fine precipitates compared to the condition aged at 650 °C for 500 h. Therefore, it is interpreted that the solute content in the 750 °C aged condition is lower than in the 650 °C aged condition. A higher solute content after aging at 650 °C corroborates with higher friction stress values and less cyclic plastic deformation per cycle compared to the microstructure aged at 750 °C. The approximately equal back stress between the two aged conditions tested at 550 °C, along with the stable cyclic stress behavior, indicates similar cyclic deformation behavior in both microstructures (i.e. wavy slip). Increased cyclic plastic deformation over the SA condition after aging at 650 °C, although slightly less than the cyclic plasticity after aging at 750 °C, retards the propagation of internal damage and results in a significantly longer creep-fatigue life compared to the SA condition.

These results indicate that the microstructure (i.e. solute concentration and precipitation) can be optimized for high strength and resistance to grain boundary damage propagation during creep-fatigue at 550 °C. Furthermore, the shorter creep-fatigue life at 650 °C in the condition aged at 650 °C for 500 h indicates that the optimal microstructure for creep-fatigue performance may not be the same at both testing temperatures. Further microstructural evaluation is necessary to support the hypothesized differences in creep-fatigue behavior between the two aged microstructures. Microstructural characterization and fractography have not been carried out on the failed creep-fatigue tests from the 650 °C, 500 h aged microstructure.
Figure B.4 (a) Friction stress, $\sigma_f$, and (b) back stress, $\sigma_b$, for the aged and SA microstructures tested at 550 and 650 °C.

Figure B.5 (a) Cyclic plastic strain, $\varepsilon_{ap,cyc}$, and (b) creep strain, $\varepsilon_{cr}$, for the aged and SA microstructures tested at 550 and 650 °C.
APPENDIX C
STRESS RELAXATION DATA

Mid-life stress relaxation data for all temperatures tested in creep-fatigue with a solution annealed microstructure and a 30 min hold time are shown in Figure C.1. Using a non-linear solver, the stress relaxation data is fit to the following relation

$$\sigma = a[b + t]^c$$  \hspace{1cm} C.1

as discussed in Chapter 3. The fitting constants, $a$, $b$, and $c$ for each temperature at the mid-life cycle are shown in Table C.1. These values can be used, along with LCF test data and the analysis presented in Appendix A, to determine the individual damage components during creep-fatigue at each temperature. The damage components at each temperature can be added to the damage diagram for solution annealed Alloy 709, shown in Appendix A.

Figure C.1 Stress relaxation data during the 30 min tensile hold of the approximate mid-life cycle for creep-fatigue tests at 500, 550, 600, 650, and 700 °C with solution annealed material.

The mid-life stress relaxation data and fit curves for creep-fatigue tests at 550 and 650 °C with specimens aged at 750 °C for 100 h are shown in Figure C.2, with the stress relaxation data from the solution annealed microstructure tested at the same conditions for reference.
Table C.1 Constants for Mid-Life Stress Relaxation Function from Solution Annealed Material

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Heat ID</th>
<th>Approximate Mid-Life Cycle</th>
<th>a</th>
<th>b</th>
<th>c</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>58776</td>
<td>900</td>
<td>441.29</td>
<td>551.4</td>
<td>-0.0110</td>
</tr>
<tr>
<td>550</td>
<td>011594</td>
<td>500</td>
<td>435.38</td>
<td>167.04</td>
<td>-0.0165</td>
</tr>
<tr>
<td>550</td>
<td>011593</td>
<td>500</td>
<td>434.50</td>
<td>62.11</td>
<td>-0.0147</td>
</tr>
<tr>
<td>600</td>
<td>58776</td>
<td>150</td>
<td>409.35</td>
<td>6.586</td>
<td>-0.0252</td>
</tr>
<tr>
<td>650</td>
<td>011594</td>
<td>500</td>
<td>362.37</td>
<td>13.29</td>
<td>-0.0778</td>
</tr>
<tr>
<td>650</td>
<td>011593</td>
<td>500</td>
<td>364.03</td>
<td>9.78</td>
<td>-0.0782</td>
</tr>
<tr>
<td>700</td>
<td>58776</td>
<td>300</td>
<td>334.22</td>
<td>3.73</td>
<td>-0.1160</td>
</tr>
</tbody>
</table>

Table C.2 Constants for Mid-Life Stress Relaxation Function from Material Aged at 750 °C for 100 h

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Heat ID</th>
<th>Approximate Mid-Life Cycle</th>
<th>a</th>
<th>b</th>
<th>c</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>58776</td>
<td>1500</td>
<td>329.94</td>
<td>127.98</td>
<td>-0.0178</td>
</tr>
<tr>
<td>650</td>
<td>58776</td>
<td>700</td>
<td>344.76</td>
<td>16.09</td>
<td>-0.0906</td>
</tr>
</tbody>
</table>

Figure C.2 Stress relaxation data during the 30 min tensile hold of the approximate mid-life cycle for creep-fatigue tests at 550 and 650 °C with specimens aged at 750 °C for 100 h. Solution annealed mid-life stress relaxation data from tests at 550 and 650 °C are also shown.
All of the following MATLAB® scripts import a .csv file of the “specimen” data from a creep-fatigue test which contains nine columns: run time (sec), bottom thermocouple value (°C), top thermocouple value (°C), axial displacement (mm), axial force (N), engineering strain (mm/mm), axial segment count, engineering stress (MPa), and cycle count.

D.1 Stress Relaxation Curve Fitting Program

The following MATLAB® script extracts the stress relaxation data from each cycle where it is available, uses a non-linear solver to fit the data to the relation discussed in Chapter 3, and exports a .csv file containing the constants that describe the relaxation behavior. The script contains an option to fit the stress relaxation data for every cycle, or for an individual specified cycle. The creep-rate exponent, \( n \), is also determined and exported, if desired. Finally, the strain rates during relaxation of a specified cycle can be determined, if desired. Note that a line beginning with ‘%%’ marks the beginning of each sub program described above.

```matlab
%% Read in data and initialize arrays
clear
format long
data = csvread('CF650_1.csv', 0, 0); %filename here
cols = [1, 7, 8, 6, 9];
data = data(:, cols); %Data has [time, step, stress, strain, cycle]

% Initialize banks
steps = unique(data(:,2));
coeffs = [];

%% This step loops through all relaxation data
% Coefficients for constitutive model are fit and stored for each relaxation
% cycle. Then the creep stress exponent, \( n \), is determined and stored.
% No plotting in this step.

% First, cycle through the actual cycle counts
for count = 1:length(steps)
    % make sure cycle step is odd, where relaxation occurs
```
cyc = steps(count);
if mod(cyc,2) == 1

% extract subset of odd-step data, from zero strain to end of rlx
    idx = find(data(:,2)==cyc);
    subset = data(idx,:);

% preallocate array for relevant strain data
    goodstuff = [];

% Now iterate through subset for relevant part (strain>=0.005)
    for subrow = 1:length(idx)
        % look for change in timestep
        if ((subset(subrow+5,1)-subset(subrow,1)) > 1.5)
            goodstuff(:,1) = subset(subrow:end,:);
            break
        end
    end

%---->This is where the curve fitting happens.....
    xdata=[];
    ydata=[];
    % Reinitialize time to zero and store data in xdata
    for i = 1:length(goodstuff(:,1))
        xdata(end+1,1) = goodstuff(i,1) - goodstuff(1,1);
    end
    ydata=goodstuff(:,3); %stress in ydata
    fun = @(x,xdata)x(1).*((x(2)+xdata).^x(3)); %define function
    x0 = [400,20,-0.02]; %initial guess
    lb = [0,0,-.15]; %lower bound
    ub = [700,700,0]; %lower bound
    x = lsqcurvefit(fun,x0,xdata,ydata,lb,ub); %curve fit with data
    %store coefficients and cycle number
    coeffs(end+1, 1:4) = zeros(1,4);
    coeffs(end,1) = (cyc+1)/2;
    coeffs(end, 2:4) = [x(1),x(2),x(3)];
end
end

% This step loops through ONLY A SPECIFIC relaxation cycle and plots
% Coefficients for constitutive model are fit and stored for each relaxation
% cycle. Then the creep stress exponent, n, is determined and stored.

% ENTER DESIRED CYCLE HERE
rlx_cycle = 500;

% cycle through the actual cycle counts
for count = 1:length(steps)
    % make sure cycle step is odd, where relaxation occurs
    cyc = steps(count);
    if mod(cyc, 2) == 1
        % extract subset of odd-step data, from zero strain to end of rlx
        idx = find(data(:,2)==cyc);
        subset = data(idx,:);

        % preallocate array for relevant strain data
        goodstuff = []; % Only store data and fit for the specific cycle
        if ((cyc+1)/2 == rlx_cycle)
            % Now iterate through subset for relevant part (strain>=0.005)
            for subrow = 1:length(idx)
                % look for change in timestep
                if ((subset(subrow+5,1)-subset(subrow,1)) > 1.5)
                    goodstuff(:,,:) = subset(subrow:end,:);
                    break
                end
            end
        end

        % This is where the CURVE FITTING happens.....
        xdata=[];
        ydata=[];
        % Reinitialize time to zero and store data in xdata
        for i = 1:length(goodstuff(:,1))
            xdata(end+1,1) = goodstuff(i,1) - goodstuff(1,1);
        end
        ydata=goodstuff(:,3); %stress in ydata
        fun = @(x,xdata)x(1).*(x(2)+xdata).^x(3); %define function
        x0 = [400,20,-0.02]; %initial guess
        lb = [0,0,-.15]; %lower bound
        ub = [700,700,0]; %upper bound
        x = lsqcurvefit(fun,x0,xdata,ydata,lb,ub); %curve fit with data
% store coefficients and cycle number
coeffs(end+1, 1:4) = zeros(1,4);
coeffs(end, 1) = (cyc+1)/2;
coeffs(end, 2:4) = [x(1), x(2), x(3)];

% plot curve fit with data for specific cycle
plot(xdata, ydata, 'ko', xdata, fun(x, xdata), 'b-');
hold on
cycle = (cyc+1)/2
n = 1-1/x(3)
x(1) % a
x(2) % b
x(3) % c
break
end
end

%% Solve for n and export data
% Run the non-cycle specific fitting segment first
for i = 1:length(coeffs)
    coeffs(i,5) = (1-1./coeffs(i,4));
end
csvwrite('CF650_rlxcoeffs.txt', coeffs);

%% Solve for relaxation strain-rates of a specific cycle
% Run the first stress relaxation fitting segment (not cycle specific)

% specify cycle number 1-101, 150-151, 200-201, 250-251....
cycleNum = 300;
indx = find(coeffs(:,1)==cycleNum);
stress = [];
strainrate = [];

% Uncomment appropriate elastic modulus
E = 144900; % MPa - Youngs mod at 700C
E = 151000; % MPa - Youngs mod at 650C
E = 156000; % MPa - Youngs mod at 600C
E = 155200; % MPa - Youngs mod at 550C
E = 158700; % MPa - Youngs mod at 500C

% Pull fit coeffs for specified cycle
bo = coeffs(indx, 2);
to = coeffs(indx, 3);
b1 = coeffs(indx, 4);

% Calculate strain rate for each timestep in relaxation cycle
for t = 1:1800
    stress(end+1) = bo*(t+to)^b1;
    strainrate(end+1) = -1*(bo*b1)/E*(t+to)^(b1-1);
end

% Plot stress v. strain-rate
loglog(stress, strainrate, 'bo')
hold on

D.2 Hysteresis Loop Partition Analysis Program

The following MATLAB® script loops through each available hysteresis loop and
determines the plastic strain, creep strain, and cyclic plastic strain. Additionally, the critical
strain for serrations is determined for each hysteresis loop as the strain amplitude where the
stress drop is greater than a specified value (e.g. 0.5 MPa). The tensile stress and strain data is
extracted from each hysteresis loop and fit to a Ramberg-Osgood relation to determine the cyclic
yield stress, friction stress, and back stress according to the procedure described in Chapter 3.
Finally, work-hardening rates are determined from the Ramberg-Osgood fit for any specified
cycle and plotted against the tensile stress versus strain. Three .csv files are exported: one with
the critical strain data, one with the friction and back stress data, and the other the work-
hardening data.

%% Read in data and initialize arrays
clear
format long
data = csvread('CF650_1.csv', 0, 0); %Enter *specimen* file name
here
cols = [7, 8, 6, 9]; %[step, stress, strain, cycle]
data = data(:, cols); %Data has [step, stress, strain, cycle]

%Initialize banks
cycles = unique(data(:, 4));
[m, n] = size(data);
coeffs = [];
WH = [];
hstress = [];
hstrain = [];
crit_strain = [];
%Loop through each cycle
for loop = 1:length(cycles)
    % --Pull Individual Hysteresis Loops-- %
    % reininitialize the stress and strain data each time
    hstress = [];
    hstrain = [];
    % look through all data and pull stress and strain for valid cycle
    for row = 1:m-1
        if data(row,1) == (cycles(loop)*2)
            hstress(end+1)=data(row,2);
            hstrain(end+1)=data(row,3);
        end
        if data(row,1) == (cycles(loop)*2+1)
            hstress(end+1)=data(row,2);
            hstrain(end+1)=data(row,3);
        end
    end
    % if cycle does not have data (e.g. 125) go to next iteration of main
    % for loop without doing anything else
    if length(hstress) == 0
        continue
    end

    % Fill array with cycle number, stress, and strain
    hyst_data = [hstress; hstrain];
    hyst_data = hyst_data';

    % --Determine critical strain for serrations here-- %
    drop_flag = 0; % flag for stress drop
    drop_start = 0; % index for start of stress drop
    drop_end = 0; % index for end of stress drop
    drop_mag = 0; % magnitude of stress drop
    drop_strain = 0; % strain at first significant drop
    idx_1 = 0; % index for strain sign change 1
    idx_2 = 0; % index for strain sign change 2

    for k = 1:(length(hyst_data)-1)
        % first look for strain sign change to calculate plastic strain
        if sign(hyst_data(k+1,1)) ~= sign(hyst_data(k,1))
            if hyst_data(k+1,1) < 0 % first sign change will be + to -
                idx_1 = k;
            end
        end
    end
else idx_2 = k;
end
end
% then determine where load drop occurs
% positive stress in tensile half
if hyst_data(k,1) > 0 &&
(hyst_data(k+1,2)>hyst_data(k,2))
% look for initial load drop
if (hyst_data(k+1,1) < hyst_data(k,1)) &&
drop_flag==0
    drop_start = k;
    drop_end = k+1;
    drop_flag = 1;
end
% if load is still dropping...
if (hyst_data(k+1,1) < hyst_data(k,1)) &&
drop_flag==1
    drop_end = k+1;
end
% if load is done dropping...
if (hyst_data(k+1,1) > hyst_data(k,1)) &&
drop_flag==1
    % magnitude of the stress drop
    drop_mag = hyst_data(drop_start,1) -
               hyst_data(drop_end,1);
    if drop_mag > 0.5 % check if the drop is
greater than 0.5 MPa
        % serrations start at the end of the
        % first sig drop
        drop_strain = hyst_data(drop_end,2);
        break;
    else
        drop_flag = 0; % reset flag
    end
end
end
end

% Plot hysteresis loop - UNCOMMENT FOR DEBUGGING!!
%------------------%
plot(hstrain, hstress, '-k')
hold on
% plot first stress drop
plot(hyst_data(drop_end,2),hyst_data(drop_end,1),'bo')
disp(cycles(loop))
pause;
clf;
%------------------% 

% --Now calculate plastic strain, creep strain, and critical strain for
% the given cycle-- %
pl_strain = ((hyst_data(idx_1+1,2)+hyst_data(idx_1,2))/2)- ((hyst_data(idx_2+1,2)+hyst_data(idx_2,2))/2);
stress_rlx = max(hyst_data(:,1)) - hyst_data(length(hyst_data)-1,1); %max stress minus stress at end of hold
unload_mod = (hyst_data(1,1)-hyst_data(10,1))/(hyst_data(1,2)-hyst_data(10,2)); %determine unloading modulus for first 10 pts
cr_strain = stress_rlx/unload_mod; %determine creep strain
cyc_pl_strain = pl_strain - cr_strain; %cyclic plastic strain

crit_strain(end+1, 1:7) = zeros(1,7);
crit_strain(end,1) = cycles(loop);
crit_strain(end,2) = pl_strain;
crit_strain(end,3) = stress_rlx;
crit_strain(end,4) = unload_mod;
crit_strain(end,5) = cr_strain;
crit_strain(end,6) = cyc_pl_strain;
crit_strain(end,7) = (cyc_pl_strain - (0.005 - drop_strain));

% --Now determine where strain reversal occurs-- %
min_strn = min(hyst_data(:,2)); %find and store min strain (at reversal)
rev_idx = find(hyst_data(:,2) == min_strn); %find index of min strain
min_strs = hyst_data(rev_idx,1); %store min stress (at reversal)
tens_strs = []; %array of stress values after reversal
tens_strn = []; %array of strain values after reversal
% loop through hyst_data and pull only tensile data up to hold
for j = rev_idx:(length(hyst_data)-1)
    if(hyst_data(j,2) < 0.0048)
        tens_strs(end+1) = hyst_data(j,1);
        tens_strn(end+1) = hyst_data(j,2);
    else
        break;
    end
end

% Plot tensile part of hysteresis loop only - UNCOMMENT FOR DEBUGGING!!%
%----------------%
plot(tens_strn, tens_strs, 'ko')
pause;
clf;
%----------------%

% -- Now determine linear portion of data and effective modulus %
% compare first several modulii (2-1) v. (3-2) v. (4-3) v. (5-4)
E_21 = (tens_strs(2)-tens_strs(1))/(tens_strn(2) - tens_strn(1));
E_32 = (tens_strs(3)-tens_strs(2))/(tens_strn(3) - tens_strn(2));
E_43 = (tens_strs(4)-tens_strs(3))/(tens_strn(4) - tens_strn(3));
E_54 = (tens_strs(5)-tens_strs(4))/(tens_strn(5) - tens_strn(4));

vis_idx = 0; % index for viscous stress
if abs(E_32 - E_21) > 1e5
    vis_idx = 1;
end
if abs(E_43 - E_32) > 1e5
    vis_idx = 2;
end
if abs(E_54 - E_43) > 1e5
    vis_idx = 3;
end

%delete non-linear first points - remove viscous stress
if vis_idx > 0
    for k=1:vis_idx
        tens_strn(:,k) = [];
        tens_strs(:,k) = [];
    end
end

%now determine minimum stress and strain
min_strn = tens_strn(1);
min_strs = tens_strs(1);
%zero stress and strain data
tens_strn = tens_strn - min_strn;
tens_strs = tens_strs - min_strs;

%store max stress amplitude
max_strs = max(tens_strs);

% --Now fit stress strain data to Ramberg-Osgood relation--
% E_eff = 0;
%define effective modulus from first 0.15% strain
for lin_idx = 1:length(tens_strn) - 1
    if tens_strn(lin_idx) > 0.0015
        break
    end
end
E_eff = tens_strs(lin_idx)/tens_strn(lin_idx); %effective mod

%define function, provide guess, and execute curvefit
fun = @(y,tens_strs) tens_strs./E_eff+(tens_strs./y(1)).^(y(2));
y0 = [1500,6];
y=lsqcurvefit(fun,y0,tens_strs,tens_strn);
H = real(y(1)); %H' in R-O relation
n_pr = 1/real(y(2)); %n' in R-O relation

%plot data and fit curve together - UNCOMMENT FOR DEBUGGING!!%
%-----------------
plot(tens_strn,tens_strs,'bo',fun(real(y),tens_strs),tens_strs,'-k')
offset = 0.001; %define plastic strain offset for yield stress

%determine yield stress at offset from curve fit variables
fl_strs = H.*(offset).^(n_pr); %flow stress at offset

% --Add cycle number, coefficients, and flow stress to array-- %
%coeffs contains
[cycles,H',1/n',n',fl_strs,E_eff,max_stress,sig_f,sig_b]

coeffs(end+1, 1:9) = zeros(1,9);
coeffs(end,1) = cycles(loop);
coeffs(end, 2:4) = [H,1/n_pr,n_pr];
coeffs(end, 5) = fl_strs;
coeffs(end, 6) = E_eff;
coeffs(end, 7) = max_strs;
coeffs(end, 8) = (fl_strs)/2; %friction stress
coeffs(end, 9) = (max_strs-fl_strs)/2; %back stress

%store work-hardening values at beginning and end of
tensile plasticity
WH(end+1, 1:3) = zeros(1,3);
WH(end,1) = cycles(loop);
WH(end,2) = n_pr*H*offset^(n_pr-1);
WH(end,3) = n_pr*H*(0.01)^(n_pr-1);
end

% --Output Data to a .CSV file-- %
csvwrite('CF650_1_stress_partition.txt',coeffs);
csvwrite('CF650_1_WHcycles.txt',WH); % store the range of WH rates

% Work hardening for specific cycles
%plot work-hardening rate versus plastic strain
wh_cycle = 500;
cyc_idx = find(coeffs(:,1) == wh_cycle)
H=coeffs(cyc_idx,2);
n_pr=coeffs(cyc_idx,4);
max_strs=coeffs(cyc_idx,8);
E_eff=coeffs(cyc_idx,7);
yield=coeffs(cyc_idx,5);
tensile_stress = 0:10:max_strs;
plastic_strain = (yield/E_eff+.001):.0001:.01;

stress_strain =
tensile_stress./E_eff+(tensile_stress./H).^(1/n_pr);

work_hardening = n_pr.*H.*plastic_strain.^(n_pr-1);

plotyy(stress_strain, tensile_stress, plastic_strain, work_hardening)

hold on
plot((yield/E_eff+.001), yield,'ko')
hold on
APPENDIX E

TABULATED HARDNESS AND DISLOCATION DENSITY DATA

This appendix contains tabulated values and standard deviations of room temperature Vickers hardness and dislocation density for all conditions measured in this study.

Table E.1 Vickers Room Temperature Hardness Values for Solution Annealed Material

<table>
<thead>
<tr>
<th>Heat ID</th>
<th>Vickers Hardness (HV)</th>
<th>Standard Deviation (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>011593</td>
<td>193.4</td>
<td>5.7</td>
</tr>
<tr>
<td>011594</td>
<td>175.5</td>
<td>3.8</td>
</tr>
</tbody>
</table>

Table E.2 Vickers Room Temperature Hardness Values for Static Aged Material from Heat 011594

<table>
<thead>
<tr>
<th>Condition</th>
<th>Vickers Hardness (HV)</th>
<th>Standard Deviation (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>550 °C, 20 h</td>
<td>173.1</td>
<td>4.3</td>
</tr>
<tr>
<td>550 °C, 500 h</td>
<td>178.4</td>
<td>3.7</td>
</tr>
<tr>
<td>650 °C, 10 h</td>
<td>174.5</td>
<td>4.1</td>
</tr>
<tr>
<td>650 °C, 500 h</td>
<td>183.0</td>
<td>5.7</td>
</tr>
<tr>
<td>750 °C, 100 h</td>
<td>197.7</td>
<td>4.2</td>
</tr>
<tr>
<td>750 °C, 200 h</td>
<td>195.9</td>
<td>4.0</td>
</tr>
</tbody>
</table>

Table E.3 Vickers Room Temperature Hardness Values for Failed LCF and Creep-Fatigue Tests from Solution Annealed Material

<table>
<thead>
<tr>
<th>Condition</th>
<th>Heat ID</th>
<th>Vickers Hardness (HV)</th>
<th>Standard Deviation (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>550 °C, LCF</td>
<td>011594</td>
<td>256.8</td>
<td>7.8</td>
</tr>
<tr>
<td>650 °C, LCF</td>
<td>011594</td>
<td>239.5</td>
<td>8.0</td>
</tr>
<tr>
<td>500 °C, Creep-Fatigue (30 min hold)</td>
<td>58776</td>
<td>268.0</td>
<td>8.7</td>
</tr>
<tr>
<td>550 °C, Creep-Fatigue (30 min hold)</td>
<td>011594</td>
<td>268.2</td>
<td>7.9</td>
</tr>
<tr>
<td>600 °C, Creep-Fatigue (30 min hold)</td>
<td>58776</td>
<td>257.1</td>
<td>9.4</td>
</tr>
<tr>
<td>650 °C, Creep-Fatigue (30 min hold)</td>
<td>011594</td>
<td>234.7</td>
<td>11.5</td>
</tr>
<tr>
<td>700 °C, Creep-Fatigue (30 min hold)</td>
<td>58776</td>
<td>215.1</td>
<td>5.7</td>
</tr>
</tbody>
</table>
Table E.4 Vickers Room Temperature Hardness Values for Interrupted Creep-Fatigue Tests (30 min Holds) from Solution Annealed Heat 011594

<table>
<thead>
<tr>
<th>Condition</th>
<th>Vickers Hardness (HV)</th>
<th>Standard Deviation (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>550 °C, 50 cycles</td>
<td>237.2</td>
<td>6.1</td>
</tr>
<tr>
<td>550 °C, 400 cycles</td>
<td>264.2</td>
<td>7.2</td>
</tr>
<tr>
<td>550 °C, 600 cycles</td>
<td>264.2</td>
<td>9.6</td>
</tr>
<tr>
<td>650 °C, 25 cycles</td>
<td>226.7</td>
<td>6.7</td>
</tr>
<tr>
<td>650 °C, 50 cycles</td>
<td>241.1</td>
<td>7.7</td>
</tr>
<tr>
<td>650 °C, 400 cycles</td>
<td>238.6</td>
<td>8.4</td>
</tr>
</tbody>
</table>

Table E.5 Vickers Room Temperature Hardness Values for Failed Creep-Fatigue Tests (30 min Holds) from Pre-Aged Heat 58776

<table>
<thead>
<tr>
<th>Condition</th>
<th>Vickers Hardness (HV)</th>
<th>Standard Deviation (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>550 °C, Aged at 750 °C for 100 h</td>
<td>221.6</td>
<td>6.7</td>
</tr>
<tr>
<td>650 °C, Aged at 750 °C for 100 h</td>
<td>202.0</td>
<td>5.9</td>
</tr>
</tbody>
</table>

Table E.6 Dislocation Density Values for Failed LCF and Creep-Fatigue Tests from Solution Annealed Material

<table>
<thead>
<tr>
<th>Condition</th>
<th>Heat ID</th>
<th>Dislocation Density (m⁻²)</th>
<th>Standard Deviation (m⁻²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>550 °C, LCF</td>
<td>011594</td>
<td>1.31×10¹⁴</td>
<td>2.28×10¹³</td>
</tr>
<tr>
<td>650 °C, LCF</td>
<td>011594</td>
<td>4.44×10¹³</td>
<td>7.21×10¹²</td>
</tr>
<tr>
<td>500 °C, Creep-Fatigue (30 min hold)</td>
<td>58776</td>
<td>1.28×10¹⁴</td>
<td>3.13×10¹³</td>
</tr>
<tr>
<td>550 °C, Creep-Fatigue (30 min hold)</td>
<td>011594</td>
<td>1.46×10¹⁴</td>
<td>3.18×10¹³</td>
</tr>
<tr>
<td>600 °C, Creep-Fatigue (30 min hold)</td>
<td>58776</td>
<td>1.54×10¹⁴</td>
<td>9.67×10¹²</td>
</tr>
<tr>
<td>650 °C, Creep-Fatigue (30 min hold)</td>
<td>011594</td>
<td>7.91×10¹³</td>
<td>1.83×10¹³</td>
</tr>
<tr>
<td>700 °C, Creep-Fatigue (30 min hold)</td>
<td>58776</td>
<td>3.48×10¹³</td>
<td>4.18×10¹²</td>
</tr>
</tbody>
</table>

Table E.7 Dislocation Density Values for Interrupted Creep-Fatigue Tests (30 min Holds) from Solution Annealed Heat 011594

<table>
<thead>
<tr>
<th>Condition</th>
<th>Dislocation Density (m⁻²)</th>
<th>Standard Deviation (m⁻²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>550 °C, 50 cycles</td>
<td>1.55×10¹⁴</td>
<td>2.34×10¹³</td>
</tr>
<tr>
<td>550 °C, 400 cycles</td>
<td>1.58×10¹⁴</td>
<td>3.95×10¹²</td>
</tr>
<tr>
<td>550 °C, 600 cycles</td>
<td>1.78×10¹⁴</td>
<td>4.19×10¹²</td>
</tr>
<tr>
<td>650 °C, 25 cycles</td>
<td>5.84×10¹³</td>
<td>1.89×10¹³</td>
</tr>
<tr>
<td>650 °C, 50 cycles</td>
<td>1.58×10¹⁴</td>
<td>7.89×10¹²</td>
</tr>
<tr>
<td>650 °C, 400 cycles</td>
<td>7.33×10¹³</td>
<td>4.03×10¹²</td>
</tr>
</tbody>
</table>
Table E.8 Dislocation Density Values for Failed Creep-Fatigue Tests (30 min Holds) from Pre-Aged Heat 58776

<table>
<thead>
<tr>
<th>Condition</th>
<th>Dislocation Density (m$^2$)</th>
<th>Standard Deviation (m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>550 °C, Aged at 750 °C for 100 h</td>
<td>4.31×10$^{13}$</td>
<td>4.50×10$^{12}$</td>
</tr>
<tr>
<td>650 °C, Aged at 750 °C for 100 h</td>
<td>3.44×10$^{13}$</td>
<td>4.79×10$^{12}$</td>
</tr>
</tbody>
</table>