To the Graduate Council:

I am submitting herewith a dissertation written by Hongbo Tian entitled “Effects of Environment and Frequency on the Fatigue Behavior of the Spallation Neutron Source (SNS) Target Container Material - 316 LN Stainless Steel.” I have examined the final electronic copy of this dissertation for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Doctor of Philosophy, with a major in Materials Science and Engineering.

Dr. Peter K. Liaw

Major Professor

We have read this dissertation and recommend its acceptance:

Dr. Raymond A. Buchanan

Dr. John D. Landes

Dr. Louis K. Mansur

Accepted for the Council:

Dr. Anne Mayhew

Vice Provost and Dean of Graduate Studies

(Original signatures are on file with official student records.)
Effects of Environment and Frequency on the Fatigue Behavior of the Spallation Neutron Source (SNS) Target Container Material - 316 LN Stainless Steel

A Dissertation

Presented for the

Doctor of Philosophy

Degree

The University of Tennessee, Knoxville

Hongbo Tian

December 2003
DEDICATION

This dissertation is dedicated to my wife

Mrs. Jing He

and

My parents

Mr. Fu Tian and Mrs. Yanshu Ding

and

My younger brother

Mr. Hongliang Tian

For their love
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ABSTRACT

As the candidate target container material of the new Spallation Neutron Source (SNS) being designed and constructed at the Oak Ridge National Laboratory (ORNL), 316 low-carbon nitrogen-added (LN) stainless steel (SS) will operate in an aggressive environment, subjected to intense fluxes of high-energy protons and neutrons while exposed to liquid mercury. The SNS is an accelerator-based neutron source that provides pulsed beams of spallation neutrons by bombarding a mercury target with 1 GeV protons. The function of the SNS is to convert a short pulse (< 1 µs, 60 Hz, 17 kJ/pulse), high-average-power (1 MW), 1-GeV proton beam into 18 lower-energy (< 1 eV), short-pulsed (~ tens of µs) neutron beams optimized for use by neutron-scattering instruments.

The current project is oriented toward materials studies regarding the effects of test environment and frequency on the fatigue behavior of 316 LN SS. Class 316 LN SS is a low-carbon, nitrogen-added austenitic stainless steel, which possesses excellent resistance to both wear and corrosion, and is widely used in the nuclear industry. However, this material hasn’t been systematically investigated for its feasibility in the Spallation Neutron Source with a mercury target. In order to study the structural applications of this material and improve the fundamental understanding of the fatigue damage mechanisms, fatigue tests were performed in air and mercury environments at various frequencies and R ratios (R = σ_min/σ_max, σ_min and σ_max are the applied minimum and maximum stresses, respectively).
Fatigue data were developed for the structural design and engineering applications of this material. Specifically, high-cycle fatigue tests, fatigue crack-propagation tests, and ultrahigh cycle fatigue tests up to $10^9$ cycles were conducted in air and mercury with test frequencies from 10 Hz to 700 Hz. Microstructure characterizations were performed by optical microscopy (OM), scanning-electron microscopy (SEM), and transmission-electron microscopy (TEM). Fractographic studies characterized the crack-initiation and propagation behavior of the alloy. It was found that mercury doesn’t seem to have a large impact on the crack-initiation behavior of 316 LN SS. However, the crack-propagation mechanisms in air and mercury are different in some test conditions. Transgranular cracks seem to be the main mechanism in air, and intergranular in mercury.

A detailed study on the dislocation structure of 316 LN SS after fatigue was performed, parallel to a collaborative work on the residual-stress evolution of the material using the neutron-scattering technique with researchers ORNL. The study showed that most dislocations in 316 LN SS after high-cycle fatigue are of an edge type, which corresponds to the result of the theoretical calculation by researchers at ORNL.

A significant specimen self-heating effect was found during high-cycle fatigue. Theoretical calculation was performed to predict temperature responses of the material subjected to cyclic deformation. The predicted cyclic temperature evolution seems to be in good agreement with the experimental results.
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PART 1

INTRODUCTION
Type 316 stainless steel (SS) has been widely used in the nuclear industry, because of its excellent ductility, corrosion resistance, and irradiation performance. It’s often used over a wide temperature range, and employed in components that are loaded under severe conditions (stress, strain cycling, and thermal cycling). Specifically, 316 LN (low-carbon, nitrogen-containing) SS was selected as a candidate container material for the mercury target of the Spallation Neutron Source (SNS) at the Oak Ridge National Laboratory (ORNL). The high-energy proton beam is produced from the ion source, and hits the target located in the stainless-steel container (Figure 1-1). The target module includes the mercury vessel, the water-cooled shroud, and seals mounted on a flange that form the interface to the core vessel and process piping. A detailed description of the target module can be found in the literature [1, 2]. The mercury vessel and the shroud can be seen as a four-walled structure with three channels. From the innermost to the outermost, there are mercury, helium, and water channels. The mercury target container has a width of 404 mm, a height of 104 mm, and a length of 774.5 mm. The bulk mercury flow enters from two regions outside the beam profile and returns through the 224 mm x 80 mm center portion of the vessel (Figures 1-2).

Because of the enormous amount of energy that the short, powerful pulses of the incoming 1-GeV proton beam will deposit in the spallation target, it was decided to use a liquid mercury target rather than a solid target, such as tantalum or tungsten. The SNS will be the first scientific facility to use pure mercury as a target for a proton beam. Mercury was chosen to be the target material due to several reasons: (1) it is not damaged
by radiation, as are solids; (2) it has a high atomic number, making it a source of numerous neutrons (the average mercury nucleus has 120 neutrons and 80 protons); and (3) because it is liquid at room temperature, it is better able than a solid target to dissipate the large, rapid rise in the temperature and withstand the shock effects arising from the rapid high-energy pulses.

High-power SNS will place exceptional demands on materials performance. The satisfactory performance of materials for sufficiently long-time periods will determine the viability of the target station. Components that require special considerations include the mercury vessel and shroud, beam windows, support structures and plumbing, moderator vessels, and beam tubes, for example. Being a core component of the SNS facilities, the target container needs the maximum attention in terms of safety.

The interaction of the energetic proton beam with the mercury target leads to very high heating rates in the target during each pulse. Although the resulting temperature rise during a single pulse is relatively small (a few °C), the rate of temperature rise is enormous (~10^7 °C/s) during the very brief beam pulse (0.5 µs). The pulsing will create pressure waves by rapid heating in the mercury and cause stress transients in the target container. The stress mode on the container varies at different parts of the container. Two of the most common loading conditions are the tension-compression and tension-tension stress modes.
It is desirable to assess whether the frequency effect and liquid-metal embrittlement (LME) can occur and affect mechanical characteristics of the alloy in contact with mercury, and to understand and characterize the key processes that control mercury-environment crack initiation and propagation behaviors of target materials.

Most of the investigations on the frequency effect have been done at relatively low frequencies using strain-controlled fatigue tests, and those studies at high frequencies using ultrasonic fatigue machines have been focusing on the problem of elastic cyclic loading. The typical specimens used in the study of the frequency effect on the fatigue life are compact-tension samples, and relatively few investigations have been performed using smooth-bar specimens. In addition, a variety of materials were employed in the study of the frequency effect including stainless steel, but there is almost no information about the influence of test frequency on the fatigue life of 316 LN SS, which is the critical candidate material of the SNS target container where the frequency effect of pressure waves on the container is of great concern. In addition, there is little information on fatigue behavior of structural materials, including 316 LN SS, in mercury environments.

In the present study, the dominant mechanisms, which controlled the fatigue behavior of 316 LN SS, was characterized by electron-microscopy techniques. Theoretical calculation, based on the understanding of the relationship between the fatigue process
and heat transfer-theory, was attempted. The predicted cyclic temperature variations were found to be in good agreement with the experimental results.

The study on the environmental effects, frequency effects, microstructure, and their correlations with fatigue behavior, and theoretical calculations are discussed from PARTS 1 to 10. In PART 2, the background information on the microstructural characterization, mechanistic understanding, and theoretical modeling of pertinent materials are summarized, and the critical issues of the fatigue behavior of 316 LN SS are addressed. In PART 3, the experimental procedures are detailed. In PART 4, the environmental effects on the fatigue behavior of 316 LN SS are discussed. In PART 5, the temperature evolution during high-cycle fatigue is illustrated. In PART 6, the frequency effects on the fatigue behavior of 316 LN SS are studied. In PART 7, theoretical calculations are constructed to predict the temperature evolution during fatigue. In PART 8, the characterizations on the dislocation structure after fatigue were presented. The conclusions and future work are given in PARTs 9 and 10, respectively.
PART 2

LITERATURE REVIEW
2.1. Introduction

High-power spallation neutron source (SNS) will place exceptional demands on materials performance.\textsuperscript{[1]} The satisfactory performance of materials for sufficiently long time periods will determine the viability of the target station. Components that require special consideration include the mercury vessel and shroud, beam windows, support structures and plumbing, moderator vessels, and beam tubes, for example. An austenitic stainless steel of Type 316 has been selected as the mercury target vessel material. The materials R&D program for the SNS is oriented toward materials qualification: informed selection of materials based on the existing experimental data and analyses, testing in actual and simulated application environments, lifetime estimates for the SNS environment, and iteration and optimization of properties to improve performance. The SNS is expected to operate in a pulsed mode at a main frequency of 60 Hz, which will drive pressure waves that call attention to fatigue responses of the vessel. However, most mechanical data on Type 316 stainless steel has been accumulated in steady conditions, and relatively less information for fatigue loading conditions is available. There is almost no information on fatigue in mercury environments. Therefore, research work of fatigue tests in mercury is necessary. The main issues in the research are effects of test frequency, temperature, and liquid metal embrittlement (LME) on fatigue behavior of 316 LN SS.

The target material for the bombardment of a proton beam will be liquid mercury (Hg) contained in the 316 LN SS container. The function of the neutron-source system is to
convert a short-pulsed, high-power high-energy proton beam into a short-pulsed, low-energy neutron beam optimized for use in neutron-scattering instruments.\textsuperscript{[1]} The container will be subjected to irradiation by both protons and neutrons, as well as to cyclic pressure waves produced by pulse-induced rapid heating of the contained mercury. Therefore, an investigation of the fatigue behavior of 316 LN SS in the mercury environment is necessary to assure the integrity of target-container materials. In particular, it is desirable to assess whether the phenomenon of LME can occur and affect the mechanical behavior of the alloy in contact with mercury.
2.2. Critical Issues in the Selection of Target Container Materials

Since the target container module is subjected to the most severe working conditions in terms of radiation damage, stress, and contact with mercury, many critical issues have to be addressed for the target container material. These issues can be discussed in several areas, including radiation effects, cavitation erosion, and compatibility with mercury.

**Radiation Damage**

Typically fission reactors are steady-state devices, where the power levels, and, hence, the displacement-damage rate usually exhibits only gradual variations with time. The SNS will be pulsed, with a proton beam pulse length of \(~0.7\ \mu\text{s}\) and a repetition rate of 60 Hz. Thus, while the time-averaged damage rate in the SNS target container will be similar to that in a high-flux fission reactor core or in a magnetically confined fusion power-reactor first wall, the instantaneous damage rate during a proton pulse will be about four orders of magnitude higher \((10^{-2} \text{ vs } 10^{-6} \text{ dpa/s})\).\[^2\]

Stainless steel loses ductility with increasing dose, with uniform elongations of less than a few percent being reached at typical doses of tens of dpa in reactor experiments.\[^2\] When factoring in the additional deleterious effects of gaseous transmutation products, the loss of uniform elongation with dose may be more rapid.
Such irradiations may have very detrimental effects on the microstructure and the mechanical properties of these steels. There resistance to different kinds of service exposure effects, such as fatigue and corrosion, may be reduced.

**Cavitation Erosion**

Cavitation could occur in the mercury and might lead to cavitation erosion of the container. Cavitation erosion stems from the thermal shock produced by the high-intensity proton beam pulse impinging on the mercury. When the pulses of the proton energy are absorbed in the mercury volume, thermal expansion following the subsequent rapid heating produces pressure waves. These pressure waves propagate at the speed of sound to the vessel walls and are followed by rarefactions. In these regions under tensile stresses, the liquid loses cohesion and cavitates. Although cavitation bubbles may form throughout the mercury volume, it is those near the container walls that can cause erosion. Collapse near the container wall can give rise to high-velocity liquid jets and shock waves. These jets and shock waves may erode the surface.\(^2\)

**Compatibility**

There are two main issues in this area.\(^2\) The first is corrosion and, in particular, the investigations into temperature gradient mass transfer. If such a process were pronounced, loss of mass, i.e., thinning of the target wall, would be of concern. The temperature gradient mass transfer generally occurs by the dissolution of material in
higher temperature regions in contact with the liquid, and with the deposition of the material in cooler regions. Although the loop is closed, an overall equilibrium solubility in the liquid would not be established because of the temperature gradient. The process of the mass transfer could go on continuously.

The other area in the compatibility issue is the possible environmental effect on mechanical properties of the structural material. For instance, mercury could change the mechanical properties of 316 LN SS by affecting grain boundaries.

In the SNS, the neutron source is working in a pulsed mode as described above. The repeating bombardment of the neutron beam on the container causes one form of the cyclic stresses that are experienced by the target container. Other time-varying stresses include the pressure waves applied by the expansion and contraction of mercury in the container. In addition, the normal startup and shutdown will result in temperature and stress transients applied to the target container. Therefore, it’s important to obtain fatigue test information on the target container structural material.

The main frequency of the pressure wave is 60 Hz. A large number of cycles will be accumulated during operation. Since the frequencies and the magnitudes of the expected stresses vary substantially, the effects of frequency and load on the fatigue behavior of 316 LN SS need to be stressed in the investigation.
2.3. Material Composition and Mechanical Behavior

The basic composition of 316 LN SS was listed in Table 1.\textsuperscript{[3]} In order to understand the selection philosophy of choosing the 316 LN stainless steel as a candidate material. An understanding of the main effects of alloying elements in 316 LN SS is needed.

Carbon (C)
1. A strong austenite former.
2. Added to some high-strength alloys for hardening and strengthening effects.
3. Adversely affecting weld-metal corrosion resistance and toughness at low temperatures.

Chromium (Cr)
1. A ferrite and carbide former.
2. A primary contributor to scaling and corrosion resistance.
3. In the stainless steels, this element having little or no effect on high-temperature strength and creep strength.

Molybdenum (Mo)
1. A ferrite and carbide former.
2. Used to improve high-temperature strength and creep resistance.
3. Used to improve general corrosion resistance of steels in non-oxidizing media, and the resistance to pitting corrosion in all media.

Nitrogen:
1. A strong austenite former.
2. Used to minimize grain grown in high chromium steels at high temperatures.
3. Adversely affecting weld metal toughness at cryogenic temperatures.
4. Raising strength.

Nickel (Ni)

1. An austenite former.
2. Used to improve the general corrosion resistance against non-oxidizing liquids.
3. Sometimes added in small amounts to straight chromium grades to improve the mechanical properties.

The Type 316 LN stainless steel (SS) is a low-carbon nitrogen-added austenitic stainless steel, which is similar in the chemical composition to the 316L stainless steel. The usual 316L austenitic stainless steel is fully austenitic after the solution heat treatment but has poor mechanical properties. It has already been shown that nitrogen alloying clearly improves monotonic and cyclic behavior of 316L stainless steel at room and high temperature.\[4\] However, even though the $M_s$ temperature (the temperature at which the martensite starts to form on cooling) is not reached for the 316L, the stress-induced martensitic transformation may occur at or below the $M_d$ temperature (the temperature from which the martensite starts to form under a given amount of deformation), as well as during monotonic deformation or during cyclic deformation. The formation of the stress-induced martensite may sometimes induce some mechanical damage.\[5\] According to
the phase diagram, nitrogen is an austenite stabilizer. The influence of nitrogen on stabilizing the austenite may change in the presence of strain at low temperatures, because the mechanisms of the martensitic transformation may change with deformation. The work reported by J. – B. Vogt et. al. examines the effect of nitrogen and temperature on the fatigue behavior of 316L austenitic stainless steel.\cite{6} A correlation between the macroscopic behavior and the microstructure is developed for the low-cycle fatigue (LCF) and fatigue crack propagation. Two austenitic stainless steels (316L and 316LN) were studied, whose chemical composition differ significantly in their nitrogen content. Table 2 summarizes the different tensile characteristics (yield strength $\sigma_y$, ultimate tensile strength $\sigma_u$, total elongation $A$, and reduction in area $S$) deduced from tests performed at 27 and -196 °C.\cite{6} It shows that nitrogen increases the fatigue strength at 27 and -196 °C. Nitrogen and a decrease in temperature favor planar slip by reducing the tendency for cross slip and avoiding a thidimensional cellular structure. In the 316LN alloy, the stacking fault energy (SFE) that is responsible for planar slip is reduced by low temperatures and by the effect of nitrogen at low temperatures. Note that a lower SEF favors planar slip instead of cross slip. The result of the fatigue-crack-growth rate (FCGR) may be interpreted as a consequence of cyclic hardening due to a combination of interstitial-solute strengthening, strain-induced phase transformation, and a decrease in temperature. Lowering temperature from 27 to -196 °C on both 316L and 316LN materials decreases SEF, and hardens the materials at the crack tip locally, which produces a decrease of the crack-opening displacement (COD), resulting in a decrease of
FCGR. Since the decrease in SFE in 316LN is even greater and with the presence of interstitial solutes of nitrogen, the fatigue resistance of 316LN is larger than 316L SS.\textsuperscript{[6]}
2.4. Fatigue Mechanisms

Fatigue has long been recognized as one of the major causes for the catastrophic damage in components or even entire systems. Fatigue of metallic materials under varying mechanical loading is known to be a process of gradual accumulation of damage. The progression of fatigue damage can be broadly classified into the following stages:\[7\] (a) substructural changes, which cause the nucleation of permanent damage, (b) the creation of microscopic cracks, (c) the growth and coalescence of microscopic flaws to form ‘dominant’ cracks, which may eventually lead to catastrophic failure, (d) stable propagation of the dominant macrocrack, and (e) structural instability or complete fracture.

Since Forsyth\[8\] first documented slip-induced surface roughening during fatigue, Wood\[9\] proposed mechanisms to rationalize the origin of fatigue cracks. The basic premise of his postulate was that repeated cyclic straining of the material led to different amounts of net slip on different glide planes, and the irreversibility of shear displacements resulted in the ‘roughening’ of the material surface. It is well known now that fatigue cracks initiate along the bands of localized deformation known as slip bands.

There are numerous studies of the initiation and propagation of fatigue cracks. The optical microscopy (OM), scanning-electron microscopy (SEM), and transmission-electron microscopy (TEM) techniques were used to explore the microstructural
evolution along the slip bands. More recently, techniques were developed, especially scanning tunneling microscopy (STM) and atomic force microscopy (AFM), to measure the surface displacement due to fatigue, on the order of 20 nm. [10-14]
2.5. Effect of Environment on Fatigue Behavior of Materials

Some Researches on Liquid-Metal Embrittlement Effect

In the past, LME was studied mainly by means of uniaxial tensile tests or slow-strain-rate tensile tests, rather than cyclic loading tests.\textsuperscript{[15-21]} Several materials tested in mercury were found to exhibit more brittle fracture characteristics than in air.\textsuperscript{[15-21]} Some research results of nickel-based and aluminum alloys exhibited similar fracture modes in gaseous hydrogen and liquid mercury, while other results reported that the fracture features were distinguishable in these two conditions, with more intergranular cracking in mercury than hydrogen.\textsuperscript{[15-21]} LME was found to be more severe in nickel and copper alloys than in steels because of the limitation of wetting between the mercury and steel.

Interestingly, although many literatures reported significant LME effects on alloys, little research indicated obvious LME on stainless steels. LME was found to be more severe in nickel and copper alloys than in steels when they are subjected to the corrosion effect of the mercury environment for a long time of service because of the limitation of wetting between the mercury and steel.\textsuperscript{[18]} Related LME phenomena are documented in many literatures.\textsuperscript{[15-21]} This process of LME produces a drastic reduction in the fracture resistance of the solid, but has no apparent changes in its yield or flow behavior. Nevertheless, this effect hasn’t been widely observed in steels.
Pawel, DiStefano, and Manneschmidt investigated the corrosion behavior of 316 LN SS in a mercury thermal convection loop.\cite{22} Two thermal convection loops fabricated from 316 LN SS, containing mercury (Hg) and Hg with 1,000 wppm gallium (Ga), respectively, were operated continuously for about 5,000 h.\cite{22} In each case, the maximum loop temperature was constant at about 305 °C, and the minimum temperature was maintained at approximately 242 °C. Coupons in the hot leg of the Hg loop developed a porous surface layer substantially depleted of nickel and chromium, which resulted in a transformation to ferrite.\cite{22} Analyses of the corrosion-rate data as a function of temperature in the Hg loop suggests that wetting by mercury occurred only above about 255 °C, and that the rate-limiting step in the corrosion process above 255 °C is solute diffusion through the saturated mercury-boundary layer adjacent to the corroding surface. The later factor indicates that the corrosion of 316 LN SS in a mercury loop may be velocity-dependent. Experimental results suggest no obvious LME effect in the Hg loop that contributes to the degradation of coupons. The main mechanism that induced the depletion of elements is diffusion.

Classical LME processes are generally found to occur without notable penetration of the embrittling species into the solid or dissolution of the solid metal into the embrittling liquid. Various other liquid metal degradation phenomena are reviewed elsewhere, such as the grain-boundary penetration, selective attack of solid phases, or reaction between the solid and liquid.
During fatigue tests in the mercury environment, the specimens are subjected to cyclic loading, and there also exists the processes of crack initiation and propagation. Therefore, the loading condition would become another important factor besides the temperature effect mentioned above. With the occurrence of crack initiation and propagation, the interaction between the mercury penetrating into the crack tip and the plastic zone area of the crack tip becomes more obvious. At different loading conditions, the depths of penetration of mercury into the crack are different due to various levels of openings of a crack.

However, complete wetting of the liquid onto the solid is required for active LME. If, for any reason, the liquid “de-wets” the solid metal surface, and LME is essentially stopped.[17] Since mercury wets only fresh surfaces and not oxides, the oxidation of the crack tip will inhibit the LME effect. The interaction of the mercury and the crack tip would become a time-dependent process related to the extent of this inhibition effect. During this process, the dominating step would be the diffusion of mercury through the adjacent oxide film to the metal surface. Some literature suggested that the source of oxygen available to the crack tip is provided by the mercury itself. Since the crack tip is filled with liquid mercury because of hydraulic pressures and capillary effects, air has no direct contact with the fresh surface created at the crack tip. Chemical studies on fresh mercury show that it oxides at a very slow rate.[23] When mercury wets the fresh surface, oxygen contained in mercury oxidizes the surface at a certain rate, which is determined by the concentration of oxygen in the mercury and other factors like temperature. As a
result, the surface is de-wetted, and the forming oxide inhibits the wetting between the mercury and metal surface. This process is gradually developed and time-dependent.

Considering the above process, we can also explain the difference of the mercury effect on the fatigue life of 316 LN SS. The loading condition may influence this time-dependent oxidation process. At high stress levels, the crack is propagating at a rate that makes the oxidation process not so fast as to inhibit the LME effect. Therefore, the LME effect may surpass the oxidation effect carried out by the liquid mercury itself, and the fatigue life is shorter as compared with the test in the air environment. On the contrary, at low stress levels, the oxidation of the fresh surface may be predominant as compared with the slow crack-initiation and propagation rates, and, hence, the fatigue life is increased due to the decreasing wetting effect between the mercury and crack tip with the presence of the oxide film.

Wheeler [18] put forward the mechanism of a competition between LME and concurrent crack-tip oxidation. A schematic illustration of this proposed competition is shown in Figure 2-1.

Minimization of Oxidation-Induced Embrittlement at the Crack Tip

Another factor that seems to contribute to the shortening of fatigue lives at low stress levels in Hg is the minimization of oxidation-induced embrittlement at the crack tip.
Whether through wetting or capillary effects, as mentioned above, the crack is covered by penetrating mercury. Therefore, during the process of fatigue tests in the mercury environment, air has no access to the crack tip, which can lead to two effects. One of them is the decrease of the oxidation effect of the crack tip as a result of lacking the direct contact between air and fresh surfaces at the crack tip. The content of brittle oxide at the plastic area is less compared to the condition with the presence of the direct contact between the air and crack tip. The plastic zone in the former condition is larger than the latter one; thus, the stress at the crack tip is dissipated much more. The other effect can be attributed to a much-reduced role of oxide-induced crack closure in the mercury environment due to far less crack surface oxidation. It’s reported that corrosion debris, which forms as a result of fretting oxidation between the crack flanks, is about 20-40 times thicker in moist air compared to inert oil environments.\textsuperscript{[24-34]} This results in significant closure in moist air since the oxide films here become comparable in size to the crack-tip opening displacements.

The crack closure leads to the suppression of the effective driving force for crack advance through the premature contact between the crack surfaces at positive loads during the fatigue cycle such that, under small scale yielding conditions, the stress-intensity range ($\Delta K$) is reduced from the nominally applied loads, geometry, and crack-length measurements, to some lower effective value, $\Delta K_{\text{eff}}$, actually experienced at the crack tip \textsuperscript{[35]}.\[\]
Specifically, below $10^{-6}$ mm/cycle, where growth rates approach a fatigue threshold stress intensity range, $\Delta K_0$, below which long cracks remain dormant or propagate at experimentally undetectable rates, the origin of such fatigue crack closure is found to be not solely as a result of the cyclic plasticity [35], as first envisioned by Elber's plasticity-induced closure concept [35], but additionally from the formation of insoluble crack surface corrosion deposits [24-35], irregular fracture morphologies coupled with inelastic Mode II crack-tip displacements, the wedging action of viscous fluids contained within the crack, and from metallurgical phase transformations.

The relative effect of oxide-induced crack closure on near-threshold growth rates can be appreciated by considering the simple model of a rigid wedge inside a linear elastic crack [30]. The model considers the reduction in the stress-intensity range from its normal value ($\Delta K = K_{\text{max}} - K_{\text{min}}$) to some effective value ($\Delta K_{\text{eff}} = K_{\text{max}} - K_{\text{cl}}$), and estimates the closure stress intensity, $K_{\text{cl}}$, in terms of the maximum oxide wedge thickness located at distance, $2l$, behind the crack tip

$$K_{\text{cl}} = \frac{sE}{4\sqrt{\pi l}(1 - \nu^2)}$$

(2-1)

where $E/(1 - \nu^2)$ is the effective elastic modulus in plane strain [30].
At a certain nominal $\Delta K$, half of the applied stress intensity range in air may be lost to closure, whereas less than 1% is affected in oil. This clearly indicates that the driving force for crack extension in oil is far less restricted by oxide-induced closure.

**Viscous Fluid-Induced Crack Closure**

Both effects of the mercury environments described above, in terms of the LME effect and minimizing of oxide-induced closure, are to some extent dependent on the chemical properties of liquid metal. There is also a crack-closure effect induced by viscous properties of liquid mercury, which is independent of the mercury chemistry. As modeled in a series of papers by Tzou, et al. [36], the description of this closure phenomenon and its role in governing the effect of paraffin oil, the liquid that they used as a viscous fluid and viscosity on growth rates must include an analysis of both the extent of penetration and the internal oil pressure, since they have opposite dependencies on viscosity.

The analysis of crack closure induced by a viscous fluid inside a growing fatigue crack is based on three principal steps, namely (i) a hydrodynamic analyses of the fluid pressure under cyclic loading for both full and partial fluid penetration, (ii) analysis of the resultant stress intensity, $K^*$, due to the fluid pressure, and (iii) the superposition of the variation of $K^*(t)$ with time, $t$, during the cycle with the variation of applied stress intensities, $K(t)$, to yield the effective (near-tip) stress intensity, $K_{\text{eff}}(t)$ [36].
The penetration distance, \( d \), is estimated as \(^{[36]}\),

\[
d^2(t) = \left( \frac{\gamma \cos \beta}{3 \eta \rho} \right) \int_0^t <h>(t) \, dt
\]  

(2-2)

where \( \gamma \) is the surface tension of the oil, \( \beta \) the wetting angle between the fluid and the crack wall, \( \eta \) the kinematic viscosity, and \( \rho \) is the density.

In case of the complete fluid penetration into an arbitrary tensile edge crack, a fatigue crack of length, \( a \), in a test piece of thickness, \( B \), width, \( W \), and remaining uncracked ligament, \( b=(W-a) \), is subjected to remotely applied sinusoidally varying loads, \( P \), such that the nominal stress-intensity factor is given by

\[
k = \frac{P}{BW^{1/2}} f(a/W)
\]  

(2-3)

where

\[
f(a/W) = 1.12 \sqrt{\frac{a}{W}}
\]  

(2-4)

The corresponding final solutions are provided for three other cases: the partial penetration of fluid in an edge crack, and both partial and full penetration in a cracked compact-tension specimen. In the latter case it is assumed that the fluid pressure is only
generated within the fatigue crack and not within the notch, i.e., over a distance $\bar{a} = (a - a_0)$ rather than $a$, where $a_0$ is the length of the notch $^{36}$.

The overall shape of the idealized fatigue crack is taken to be trapezoidal, with crack mouth opening displacement, $\text{CMOD} = h_m$, and crack tip opening displacement, $\text{CTOD} = h_\delta$, such that the average crack opening, $\langle h \rangle$, at the time, $t$, is given by

$$\langle h \rangle (t) = 1/2 [h_m(t) + h_\delta(t)]$$  \hspace{1cm} (2-5)

The rate of the change of the crack opening is expressed as

$$\dot{\langle h \rangle} = \frac{K_{\text{eff}}^2}{2 \sigma_y E} \left[ \frac{a + 2rb}{rb} \right]$$  \hspace{1cm} (2-6)

where $h_m = \left(\frac{a + rb}{rb}\right) h_\delta$, $h_\delta = 0.49 \left(\frac{K_{\text{eff}}}{\sigma_y E}\right)^2$, $r$ is the non-dimensional rotational factor and the specimen ligament is of size $b$.

The approximate stress intensity due to the presence of these concentrated forces is suggested as $^{36}$.
\( K^* = \frac{2Q}{\sqrt{\pi a}} F \) \hspace{1cm} (2-7)

where \( F = \int_0^a p(x)dx \)

The estimation of the effective stress intensities, \( K_{\text{eff}}(t) \), during the fatigue cycle in the presence of viscous fluid crack closure involves the superposition of the stress intensity due to the fluid, \( K^*(t) \), and the applied stress intensity, \( K(t) \)

\[
K_{\text{eff}}(t) \equiv K(t) + K^*(t) = K_m + \frac{\Delta K}{2} \sin \omega t + \frac{2Q}{\sqrt{\pi a}} F(t) \hspace{1cm} (2-8)
\]

where \( K_m = 1/2(K_{\text{max}}+K_{\text{min}}) \) and \( \omega \) is the angular frequency \[36\].

**Environmentally Assisted Cracking**

The exact mechanisms of the interaction of the mercury and crack tip haven’t been reported extensively. However, many research works has been done, showing the environmentally assisted cracking (EAC) in steels in various media, which may help understand the environmental effects of mercury on fatigue behavior. The fatigue crack propagation (FCP) response of low-alloy ferritic steels has been a subject of continuing research interest since the pioneering work of Kondo et al. (1972) showed that such steels
could be susceptible to environmentally assisted cracking (EAC) in elevated temperature aqueous environments.\textsuperscript{[37]} Kondo, studying an ASTM A302-B steel in a boiling water reactor (BMR) environment at temperatures of 200 °C and 260 °C, demonstrated that, under certain conditions, FCP rates could greatly exceed those that would normally be expected in an air environment under the same conditions. Since Kondo’s early work, many publications have addressed EAC in both BWR and pressurized water reactor (PWR) environments. The result of this work is that the EAC process is reasonably well characterized for these environments and, more importantly, an understanding of the mechanisms responsible for EAC has emerged.

A typical work studying EAC was done by James, et al.\textsuperscript{[38]} They investigated the effect of the water-flow rate upon the environmentally assisted cracking response of a low-alloy steel. The presence of a critical concentration of sulfur species at the tip of a crack is thought to be a prerequisite for EAC to occur in low-alloy steels in BWR and PWR aqueous environments. Sulfur can be “supplied” to the crack tip by a growing crack intersecting embedded MnS inclusions that are present in the steel as an impurity. MnS inclusions readily dissolve in water at elevated temperatures; hence, a growing crack can be supplied with sulfide ions. Sulfides can be removed form the crack tip by any of several mass-transport processes: 1) diffusion due to either a concentration gradient or a chemical potential gradient; 2) ion migration due to an electrochemical potential gradient; 3) fatigue “pumping”, and 4) a convective flow within the crack enclave induced by the external fluid flow.\textsuperscript{[38]} EAC can result when the supply of sulfides exceeds the loss by
the mass transport. James et al. presented results of experiments conducted on relatively large semi-elliptical surface cracks subjected to free stream. Several other investigations have demonstrated that fluid flow can be quite effective in mitigating EAC: Scott et al. (1983, 1984), Kitigawa et al. (1987), Lenz et al. (1988), and Katada et al. (1990). All of these studies have been conducted on compact-tension-like specimens where the crack mouth opening displacements (CMOD) and mass transport path may differ considerably from the types of cracks that one might be more likely to encounter in service. One study (James et al., 1995) has dealt with flow rate effects in tight semi-elliptical surface cracks.

Because the mass transport of sulfides from the crack tip region is an important consideration, the flushing characteristics of compact-type specimens may be quite different from those of more natural semi-elliptical surface cracks. James et al. developed a surface-cracked specimen that features a semi-elliptical surface crack that forms a “natural” intersection with the free surface, i.e., no machined notches nor electrical-discharge machined slits to provide an easier mass transport path. The specimen was orientated such that the surface location of the crack had a T-S orientation (T denotes transverse direction perpendicular to rolling direction; S denotes the short transverse direction perpendicular to T; T-S denotes fracture plane normal to T with propagation parallel in S direction), while the deepest penetration had a T-L orientation (L denotes direction parallel to rolling direction; T-L denotes fracture plane normal to T with propagation in L direction). The details of the specimen development are given
elsewhere (James and Wilson, 1994b). As discussed by James and Wilson (1994b), the specimen produced a mixture of linear bending stresses ($\sigma_b$) and uniform stresses ($\sigma_u$) in the test section. They also described the results of a finite element stress analysis on this specimen, verifying that the simple assumption of uniform tension plus linear bending is appropriate for this specimen.

The results for the flow rate are plotted, employing the “time-domain” format. (Shoji, 1981, 1983) It is well established that the rupture of oxide films at the crack tip is a necessary step in the EAC process, and that the oxide-rupture rate is dependent upon the crack-tip strain rate. It has also been postulated that the crack-tip strain rate represents the mechanical crack-driving parameter in the EAC process. Shoji and co-workers have suggested that the “time-based air rate” is proportional to the crack-tip strain rate, and is a convenient method of characterizing the mechanical crack-driving parameter in corrosion fatigue.
2.6. Effects of Test Temperature on Fatigue Behavior of 316 LN SS

The present researches on the effects of temperature on fatigue behavior of 316 SS mostly concentrate on the low-temperature range below room temperature. The reason is that 316 SS is one of the primary candidate materials in fusion reactors, where materials with good mechanical properties at low temperatures are required. Botsheken, et. al. investigated the influence of martensitic transformation on the low-cycle fatigue behavior of 316LN SS at -196 °C.\[49\] Similar work was conducted by Vogt et al., as mentioned above.\[6\] Both works revealed that the strain-induced martensite transformation is responsible for the higher tensile elongation at -196 °C. As proposed by Botshekan et al., carbon and nitrogen elements play an essential role in the plastic behavior and stabilization of the austenite; on the other hand, the nitrogen facilitates the formation of stacking faults and then of the hexagonal ε-martensite phase during straining. Depending on the stacking fault energy, the formation of the α-martensite occurs directly from the austenite \[50\] or via the hexagonal e-martensite phase. At -196 °C, yield strength (YS) and (UTS) were found to be twice of those at 27 °C, and the elongation is also higher. The softening-hardening curves in low-cycle fatigue at -196 °C show a secondary hardening at high strain level, due to cyclic strain-induced martensite transformation.

As suggested by previous works,\[51\] the tensile strength is marked by a strong increase with decreasing temperature. The yield strength is significantly influenced by the nitrogen content. Steels without nitrogen show only a slight increase in the yield strength
with decreasing temperature whereas for the nitrogen-alloyed stainless steels, a strong increase is found.\textsuperscript{[52]}
2.7. Effect of Frequency on Fatigue Behavior of Materials

The influence of frequency on the fatigue crack propagation behavior of materials has been studied under various test conditions.\(^{[53-73]}\) Increasing test frequency has been reported to both increase and decrease lifetimes, as well as have negligible effects on the fatigue lives of materials.

Creep and Fatigue

The effects of frequency, waveform, and tensile ramp time on the cyclic crack-propagation rate of AISI 304 stainless steel at 570 °C in air has been studied by Plumtree and Schafer.\(^{[53]}\) Single edge-notch specimens (50.8 mm wide and 4.84 mm thick) were fatigued on a servo-controlled electro-hydraulic test machine under load control. A slow-fast triangular waveform resulted in the fastest crack-growth rate, and the fracture surface was intergranular. Their results showed that the crack-propagation rate increased with a decrease in the frequency below 5 Hz using a balanced waveform.

An empirical relationship was proposed by Plumtree and Schafer\(^{[53]}\) for the crack-growth rate with test frequencies ranging from 0.005 Hz to 5 Hz:

\[
\frac{da}{dN} = C \Delta K^{\alpha} f^{\beta} \tag{2–9}
\]
where $\Delta K$ is the stress-intensity factor range ($\Delta K = K_{\text{max}}, K_{\text{min}} = 0, K_{\text{min}}$ and $K_{\text{max}}$ are the applied minimum and maximum stresses, respectively), $f$ is the frequency, and $C$, $\alpha$, and $\beta$ are material constants. The equation has been shown to be useful for the prediction of fatigue crack-propagation rates in both polymers and metals.\(^{[53]}\) This type of relationship has been observed by Guinemer and Plumtree,\(^{[66]}\) James,\(^{[67]}\) and Solomon and Coffin,\(^{[68]}\) at elevated temperatures, and Yokobori and Sato at room temperature.\(^{[69]}\)

Mukherjee and Burns\(^{[70]}\) used a statistical analysis to determine what testing variables, including frequency, were important in the prediction of fatigue crack-propagation rates in poly(methyl methacrylate) (PMMA). Their equation took the form of

$$\frac{da}{dN} = C\Delta K^\alpha f^\beta K_{\text{mean}}^{-\nu} \quad (2-10)$$

where $K_{\text{mean}}$ is the mean stress-intensity factor, $K_{\text{mean}} = (K_{\text{min}} + K_{\text{max}})/2$, and $\nu$ is a constant.\(^{[70]}\) This equation reduces to Equation (2-9) if tests are carried out at a single value of $K_{\text{mean}}$.

The influence of frequency on high-temperature fatigue crack-propagation rates has been attributed to a creep component or creep-fatigue interaction (Sadananda and Shahainian),\(^{[71]}\) whereas other studies have indicated that this effect is due to an environment-assisted cracking component (Coffin;\(^{[72]}\) James;\(^{[67]}\) Solomon and Coffin\(^{[68]}\)). Nevertheless, it has been hypothesized that time-dependent or creep/environment
behavior and time-independent or fatigue behavior could be accounted for separately using a linear superposition model (Guinemer and Plumtree;\cite{66,73}) such that

\[
da/dN = F(\text{Creep/environment}) + G(\text{Fatigue})
\]

(2-11)

The data analysis using multiple linear regression techniques showed the second term to be negligible, indicating that the role played by pure fatigue was not significant.

**High-Frequency Fatigue**

The frequency effect on the high-cycle fatigue behavior of materials have been conducted by some researchers.\cite{74-78} Morrissey et al. conducted high-cycle fatigue experiments on Ti-6Al-4V at different R ratios, where \( R = \sigma_{\text{min}}/\sigma_{\text{max}} \), \( \sigma_{\text{min}} \) and \( \sigma_{\text{max}} \) are the applied minimum and maximum stresses, respectively. It was found that at a lower \( R \) ratio of 0.1, the fatigue strength increases with increasing frequency. At a higher \( R \) ratio of 0.8, the frequency effect vanishes at the lower frequencies.\cite{74} Fatigue results of Papakyriacou et al. indicated that some niobium and tantalum alloys showed prolonged lifetimes and increased mean endurance limits at a higher loading frequency of 20 kHz than at 100 Hz.\cite{75}

The frequency effects on fatigue-propagation rates were conducted on various kinds of materials, one of which was performed by Padula et al. on the powder-metallurgy, nickle-
base superalloy KM4 at room temperature.\textsuperscript{[57]} Tests were done at 50 Hz and 1,000 Hz in an advanced servohydraulic testing machine at R-ratios between 0.4 and 0.7. The experimental results showed no effect of frequency on the fatigue behavior at room temperature, which is expected in this type of alloy, and this result yields confidence in the reliability of the servohydraulic fatigue testing system.

**Ultrasonic Fatigue**

Studies to compare the fatigue lives at ultrasonic frequencies (about 20 kHz) of different materials have been carried out by various investigators.\textsuperscript{[58-64]} While results indicate that the fatigue life is shortened in some materials at the ultrasonic frequency, several papers report an enhancement for other materials.\textsuperscript{[58-64]}

Ultrasound (around 20 kHz) of very high intensity can cause damage to materials and their subsequent failure under resonance conditions. The failure is essentially by fatigue of the push-pull type, and, thus, the process is known as “ultrasonic fatigue.” The most important feature of this phenomenon is that the fatiguing stress wave of sufficient magnitude (based on the input power to the transducer and the difference in the cross-sectional areas of the specimen) is set up, which causes the material to fail usually within minutes ($>10^5$ cycles). Thus, a high-cycle fatigue test can be completed in a matter of minutes. Ultrasonics in the kHz range can, therefore, be used for destructive testing, in contrast to ultrasonics used for nondestructive evaluation testing (MHz range). The
cyclic stress amplitude can be estimated by measuring the displacement amplitude of vibration.\[79\] Studies on ultrasonic fatigue are rather limited because of the lack of the commercial equipment for generating high-intensity vibrations and also the absence of a standardized procedure. Thus, all the studies so far have, by and large, been carried out using in-house gadgets only.\[80\]

The testing of ultrasonic fatigue-cycling specimens has played an important role in the study of stress limits in metallurgy and materials science, and continues to be of interest in these disciplines. Many mechanical structures are subjected to small elastic-vibration cycles at very high frequencies due to mechanical, acoustical, or aerodynamical origin, e.g., gas-turbine blades. These small cycles can contribute to the damage and provoke a catastrophic fracture of the structures.

It is believed that the ultrasonic energy can activate many mechanisms, such as the release and mobilization of dislocations, the generation of point defects and their diffusion, etc. This is because the energy is known to be absorbed primarily at lattice imperfections, such as dislocations. The extent to which this activation takes place depends largely on the intensity of vibration and is practically independent of the frequency (over a wide range).\[81\] Thus, most of the work has been done at lower ultrasonic frequencies of about 20 kHz, since it is convenient to generate such frequencies at sufficiently large intensities.
Studies to compare the fatigue lives at low frequencies (up to 96 Hz) and ultrasonic frequencies (about 20 kHz) of different materials have been carried out by various investigators.\textsuperscript{[58-64]} While some claim that the fatigue life is shortened in particular materials at ultrasonic frequencies, others report an enhancement for particular materials.

Hockenhull\textsuperscript{[59]} observed a clear “frequency effect” in an Al-Cu-Mg alloy, with the plastic deformation per cycle being reduced drastically at ultrasonic frequencies. Also, the striation spacing was much smaller, and slip more localized. In some cases, complete $45^\circ$ shear-mode fractures were obtained, indicating the predominance of stage I crack propagation at ultrasonic frequencies.

Mason and Wood\textsuperscript{[60]} observed striking differences in the microstructural changes under ultrasonic-frequency loading in contrast to low-frequency loading, particularly with regard to the distribution of slip. In normal testing frequencies, slip takes place all over the grain. At ultrasonic frequencies, slip becomes concentrated in isolated bands in particular grains. The bands then degenerate into microcracks, which form a macro-crack that propagates through the material. In a later study by the same authors\textsuperscript{[61]} on low-carbon steel, it was found that at ultrasonic frequencies, the endurance limit can be lowered substantially. It is believed that the possible reasons for the pronounced lowering in the endurance limit could be the “local breakaway of dislocations” from particular pinning points under the influence of high-frequency stresses\textsuperscript{[61]}.
Wood and MacDonald\textsuperscript{[62]} found that the damage caused by ultrasound is not only highly localized in particular areas, but is also virtually catastrophic in that the crack-propagation rate was determined to be about 1,000 times greater than that at low frequencies. This is probably because the stress intensity at the crack tip is much larger in the high-frequency situation, and, thus, the failure is apparently premature at ultrasonic frequencies. MacDonald\textsuperscript{[63]} observed a similar effect, the striations being very closely spaced, indicating a very high crack velocity. In another study, Bajons et al.\textsuperscript{[64]} noticed differences in the distribution of persistent slip bands and in the mode of cracking on ultrasonic loading, compared to conventional loading. Sriraman and Vasudevan\textsuperscript{[80]} found the apparent presence of multiple crack origins in an ultrasonically-fatigued specimen of annealed commercial aluminum, indicating the localizing nature of damage that can be catastrophic. The absence of clear striations probably corroborates with the general view that the fracture mechanisms (and perhaps even the stages involved) under ultrasonic vibrations could be different from those operating in conventional fatigue situations.

Apparent inconsistencies arise in previous works regarding the frequency effects on fatigue behavior. Reasons for the inconsistencies can be due to different test conditions and materials. However, it is important to note that when considering the frequency effect, two factors are important, specimen self-heating and strain-rate effects. Self-heating of materials will produce different specimen temperatures at various frequencies. An efficient technique of holding the specimen at the same temperature is necessary to investigate solely the strain-rate effect that is associated with the term, $f$, in Equations (2-
9) by eliminating the temperature effect. Ignoring the specimen self-heating during high-cycle fatigue tests may lead to entirely different test results and conclusions of the frequency effect.
2.8. Mechanisms of Ultrahigh Cycle Fatigue

The fatigue of metals has been extensively studied. However, most published research does not extend past around $10^7$ cycles. Because plots of the stress versus number of cycles to failure (S-N curves) of ferrous alloys and some other metals apparently reach a horizontal asymptote, it was assumed that specimens tested at stresses below the asymptote, called the fatigue limit, would have infinite lives. However, recent research has discovered fatigue failures at stresses below the fatigue limit and lives above $10^7$ cycles, termed ultra-high cycle fatigue (UHCF).

Traditional fatigue analyses identify in some metals a fatigue limit, which is the stress limit below which the metal will not fail after an infinite number of cycles [82]. In these materials, primarily steels, plots of the stress versus number of cycles to failure (S-N curves) exhibit an apparent horizontal asymptote around $10^5$ to $10^6$ cycles. Failure at lives below about $10^5$ cycles is termed low-cycle fatigue (LCF). Failure at lives above about $10^8$ cycles has been termed high-cycle fatigue (HCF). However, the calculation of traditional fatigue limits is based upon measurements to a maximum of about $10^7$ cycles. This approach was safe in the past because the fatigue lives of machinery were well below $10^6$ cycles. However, many modern applications, for example, in the transportation [83] and electronic [84-87] industries, can require fatigue lives of over $10^8$ cycles. In recent years, research has begun to extend S-N curves above $10^7$ cycles, termed ultra-high cycle fatigue (UHCF). In the mid 1980’s, failure above $10^7$ cycles was
first reported. However not until recently, in the late 1990's, did UHCF research begin in earnest. This area of research is just beginning to be fully explored. The amount of the published results of UHCF is still relatively small. Failure at stresses significantly below traditional fatigue limits has been discovered. In order to ensure safe design, the fatigue behavior of materials must be examined above $10^7$ cycles.

The Present Research on the Ultra-High Cycle Fatigue

Several studies have been completed, exploring the gigacycle fatigue behavior of metals. Some of their results are summarized in the following sections. The researchers found fatigue failures in the UHCF region at stresses below the conventional fatigue limit. A typical example is shown in the S-N curve for a 1Cr-Mo steel with $R = -1$ [88], where $R$ is the ratio of the applied minimum to maximum stresses. A plateau corresponding to the conventional fatigue limit can be observed from approximately $7 \times 10^5$ to $2 \times 10^7$ cycles, followed by failures in the UHCF region, up to about $6 \times 10^7$ cycles.

The fatigue behavior of non-ferrous alloys has also been examined [82, 89-95]. UHCF behavior similar to that for steels [96, 97] has been found for a 2024/T3 aluminum-base alloy and an ULTIMET cobalt-base superalloy [82, 89-95].
Location of Fatigue-Crack Initiation

When the researchers examined the location of the fatigue-crack initiation, all found that the fatigue cracks of specimens that failed in the UHCF region originated in the interior of the specimen, usually at non-metallic inclusions. Conversely, specimens that failed earlier did so by cracks that originated on the surface. The boundary between the surface and internal initiations is generally at the HCF plateau and very distinct. This observation holds true for both ferrous [98, 99] and non-ferrous alloys, specifically the ULTIMET superalloy [89, 91-95]. At higher applied maximum stress levels, greater than 600 MPa, cracks originated from the surface of the ULTIMET superalloy. However, at lower peak stresses, less than 600 MPa, which corresponds to the plateau region, a subsurface or close to surface-crack initiation was observed. The crack-initiation sites showed the cleavage-like feature. The mechanism behind this phenomenon will be examined later in this paper.

In order to further investigate the influence of inclusions on UHCF, Bathias [90] conducted fatigue tests on both standard specimens of a N18 nickel-base alloy and specimens of the same alloy seeded with inclusions. The fatigue strengths of the seeded specimens were found to be lower than those of the standard specimens, particularly with an R ratio of 0.8 [90]. The effect was less pronounced with an R ratio of zero. These results confirm the role of inclusions and suggest that their importance is greater at larger R ratios. Smaller R ratios result in larger stress amplitudes, and therefore, more damage, which masks the inclusion effect on fatigue life. Murakami, Toriyama, Tsubota, and
Furumura [100] went the other way; instead of increasing inclusions, they decreased them. Super-clean specimens of a SAE52100 bearing steel were prepared by an electron-beam remelting process. It was observed that some fatal cracks initiated at bainite inhomogeneities rather than inclusions. In the super-clean specimens, the size of the inclusions was so small that the effect of inhomogeneities became prevalent.

Bathias, Drouillac, and Francois [101] performed fatigue tests on a number of alloys, including steels, cast iron, Ni based alloys, Ti alloys, aluminum alloys, and magnesium alloys, up to $10^{10}$ cycles, and summarized that very often there is a surface-subsurface initiation site transition around $10^7$ cycles. But this mechanism is not unique. When the interior defects or inclusions do not show critical damage, the initiation appears at the surface.

One thing needed to be pointed out here is that it may seem that failure caused by surface initiations will show a short fatigue life, and failure induced by internal inclusions will show ultra-high cycle fatigue failure, but it’s not necessarily correct, because many data show that internal inclusions can cause failure at both short and long fatigue cycles [99, 102, 103]. It’s obvious that if a material has large internal inclusions, the failure could easily originate from those areas. In contrast, a high-purity material with less internal defects is not as susceptible to a failure started from inside, and more likely to fail due to a crack initiated on the rough surface.
Existence of Primary and Secondary Plateaus

Some materials were observed to fail in the UHCF region, but did not clearly show the HCF plateau seen in other metals [82, 88]. For example, no clear HCF plateau was observed in the S-N curves of the SCM 435 Cr-Mo steel with a carburized/nitrided surface [99]. Nishijima and Kanazawa [88] suggest a possible explanation for the absence of a HCF plateau. They argue that due to the lack of plasticity, the internal crack-growth rate for harder materials is faster than for less hard materials. Therefore, the UHCF region of the S-N curve, attributed to internal cracks, will move to shorter lives, shrinking the HCF plateau transition region, eventually to the point where it is no longer observed. The disappearance of the plateau in a hardened steel [99] supports this assertion.

The S-N curves of some materials suggest that a second plateau exists at extremely long lives. For example, a secondary UHCF plateau is seen for the N18 nickel alloy tested at 450 °C. The S-N curve of the ULTIMET superalloy [89, 91] shows a possible secondary plateau starting near 5 x 10^6. The S-N curve for the medium carbon steel [99] shows a possible secondary plateau starting near 5 x 10^8.

The stress level of the UHCF plateau correlates well with the ultimate tensile strength (UTS) of the material [82, 89, 98, 101, 104]. A linear regression reveals that the ratio of the UHCF plateau stress to the UTS is approximately 0.42 for steels, suggesting that a
single mechanism is dominant for all steels. Insufficient fatigue results currently exist to investigate the same relationship in other types of materials.

**UHCF of Silicon**

Using the micron-scale structure previously discussed, research has begun to explore UHCF of silicon [84, 85]. The high frequencies of the tests (40 kHz and 50 kHz) have allowed tests to be run up to $10^{11}$ cycles. The S-N curves of both monocrystalline and polycrystalline thin-film silicon [84, 85] exhibited fatigue failures up to $10^{11}$ cycles at stresses as low as half of the fracture strength. Both also exhibited asymptotic behavior, suggesting that a true fatigue limit might exist. A second plateau, as seen in metals, was not observed. The fatigue strength of the monocrystalline silicon was observed to be greater than that of the polycrystalline silicon, as expected, because a single crystal of silicon has a higher fracture strength than polycrystalline silicon. The UHCF of thin films of silicon has direct applications in microelectromechanical systems (MEMS). MEMS are generally constructed of silicon and often used in applications, such as computing, which subject the MEMS to many cycles at low loads.

**Internal Crack Initiation Mechanism**

Fatigue cracks in the LCF and HCF ranges are generally initiated on the surface. However, it is accepted that fatigue-crack initiation in the UHCF range moves to the
interior [88, 98-99, 105, 106]. Though internal cracks can also initiate at porosities and inhomogeneities, they most often initiate at inclusions, giving rise to fisheye-type microstructures. A fisheye is a radial microstructure with a small bright spot at the center. The inclusion is usually at the center of the bright spot. The fisheye area is also called the optically dark area (ODA) because of its visual character under a microscope. Shiozawa, Nishino, Yamamoto, and Lu [107] examined the topographic data around a non-metallic inclusion using a three-dimensional SEM analysis. They observed a very rough and granular morphology in the bright spot in the vicinity of a nonmetallic inclusion, which they called the granular-bright-facet (GBF). The result indicates that the surface roughness of the GBF area is larger than that of the ODA, and is formed by the initiation and coalesces of multiple microcracks around an inclusion. Cracks can also initiate at inclusions by three possible mechanisms: cracking of the particle, debonding of the particle from the matrix, and formation of slip bands near the particle [108].

\[ \sqrt{\text{area}} \] Parameter Model

Several researchers have investigated the effect of inclusion size on fatigue behavior [102, 107]. Murakami, Toriyama, Tsubota, and Furumura [100] have developed a model, below, to predict the fatigue limit \( (\sigma_w) \) of metals with non-metallic inclusions, based upon the size of the inclusion. This model is called the \( \sqrt{\text{area}} \) parameter model.
\[ \sigma_{\text{w}} = \frac{C(HV + 120)}{\left(\sqrt{\text{area}}\right)^{\frac{1}{6}}} \cdot \left(1 - \frac{R}{2}\right)^{\alpha} \]  

The constant, \( C \), depends on the location of the inclusion (\( C = 1.43, 1.56, \) and 1.41 for an inclusion at the surface, in the interior, or just below the surface, respectively), \( \sqrt{\text{area}} \) is the square root of the area of the inclusion, and HV is the Vickers hardness. The coefficient, \( \alpha \), depends on the hardness as follows, \( \alpha = 0.226 + \text{HV} \times 10^{-4} \). The effect of inclusion size on fatigue behavior can be normalized by plotting the relative stress versus number of cycles to failure, where the relative stress is the ratio of the applied stress to the predicted fatigue limit calculated using Equation 1. Wang, Berard, Dubarre, Baudry, Rathery, and Bathias [104] have developed an empirical modification of the \( \sqrt{\text{area}} \) parameter model, which predicts the fatigue life at a specified number of cycles, rather than the fatigue limit. In this modified model, the constant, \( C \), is replaced by a parameter, \( \beta \), which equals 3.09-0.12\log N_f \) for inclusions in the interior and 2.79-0.108\log N_f \) for inclusions on the surface.

**Slip Mechanism**

Several investigations [88, 105, 109] propose a fatigue mechanism based upon slip. Mughrabi [105] classifies materials into Type I, which are single-phase materials with no internal defects, and Type II, which contain internal defects. According to this model, fracture in the LCF and HCF regions for both Type I and Type II materials occurs due to
crack initiation on the surface, resulting from the formation of surface roughness by persistent slip bands (PSBs). The HCF limit, the first plateau in the S-N curve, corresponds to a threshold value below which PSBs will not form.

In Type II materials, once the stress is sufficiently low to remove the possibility of the formation of surface roughness by PSBs at the surface, cracks initiated at internal inclusions become dominant. Cracks at internal inclusions dominate the UHCF region, because the probability of finding an inclusion in the interior of the specimen is greater than on the surface. This was quantified by Mughrabi [105] for the case of a cylindrical specimen in Equation 2-13 below, where \( N_S \) and \( N_V \) are the number of inclusions in the surface layer and in the entire volume, respectively, and \( d_i \) and \( d \) are the diameters of the inclusion and the specimen, respectively.

\[
\frac{N_S}{N_V} = 4 \frac{d_i}{d} \tag{2-13}
\]

The volume density required to find at least one inclusion on the surface may be calculated using Equation 2-14 below, also developed by Mughrabi [105], where \( l \) is the length of the specimen.

\[
n_{crit} \geq \frac{1}{\pi \cdot d \cdot d_i \cdot l} \tag{2-14}
\]
Though the probability of finding an inclusion in the interior is greater than on the surface, cracks initiated at internal inclusions will grow more slowly than cracks initiated on the surface, due to a lower fraction of irreversible slip. The lower fraction of irreversible slip could be due to the isolation of internal cracks from the environment. In LCF and HCF, oxidation speeds crack growth on the surface by preventing full crack closure, thereby decreasing the life. However, in UHCF, interior cracks are isolated from the effects of the environment, causing crack-growth rates to be slower and resulting in longer fatigue lives. Nishijima and Kanazawa [88] also point out that the stress-intensity factor for internal cracks is less than for external cracks, contributing to the slower crack growth in the interior as compared to the surface. Therefore, failure by internal cracks will be seen at longer lives, in the UHCF region, than failure by surface cracks, in the LCF and HCF regions.

In Type I materials, there are no internal defects for crack initiation. However, surface roughness will still form, below the PSB threshold, due to irreversible slip, allowing surface-crack initiation. This process is slower by slip than by PSBs, leading to longer lives. Surface roughness also forms in the UHCF region in Type II materials, but its effect is hidden by internal initiation at defects. The slip mechanism proposes a second plateau, which corresponds to the stress value below which the fraction of irreversible slip becomes negligible.
Hydrogen Embrittlement Mechanism

Murakami, Nomoto, and Ueda [99] propose a different mechanism for UHCF. This mechanism is still based upon internal initiation at inclusions, but it proposes the underlining cause to be hydrogen embrittlement combined with fatigue. Hydrogen is known to degrade fatigue properties of materials in HCF [108]. Hydrogen tends to gather at inclusions, especially over the long times considered in UHCF. The presence of hydrogen assists the formation and growth of cracks at inclusions until they reach a critical size above which they grow on their own in the typical manner. This assertion is supported by the observance of elevated amounts of hydrogen in ODAs surrounding fisheye fractures. Further study indicates larger ODAs for specimens with longer lives. Specimens with a lower hydrogen content have smaller ODAs at the same number of cycles, and it is presumed that a lower hydrogen content retards the growth of ODAs [110]. Because this mechanism depends on a time-dependent process, diffusion, it will be manifested at longer times and lives. In addition, because it is time-dependent rather than cycle-dependent, test frequency would be expected to affect UHCF behavior. However, research by Furuya, Matsuoka, Abe, and Yamaguchi [102, 103] indicates that the size of ODAs is independent of the test frequency, and therefore, the length of time, of the test. These researchers suggest that the formation of ODAs is possibly influenced by both time-dependent hydrogen damage and cycle-dependent fatigue damage.
Initiation at Porosities and Inhomogeneities

In the absence of inclusions, internal cracks from UHCF can initiate at porosities and inhomogeneities. As found by Bathias [90], crack initiation at porosities can be significant when the R ratio is low, even when inclusions are present, particularly in nickel alloys. The porosities can initiate a crack in competition with inclusions, especially when the load ratio is equal to -1 or 0. In the gigacycle regime, the crack can initiate from the surface of cast aluminium alloys due to presence of porosities, while for nickel alloys, the initiation from porosities can occur internally [90]. Murakami [100] investigated the effect of inhomogeneities. Murakami found that when the size of inclusion is very small, as in super-clean steels prepared by electron-beam remelting, initiation at inhomogeneities, such as bainite, can become dominant, because the size of the inhomogeneities is larger than the size of the inclusions.

Mechanism of UHCF in Silicon

Silicon does not exhibit the stepwise S-N curve seen in metals, because fatigue in silicon operates by a fundamentally different mechanism. Since silicon does not experience room-temperature plasticity, extrinsic toughening, or susceptibility to stress-corrosion cracking, it is not expected to fail by fatigue in air at room temperatures. However, in both mono- and poly-crystalline silicon thin films, fatigue failures have been observed [84-87]. The fatigue behavior appears to be continuous across a range of lifetimes from approximately $10^5$ to $10^{11}$ cycles, suggesting that a single fatigue mechanism acts in all
regions of the S-N curve. It’s reported that 2 μm thick polysilicon films can degrade and fail under cyclic loading conditions in moist ambient air at cyclic stresses some 50% of the single cycle fracture strength [86]. Muhlstein, Stach, and Ritchie [85] suggest that fatigue of thin films of silicon occurs by an environmental mechanism termed reaction-layer fatigue. Reaction with the atmosphere causes the formation of a layer of SiO₂ on the surface of the silicon. Crack initiation occurs in the oxide layer. Then the freshly exposed silicon oxidizes. This process repeats until a critical crack size is reached, at which point the silicon fractures. During the fatigue process of the polycrystalline silicon cantilever beam, the native oxide at the root of the notch thickened significantly. It is suggested to be originated from a mechanical process, since the silicon specimen remained within 1 K of ambient, which does not seem to contribute much to this oxidation phenomenon. The deformation of the SiO₂ reaction layer and the associated strain energy are thought to modify the diffusivity of oxygen and kinetics of the interfacial reaction. An alkene-based monolayer is applied to the polycrystalline silicon to avoid the formation of native oxide, thus to suppress the reaction-layer fatigue. Results indicate that lifetimes of specimens with coated films are not affected by the applied cyclic stresses, which confirms that the fatigue of polycrystalline silicon beam is by the reaction-layer fatigue mechanism.
Various methods and theories have been developed to study and model the fatigue process. A diversity of theories, ranging from constitutive models, micromechanics, and damage mechanics, to empirical solutions, has been developed to predict the fatigue behavior. However, many issues regarding fatigue mechanisms and phenomena remain unsolved.

For typical engineering applications, empirical equations have been utilized extensively and successfully to describe the phenomenological fatigue behavior of materials. In the case of high-cycle fatigue, the Basquin's relation is generally used. In the case of low-cycle fatigue, the Manson-Coffin low-cycle fatigue relation is used.\(^{[11]}\) Equation (2-15) forms the basis for the strain-life approach to the fatigue design, and has found widespread applications in industrial practice.

\[
\frac{\Delta \varepsilon}{2} = \frac{\Delta \varepsilon_e}{2} + \frac{\Delta \varepsilon_p}{2} = \frac{\sigma_f'}{E} (2N_f)^b + \varepsilon_f' (2N_f)^c
\]  

(2-15)

In Equation (2-15), \(\Delta \varepsilon\) is the total strain range, \(\Delta \varepsilon_e\) is the total elastic strain range, \(\Delta \varepsilon_p\) is the total plastic strain range, \(\sigma_f'\) is the fatigue-strength coefficient, \(E\) is the Young's Modulus, \(b\) is the fatigue strength exponent, \(\varepsilon_f'\) is the fatigue-ductility coefficient, \(c\) is the fatigue-ductility exponent, and \(N_f\) is the number of cycles to failure.
From the micromechanics viewpoint, much theoretical work was conducted in an effort to explain the formation of cracks along slip bands. The theories may be categorized into two classes. The first category includes theories that explain crack formation as a gradual process that begins at the commencement of fatigue. These theories are mainly based on the formation of extrusions and intrusions. Another class of theories was put forth by Mura et al.\cite{112} Those theories\cite{10-14,112} described the slip band as an accumulation of vacancy dipoles piled up against an obstacle. In their approach, the accumulation of dislocations along slip bands due to slip irreversibility leaded to the build-up of strain energy along these slip bands. For this class of theories, cracks were assumed to initiate when the change of the free energy reaches a crack-nucleation criterion.

Furthermore, Baxter et al.\cite{113} quantitatively measured the growth of slip bands using photoelectron microscopy. Baxter et al.\cite{114} proposed that during the early stages of crack formation, the elongation process of persistent slip bands was accompanied by cyclic hardening of the material within the previously formed portion of persistent slip bands. This model was made quantitative by selecting materials properties based upon the microstructure of persistent slip bands and calculating the deformation field by the finite-element method. Based on this analysis, Baxter et al.\cite{115,116} further developed models for the prediction of the high-cycle fatigue life and fatigue limit of various alloys.
In recent years, there was a substantial development of so-called "Unified Theories", in which the irreversible processes, such as inelastic behavior and damage, were represented by a set of internal state variables.\cite{117} The internal state variables were typically described by constitutive laws of plastic deformation with the objective of the inelastic analyses of structural components. Initially, the theoretical development had its origin in the works of Malvern,\cite{118} etc. whose model did not contain evolving internal state variables to describe inelastic behavior. The field started to gain momentum in the mid-1960s when internal-state-variable models began to appear. With the improvement of computers, rapid advances were made in the 1970s through the modeling efforts of Bodner and Partom,\cite{119,120} Hart,\cite{121} etc., and further refined in the 1980s by Kremple et al.,\cite{122-125} etc.
2.10. Modeling on the Fatigue at Very High Frequencies

Acoustic fatigue is defined as cyclic loading with very low amplitude (elastic stress) and very high frequency (some kHz). Many publications have shown that this type of loading is able to produce damage and fracture of structures. Since the elastic deformation rate is very high, the thermal effect is suspected to be nonnegligible and a coupled thermoelastic analysis is preferred. Because the experimental apparatus uses longitudinal waves to generate a cyclic stress in the specimen, Kong et al. used the longitudinal wave motion theory for the formulation of the problem. Two methods were used to formulate the evolution problem: the first one dealt with the use of classical time integration schema, and the second used the Fourier transformation to solve the evolution problem in the frequency space. Similar researches could also be found in [127-129].

Bathias et al. studied the fracture-mechanics behavior of a titanium alloy-Ti6Al4V in ultrasonic fatigue. They presented a finite-element-method calculation of the stress intensity factor, $K_I$, in ultrasonic fatigue with a positive mean stress. They also gave out the fatigue growth rates of Ti6Al4V at high frequencies at room temperature and with the stress ratio, $R$, between -1 and 0.9.

Before, some researchers used either machines superposing a static load and an ultrasonic load at low frequencies, below 100 Hz, or ultrasonic machines at ultrasound frequencies,
nearly at 20 kHz, but with a null mean load: one of the specimen extremities is fixed to a sonotrode, while the other extremity vibrates freely. In the first case, it takes 11 days to achieve $10^8$ cycles, whereas in the second case, 2 hrs are sufficient. Thus, in the second case, the experiments are more rapid and sometimes nearer to the real conditions but with a serious limitation, the mean load being null. To be able to study ultrasonic fatigue depending on the mean stress, Bathias et. al. designed a new system. The testing system is essentially a combination of a tensile machine and an ultrasound machine constituted of a power generator whose frequency is held at 20 kHz. The vibration of the specimen is executed by a piezo-ceramic type of a transducer, which transduces the acoustical wave to the specimen through a power concentrator (cone) in order to obtain more important stresses and an amplification of the displacement. The resonance length of the specimen and concentrator was calculated at a frequency of 20 kHz. The static tensile load is transmitted with a frame to the inferior cone via one end of the specimen. At the second end of the specimen, another cone is attached, which goes to the load cell. During the test, the static load is automatically kept constant. The dynamic displacement amplitude of the specimen extremity, $U_0$, is measured by an optic fibre sensor, which permits to measure the displacement from 1 µm to 199.9 µm, with a resolution of 0.1 µm. The vibratory stress intensity factor can be calculated according to this measure. For a virgin specimen (without crack), the vibratory stress and strain can be also determined at the midsection. This maximum strain value had been confirmed to be exact by the use of an electric strain gage. A system of video-camera-television has been used for the
control of crack initiation and propagation; the system refines events to 1/25th of a second and magnifies specimen surface 140-200 times.

In a vibratory system, the situation becomes more complex in the presence of inertia force and in the absence of the nominal force that is necessary to the classical fracture approach. A finite-element method was used for the calculation of $K$. In the case of a specimen in vibration applied by a mean force, ANSYS program, developed by Swanson Analysis System Inc., was used. The machine designed by Bathias et al. enabled one to determine fatigue thresholds as low as $10^{-11}$ m/cycle and with stress ratios $R$ as high as 0.8 and 0.9, which is very difficult to obtain in conventional fatigue.

The calculation of $K$ is composed of two processes. First, after using statical eq. (2-16), static stress intensity factor called the mean factor, $K_s$, can be found, which is proportional to the tension force, where $\{f\}$ is statical force and $\{u_s\}$ is statical displacement.

$$[K_s]\{u_s\}=\{f\} \quad (2-16)$$

Secondly, the program constructing the geometrical stiffness matrix under the given tension force, amplitude stress intensity factor, $K_a$, can be determined by using eq. (2-17).

$$[[K]+[K_g]u-\omega^2[M]u=\{0\} \quad (2-17)$$
where \([K]\) is the stiffness matrix, \([K]_g\) is the geometrical stiffness matrix, \([M]\) is the mass matrix, \(\{u\}\) is the displacement vector and \(\omega=2\pi f\), and \(f\) is intrinsic frequency.\textsuperscript{55}

Their results showed that at room temperature and when the environment is not aggressive, the crack-propagation mechanism are the same as in ultrasonic fatigue and in conventional fatigue. Besides, \(\Delta K_{th}\) decreases with \(R\) as \(R\) is increased from 0 to 0.9. Thus, the R stress-ratio effect exists in ultrasonic fatigue as in conventional fatigue and the closure effect remains despite the possibility of a vanishing of residual stresses by vibrations.
2.11. Project Objectives and Technical Approaches

The overall objectives were to provide the basis for engineering application and to explore the fundamental scientific problem regarding the fatigue mechanism of the material. The objectives of present project are: a) Provide mercury-environment fatigue crack initiation and growth rate data to assure the durability and reliability of Spallation Neutron Source (SNS) target materials, including 316 LN stainless steel. b) Understand and characterize the key processes that control mercury-environment crack initiation and propagation behavior of target materials. c) Formulate theoretical models for predicting crack initiation and growth characteristics, which can be used in damage-tolerant design and analyses. d) The research will address the microstructural, environmental, and mechanical aspects of the fatigue process.

The influence of loads, frequency, and environments will be emphasized. High-cycle fatigue tests will be conducted on the annealed and cold-worked specimens to provide a mechanistic understanding of the fatigue behavior. The microstructural characterization of fatigued specimens was performed. Material Test System (MTS) machines, SEM, and TEM would be employed. Attempts were made to capture and quantify the influence of microstructure on the fatigue behavior of 316 LN stainless steels, e.g., by measuring the striation spacing, the stress intensity factor range was calculated to characterize the fatigue propagation behavior at different frequencies and environments. Based on the
experimental observations, theoretical models to specimen temperature were developed for 316 LN stainless steels.
PART 3

EXPERIMENTAL PROCEDURES
3.1. High-cycle Fatigue

The Type 316 LN SS specimen material was an annealed condition with the chemical composition shown in Table 1.\textsuperscript{[1]} The material was melted by the electric-furnace, argon-oxygen decarburization process (EF/AOD) and met the American Society of Mechanical Engineers (ASME) NCA3800 QSC-245 specification.\textsuperscript{[1]} The specimens were annealed at 1,038 °C. Two specimen types were employed for fatigue tests with R ratios of -1 and 0.1 (1) to accommodate the limitations of fixture configurations of two different Material Test Systems and (2) to avoid buckling during fatigue testing with R ratio of -1. Round-bar specimens were used for fatigue tests with a R ratio of -1. The geometry of the round-bar sample is illustrated in Figure 3-1(a). The specimen had a length of 228.60 mm, a gage length of 20.32 mm, and a diameter of 7.62 mm. Button-head specimens were used for fatigue tests with a R ratio of 0.1. The geometry of the button-head specimen used is illustrated in Figure 3-1(b). The specimen has a total length of 118.8 mm, a gage length of 19.0 mm, and a diameter of 5.1 mm.

In order to conduct fatigue tests in a mercury environment, an attached container of 304 SS was used. The gage section of the specimen was immersed in mercury enclosed in the container. The container had a height of 90 mm and an inner diameter of 18 mm for the round-bar specimen, and a height of 48 mm and an inner diameter of 17 mm for the button-head specimen. The container was attached to the specimen with a silicone-
rubber adhesive sealant, so that the container could be removed from the specimen after the test, and reused.

The fatigue test procedure was in accordance with the American Society for Testing and Materials (ASTM) E466, “Conducting Constant Amplitude Axial Fatigue Tests of Metallic Materials”.\[2\]

A state-of-the-art electrohydraulic Material Test Systems (MTS) was used in the present study. It has a frequency range from 0.001 Hz to 60 Hz, and is capable of being operated in vacuum as low as 1.33x10^{-4} Pa. It can be used to run tests at temperatures as high as 2,000 °C in either vacuum or inert gas.

For some fatigue tests in air at high frequencies, a cooling apparatus that uses nitrogen gas was designed. The cool nitrogen gas was generated by passing nitrogen gas through a dewar containing liquid nitrogen. A thermocouple was mounted on the surface of the sample at the gage section to monitor the sample temperature.

A water-cooling line was employed to provide active cooling for fatigue tests in mercury. A copper coil was wound around the mercury container, and water circulation was used to remove heat from the container during fatigue tests, which reduced the sample temperature.
3.2. Ultrahigh-cycle Fatigue

Uniaxial high-cycle fatigue tests were performed using button-head round-bar specimens. The geometry of the specimen, shown in Figure 3-1(a), and the test procedures were in accordance with the ASTM E466 for "Conducting Constant Amplitude Axial Fatigue Tests of Metallic Materials". For 1,000 Hz tests, the gage sections of the test specimens were 5.08 mm in diameter and 15.24 mm in length.

For high-frequency (1,000 Hz) fatigue testing, a state-of-the-art material test system, MTS Model 1,000 Hz 810, was employed. This material test system had a Teststar II controller and operation software. It had high-speed and accurate data-acquisition capabilities. The “voice coil” servovalve enabled the material test system to operate at a frequency range from 20 Hz to 1,000 Hz and a loading capacity of ±25 KN. This high-frequency material test system provided the capabilities to perform the fatigue test under accurate control, and complete a fatigue test of life up to $10^9$ cycles within a reasonable time period (11 days). To avoid the testing noise at 1,000 Hz, the machine was situated in a well-designed, sound-proof room equipped with an air conditioner, which offered the cooling capability to prevent the over-heating of servovalves. The MTS Model 1,000 Hz 810 system was also utilized to conduct fatigue tests at 20 Hz.

High-cycle fatigue tests were performed under load control using a sinusoidal waveform at 20 Hz and 1,000 Hz with a R ratio of 0.1. The specimens were cyclically loaded until
failure or up to approximately $10^7$ cycles as a run-out for the 20 Hz tests, and $10^9$ cycles as a run-out for the 1,000 Hz tests.
3.3. Fatigue Crack-propagation Test in Air

The fatigue specimens used in the study were compact-tension (C(T)) specimens of Type 316 LN SS in annealed condition, with a width of 63.5 mm and a thickness of 6.35 mm (Figure 3-2). Tests were performed in the room temperature air on a electrohydraulic type MTS 810 test machine in load control using a triangular wave form. The stress ratio was 0.1 and the cyclic frequency was 10 Hz. Crack lengths were measured, using a crack opening displacement (COD) gage, and the crack-growth rates (da/dN) were calculated using the secant method of ASTM E647. The stress-intensity factor (K) for each increment of crack growth was computed based on the average crack length for that increment, using the relationship

\[ K = \frac{P}{BW^{1/2}} \left[ f \left( \frac{a}{W} \right) \right] \]

where,

\[ f \left( \frac{a}{W} \right) = \left[ 2 + \left( \frac{a}{W} \right) \right] \left[ c_0 + c_1 \left( \frac{a}{W} \right) + c_2 \left( \frac{a}{W} \right)^2 + c_3 \left( \frac{a}{W} \right)^3 + c_4 \left( \frac{a}{W} \right)^4 \right] \]

P is the load, B is the specimen thickness, a is the crack length, W is the specimen width, c0, c1, c2, c3, c4 are compliance coefficients as defined by ASTM E647-95, and f(a/W) is the K calibration equation.
The fatigue crack growth threshold ($\Delta K_{th}$) was obtained by decreasing $\Delta K$ control. The normalized $K$-gradient was -0.1 mm$^{-1}$ for the decreasing $\Delta K$ control test. The test was stopped when the $\Delta K$ ($\Delta K = K_{\text{max}} - K_{\text{min}}$) value corresponded to an FCP rate of $10^{-7}$ mm/cycle, according to ASTM E647-95. The fatigue crack growth threshold value was measured by performing the decreasing-$\Delta K$ fatigue crack propagation (FCG) test in air. The $\Delta K_{th}$ was determined by a fitted line from the linear regression of $\log(da/dN)$ versus $\log\Delta K$ that corresponds to a growth rate of $10^{-7}$ mm/cycle. The equation of the fitted line is $\log(da/dN) = 98.474 \log(\Delta K) - 116.87$. The fatigue crack growth threshold was measured to be 13.05 MPa$\sqrt{m}$ (8.45x10$^{-10}$ m/cycle). Slightly different relationships between the crack propagation rate and the stress-intensity factor range in the Paris law region of increasing $\Delta K$ and decreasing $\Delta K$ control tests were shown in Figure 3-3, which was contributed to the specimen-to-specimen variability. The fitted Paris law relationships are, respectively,

$$\frac{da}{dN} = 1.966 \times 10^8 \Delta K^{2.628}$$

and

$$\frac{da}{dN} = 1.966 \times 10^8 \Delta K^{2.514}$$

for the increasing $\Delta K$ control test (closed circles) and decreasing $\Delta K$ control test (open circles) (Figure 3-3).
3.4. Thermography

In order to measure the specimen temperature evolution faithfully, a nondestructive evaluation method, thermography, was utilized in some tests, besides the method mentioned before using a thermocouple. The infrared (IR) thermography is the process of detecting the invisible infrared radiation and converting the energy detected into visible light. The resultant image depicts and quantifies the energy being radiated and reflected from the object viewed, and can be transformed into temperature maps. The IR thermography is a convenient technique for developing digital temperature maps from the invisible radiant energy emitted from stationary or moving objects at any distance. There is no surface contact or any perturbation of the actual surface temperature of the objects investigated.

In the present study, a state-of-the-art, high-speed, and high-sensitivity Raytheon Radiance HS® infrared imaging system was used to record the temperature changes during high-cycle fatigue. The IR camera had a 256 x 256 pixels Focal Plane Array (FPA) InSb detector, which was sensitive to 3 – 5 µm wavelength thermal radiation. The camera operated in a snapshot mode and could be externally triggered. In the high-speed mode, i.e., 144 Hz, up to 800 full-frame images can be taken into the memory of the frame-grab card, and then transferred into the hard disk. In the low-speed mode, i.e., 2 Hz or slower, 1,000 full-frame images (about 100 MB) or more can be stored directly into the hard disk. The temperature resolution of the camera was 0.015 °C at 23 °C.
Before fatigue testing, a thin submicroscopic graphite coating was applied to the specimen in order to reduce IR reflections, and the temperature measurement of the IR camera was calibrated using a heat gun, which was used to heat up the specimen. The radiant energy intensities of each specimen were obtained by the IR camera at temperatures ranging from room temperature to a temperature greater than the estimated highest temperature of each fatigue test. Meanwhile, the corresponding temperatures were measured by a thermocouple. The relationship between the IR radiant energy intensity and temperature was established by a best-fit line of the measured results.
3.5. Microstructural Characterization

The observation of the fracture surfaces after fatigue testing can facilitate better understanding of the effects of environment on the fatigue behavior of 316 LN SS. The comparison of the fracture morphologies of specimens tested in air and mercury can give a direct explanation of the different fatigue mechanisms of the material. Therefore, the characterization of the microstructures of 316 LN SS was performed to understand their fatigue behavior.

The Nikon Epiphot and Microphot FX optical microscopes were utilized to examine the microstructure. For sophisticated microstructural characterizations and quantitative microchemical analyses, a scanning-electron microscope (SEM), Model Cambridge S-360, was used to observe fracture surfaces and identify fracture mechanisms. The samples were cut from the specimens at about 12 mm from the fracture surfaces for SEM. The metallographic specimens prepared for the OM and SEM observations were mechanically polished according to the standard procedure. The surface was polished using a 0.05 µm alumina solution.

An etchant of a 5% oxalic acid solution was used for etching. The specimen was used as an anode, and a stainless steel as the cathode was placed at a direct current (DC) potential of 10 volt and 0.6 A for 200 seconds.
For characterizing the dislocation morphology, a Hitachi 800S 200 KeV TEM was employed. Detailed analyses on the dislocation structure were performed under a Philips CM30 TEM with an accelerating voltage of 300.

Disk samples with a thickness of 0.25 mm were sliced from fatigue specimens using a Gillings HAMCO thin sectioning machine. The samples were cut at a position close to the fracture surface of the fatigue specimen. The disk samples were then cut into 3 mm diameter disks with a hand punch. The disc samples were mechanically polished to a thickness of 0.1 mm using 800-grit sand paper, and electropolished by the Struers Tenupol-3 electrolytic polish apparatus at a DC potential of 40 volts, using the solution of 5% perchloric acid and 95% menthanol. This polishing voltage was carefully tested to obtain an optimal value. If the polishing voltage is too low, the electro-polishing effect will be minimal during polishing, and the corrosion will be the dominant process, which has no polishing and thinning effects on the sample. The best polishing current is 0.5 A, and the electrolyte flow rate is set to 5.5. An optimal combination of the polishing voltage, current, and flow rate of electrolyte will help form a small hole with a thin edge at the center of the sample, instead of a big hole with a very thick edge when the polishing condition is not good. The total polishing time is about 20~30 sec.
PART 4

EFFECTS OF ENVIRONMENT ON THE FATIGUE BEHAVIOR OF
TYPE 316 LN STAINLESS STEEL
4.1. Environmental Effect at $R = -1$

Environmental Effect at 0.2 Hz

The test results are shown in Figure 4-1. The general trend of the S-N data was that the fatigue life increased with decreasing the maximum stress level. Considering the fact that fatigue data have a large scatter, the fatigue lives at 0.2 Hz in mercury are comparable with those in air. However, at stress amplitudes above 230 MPa, fatigue tests at 0.2 Hz in mercury generally exhibited shorter lives to a certain extent, as compared with those in air, with the exception of stress levels of 236 MPa and 263 MPa, where some fatigue tests in air and mercury showed comparable cycles to failure. Similar fatigue endurance limits at stress amplitudes of about 210 MPa and 220 MPa were observed for tests in air and mercury.

The fatigue test data were fitted statistically according to the ASTM E739 for “Standard Practice for Statistical Analysis of Linear or Linearized Stress-Life (S-N) and Strain-Life ($\varepsilon$-N) Fatigue Data.” Assuming a linear relationship exists,$^{[1]}$

$$\sigma_a = A \log N_f + B,$$  \hspace{1cm} (1) 

in which $\sigma_a$ and $N_f$ refer to the amplitude of the cyclic stress and the number of cycles to failure, respectively, and A and B are fitting constants. The fits are shown by lines on the
figures, such as Figure 4-1. The run-out data represents specimens that didn’t fail and were suspended after a certain number of cycles. The fits do not involve the run-out data. Horizontal lines were drawn out separately from equation (1) to fit the run-out data, indicating the fatigue endurance limits.

Environmental Effect at 10 Hz

Figure 4-2 presents the fatigue lives in air and mercury at 10 Hz. Shorter fatigue lives as well as a lower fatigue endurance limit were measured in air than in mercury at 10 Hz. For the fatigue test at 10 Hz and $\sigma_a = 263$ MPa, the specimen temperature in air was much greater than that in mercury, approximately 334 °C versus 130 °C, which decreased the yield strength, and thus, the fatigue life in air (Table 3). Note that increasing temperature decreases yield strength in Table 3. By decreasing the specimen temperature to a range of 20 - 60 °C with nitrogen cooling, longer fatigue lives were observed in air than in mercury (Figure 4-2), which was caused by a relative higher temperature in mercury, approximately 130 °C in mercury versus 20 - 60 °C in air. Moreover, the fatigue lives in air with nitrogen cooling were longer than those in air without nitrogen cooling. By controlling the specimen temperature in air with nitrogen cooling and in mercury with water cooling, comparable fatigue lives were measured at 10 Hz (Figure 4-2) with similar specimen temperatures in air and mercury in the range of 20 - 60 °C, although somewhat shorter fatigue lives were observed in mercury at higher stresses (≈ 260 MPa).
4.2. Environmental Effect at $R = 0.1$

Environmental Effect at 10 Hz

Figure 4-3 shows the effect of test environment on fatigue life at 10 Hz. There was no specimen self-heating effect at 10 Hz in air with a $R$ ratio of 0.1. The specimen temperature remained at room temperature throughout the test at all the stress levels. Fatigue tests at the stress amplitude greater than 210 MPa in mercury at 10 Hz have shorter lives as compared with those in air (Figure 4-3). However, for tests at lower stress levels (< 210 MPa), the material has comparable fatigue lives in mercury and air.

Environmental Effect at 700 Hz

Figure 4-4 presents the S-N curves at 700 Hz in air and mercury. Longer fatigue lives in mercury than in air were observed. This trend was caused by the specimen self-heating at 700 Hz, which produced a much higher specimen temperature in air than in mercury. As shown in Figure 4-5, the specimen temperature in air seemed to increase linearly with increasing test frequency. A similar phenomenon was observed for fatigue tests in mercury. However, mercury around the specimen served as a cooling medium. As a result, the specimen temperature only rose to 78 °C in mercury (Figure 4-5). At higher temperatures, the yield strength of 316 LN SS decreases (Table 3), which results in lower fatigue resistance. Comparable fatigue lives in air and mercury were measured after
cooling the specimen in air using nitrogen gas, as shown in Figure 4-6, because of the comparable specimen temperatures in cool nitrogen gas and mercury (Figure 4-5).
4.3. Microstructure Examination of the Crack Initiation and Propagation Process in Air and Mercury

Samples were etched electrolytically using a 10 wt% oxalic acid water solution. The etching voltage is 10 V, current is 0.6 A, and time is 200 s. All of the samples were cut along the longitudinal direction, and were observed under a HITACHI S-3500 scanning electron microscope (SEM). The examination of the microstructure of the sample that failed after 7,872 cycles under constant load control showed uniform austenitic grains (Figure 4-7). The microstructure also showed some annealing twins, but there were no indications of micro-cracks inside the sample. In addition to the main crack, many surface micro-cracks with crack lengths in the scale of 30 - 300 um were found on the surfaces of the samples. Figure 4-8 showed a surface micro-crack propagating inside the sample after 5,159 cycles in air. The micro-crack is straight macroscopically, and remains normal to the stress axis. The propagation is, thus, mainly transgranular. The white spots in the micrograph are pits due to the inhomogeneous etching by the oxalic acid. A more detailed examination of the evolution of surface cracks were shown from Figure 4-9 to Figure 4-12. In Figure 4-9, the morphology of the specimen surface of 316 LN SS before fatigue testing was shown, examined under an optical microscope. The specimen surface was macroscopically smooth before fatigue. The initiations of several surface micro-cracks were shown in Figure 4-10. It is clear that the initiation sites of cracks were randomly distributed on the surface of the specimen tested in air, indicating no preferred region such as grain boundaries. The propagation of a surface micro-crack
during a fatigue test in air was shown in Figure 4-11. The crack path showed little
tortuosity, which is a sign of transgranular cracking. Similarly, the propagation of a
 crack of the specimen tested in mercury showed little tortuosity, and no apparent
 preferred crack initiation in the grain boundaries were observed (Figure 4-12), indicating
 no detrimental environmental effect on the crack initiation process. However, regions
 where the crack changed its propagation path slightly and grew along the grain boundary
could be found in Figure 4-12. This trend suggested that mercury could affect the crack
 propagation by enhancing the crack growth along grain boundaries where selective
 elemental dissolution into mercury occurred during the crack-propagation process, but
 the effect is not significant enough to cause pure intergranular cracking during fatigue.
4.4. Fractography

Fractography at $R = -1$

Typically, the fatigue crack initiation occurs on the specimen surface as a result of the irreversible process of extrusion and intrusion formation through slip deformation. This trend was true for the high-cycle fatigue of 316 LN SS both in air and mercury.

The fracture surface of specimens tested in air and mercury showed a significant difference in fracture modes, especially in the crack-propagation region. Specimens tested in air exhibited typical transgranular (TG) cracking in the crack-propagation region at 0.2 Hz and 10 Hz (Figures 4-13 and 4-15), while those in mercury showed some intergranular (IG) cracking at high stress levels ($= 230$ MPa) (Figures 4-14 and 4-16). At lower stress levels ($< 230$ MPa) in mercury, IG cracking can also be seen on the fracture surfaces, but the amount of IG sites is much less than that at high stress levels.

Fractography at $R = 0.1$

The SEM micrographs of the fracture surfaces of the fatigue specimens tested in air and mercury at a $R$ ratio of 0.1 showed different fracture mechanisms, especially in the crack-growth region of the fatigue fracture surface. Figures 4-17 and 4-18 are micrographs of the typical crack-propagation region of 316 LN SS tested in air and mercury at 10 Hz,
respectively. The specimen tested in air showed typical TG cracking throughout most of the fracture surface (Figure 4-17). In contrast, the specimen tested in mercury exhibited intergranular (IG) cracking at high stress levels (= 210 MPa), as presented in Figure 4-18. At lower stress levels (< 210 MPa) in mercury, IG cracking can rarely be seen on the fracture surfaces. The IG-cracking region is believed to be a direct result of LME. Transgranular fracture was the typical mode observed in air and mercury at 700 Hz.
4.5. Environmental Effect on the Fatigue Lives of Cold-worked Specimen

In contrast to the few-MeV range of neutrons in fission reactors, spallation targets are exposed to protons and neutrons in the GeV range. Radiation effects and transmutation products, such as helium and hydrogen, can cause the target material to lose ductility and harden. The annealed 316 LN SS retains considerable work hardening ability and displays uniform elongations of 25-30% in contrast to the severe embrittlement of the ferritic/martensitic steels, which is one of the reasons why 316 LN SS was chosen to be the target container material.\[2\] Due to the limitation of the facility to test irradiated specimens in the lab, fatigue tests were performed on 20% cold-worked specimens, simulating the mechanical property of the irradiated material. The tensile curve of 20% cold-worked specimens was shown in Figure 4-19. The yield strength of cold-worked 316 LN SS is 760 MPa, which is more than two times higher than that of the annealed specimen. The S-N curves of the 20% cold-worked specimens were shown in Figure 4-20. With the controlled specimen temperature for fatigue tests in air and mercury, the environmental effect was insignificant. Both of the fatigue endurance limits in air and mercury of cold-worked specimens were higher than their corresponding value for annealed specimens.
4.6. Discussion

Environmental Effect at R = -1

At 0.2 Hz

For fatigue tests at stress levels above 230 MPa, although the fatigue lives in air and mercury are overall comparable, but there are still differences in fatigue lives in air and mercury at almost all the stress levels tested (Figure 4-1). Fatigue lives in mercury are relatively shorter than in air except at two stress levels, as discussed previously. Thus, LME is suggested to be the main factor that contributes to the somehow shorter fatigue life of 316 LN SS (Figure 4-2) in mercury, relative to that in air. At higher stress levels, mercury has more chance to interact directly with the fresh surface in the crack-tip area due to the larger plastic deformation, which exhibited the characteristic intergranular fracture in Figure 4-14.

At 10 Hz

With comparable specimen temperatures in air and mercury after cooling, comparable fatigue lives were found in air and mercury (Figure 4-2), although somewhat shorter fatigue lives in air at stress levels below 260 MPa were observed. The small difference in the fatigue lives is most likely caused by the insufficient cooling using nitrogen gas at 10 Hz., compared to the cooling by water for fatigue tests in mercury. As discussed above, even though the specimen was cooled using nitrogen gas, the specimen temperature in air
after cooling is still higher than room temperature, which could have caused a bit shorter lives in air. The shorter lives in mercury with water cooling than in air with nitrogen-gas cooling at stress levels above 260 MPa was due to the environmental effect, as evidenced by the presence of the intergranular fracture in mercury (Figure 4-16).

**Environmental Effect at R = 0.1**

*At 10 Hz*

For fatigue tests at higher stress levels (e.g., = 210 MPa), LME is supposed to be the main factor that decreases the fatigue life of 316 LN SS (Figure 4-3). The environmental effect is more significant at higher stress levels, which can be explained with regard to the greater plastic deformation. The maximum stress level ($\sigma_{\text{max}}$) at a R ratio of 0.1 is 540 MPa, which is well above the yield strength of the material (289 MPa). This means during the first half-cycle, the extensive plastic deformation damages the chromium oxide scale on the specimen and exposes the fresh metal surface to the mercury, which facilitates the LME process. At higher stress levels, mercury may have more chance to interact directly with the fresh surface in the crack-tip area. Thus, there is a greater possibility of better contact of the mercury with the fresh metal surface at higher stresses. Moreover, at higher stress levels, the fatigue life is generally determined by the crack-propagation process. During the crack-growth period at higher stress levels, the crack-opening displacement (COD) is relatively large, which facilitates the penetration of mercury into the crack tip and the contact with the fresh crack surface. Correspondingly,
the penetration depth of the mercury may be increased during a higher-stress fatigue test, and wetting of the freshly cracked surface by mercury may be enhanced. These trends result in the LME effects of mercury at higher stress levels, characteristic of the presence of the intergranular fracture (Figure 4-18).

At low stress levels (< 210 MPa), the fatigue lives in mercury are comparable to those in air as presented in Figure 4-3. This trend is a combination of the result of the lack of the penetration and wetting of mercury at low stresses, which decreased LME.

At 700 Hz
Thermography results showed that temperature might be the dominant factor that contributed to the difference in the fatigue life at 700 Hz in air and mercury (Figure 4-4). For the fatigue test at 700 Hz and $\sigma_a = 198$ MPa with a R ratio of 0.1, the specimen temperature in air was much greater than that in mercury, approximately 270 °C versus 78 °C (Figure 4-5). Mercury serves as a heat sink and effectively lowers the specimen temperature at 700 Hz, which results in longer fatigue lives in mercury than air. In Figure 4-6, similar fatigue lives were measured in air and mercury at 700 Hz by controlling the specimen temperature to a range of 58 - 78 °C using the cool nitrogen gas in air.

In Figure 4-4, decreasing the stress level decreases the difference in the fatigue lives in air and mercury. This trend can be expected to result from the fact that decreasing the
stress reduces the specimen temperature in air,\textsuperscript{[3-8]} which yields a reduced difference in the fatigue lives in air and mercury.
4.7. Conclusions (Table 4)

1. For tests at 0.2 Hz with a R ratio of -1 in mercury at stress amplitudes above 230 MPa, the fatigue life was shorter than in air. Below 230 MPa, fatigue lives in air and mercury were comparable. The effect at high stresses is attributed to liquid metal embrittlement (LME).

2. Without water cooling, fatigue tests at 10 Hz with a R ratio of -1 had shorter lives in air than in mercury due to the temperature effect. With cooling, fatigue tests at 10 Hz in mercury at stress amplitudes above 260 MPa showed shorter lives than in air. Below 260 MPa, fatigue lives in air and mercury were comparable.

3. For tests at 10 Hz with a R ratio of 0.1 and stress amplitudes above 210 MPa, the fatigue lives are shorter in mercury than in air. For lower stress amplitudes, the results were comparable. The effect at higher stress levels is attributed to LME.

4. For tests at 700 Hz with a R ratio of 0.1, the fatigue lives in air with nitrogen cooling are comparable with those in mercury. LME is minimal at 700 Hz.

5. Intergranular fracture was the typical cracking mode in mercury at 0.2 Hz and 10 Hz with a R ratio of -1 at stress amplitudes above 230 MPa. Below 230 MPa in mercury, IG cracking can also be seen on the fracture surfaces, but the amount of IG sites was much
less than that at higher stress levels. Transgranular fracture was observed in air at 0.2 Hz and 10 Hz.

6. For fatigue tests at a R ratio of 0.1, intergranular fracture was the typical cracking mode in mercury at 10 Hz with stress amplitudes above 210 MPa. Below 210 MPa in mercury, transgranular fracture was the typical cracking mode. Transgranular fracture was observed in air at 10 Hz, as well as in air and mercury at 700 Hz.

7. Above all, the present study indicated that the presence of mercury did not generally decrease the endurance limit of Type 316 LN stainless steel in the test conditions investigated.
PART 5

CHARACTERIZATION OF THE TEMPERATURE EVOLUTION
DURING HIGH-CYCLE FATIGUE
5.1. Temperature Changes During Fatigue Experiments

In the present work, a state-of-the-art infrared (IR) thermography system was used to measure the temperature development during the high-cycle fatigue of 316 LN SS (Figure 5-1). The IR thermography system had a high-resolution $256 \times 256$ pixels focal-plane-array (FPA) camera. It had a high-speed digital image acquisition capability of 150 full frames per second, high spatial resolution of 5.4 mm per pixel with microscope attachment, and great temperature sensitivity of 0.015 °C at 23 °C.

Due to the limitation of the measurement range of the IR camera, thermocouples were employed to record the specimen temperature when the specimen temperature was higher than the maximum value allowed by the IR camera.

Throughout the experiments, the temperature of the specimen was recorded by the IR thermography system, and the stress-strain responses were documented by the MTS system. Due to the specimen geometry, the stress was concentrated at the specimen-gage section, and the heat generated in the material was mainly conducted through the axial direction of the specimen. Hence, the highest temperature was always located at the midpoint of the surface of the specimen-gage section. The midpoint temperature was taken as an average temperature over $10 \times 10$ pixels domain around the midpoint of the specimen-gage section. The area of $10 \times 10$ pixels represented 1.49 mm × 1.49 mm of real dimension, and it could vary depending upon the distance between the specimen and
the IR camera. In the present study, the IR camera was kept at a relatively constant distance of about 20 cm from the specimen in order to monitor the whole gage section of the specimen and the shoulder area connecting the grip and gage sections. A thermocouple attached on the back face of the specimen was used for temperature calibration and monitoring approximate temperature variations.

Generally, the temperature evolution during high-cycle fatigue was related to the stress-strain state and fatigue damage. The mean temperature profile of a high-cycle fatigue test typically exhibits four stages: (a) the initial raise due to the heat generation resulting from irreversible deformation of the material; (b) the steady state resulting from a balance between the heat generation and heat conduction inside the material; (c) the abrupt increase due to the large plastic deformation caused by stress concentration at the crack tip; and (d) the final drop, caused by the separation of the test specimen.

In Figure 5-2, the evolution of the mean temperature, which was the average temperature of each cycle, during fatigue were plotted as a function of time. It was observed that as the cycling proceeds at 700 Hz and a maximum stress level of 439 MPa, the specimen temperature initially increased abruptly, and then began to increase slowly until reaching the highest temperature of approximately 270 °C at the time when the specimen failed.

The image in Figure 5-2 is the temperature map of the specimen of the gage section of the specimen. The brightness of color indicates the temperature difference. The brighter
the color, the higher the temperature, as represented by the calibrated color bar of Figure 5-2. It can be seen in Figure 5-2 that the highest temperature indeed was located around the midpoint of the specimen.

The measurements of the temperatures for fatigue tests in mercury were performed by measuring the temperature on the mercury container. A fine black carbon film was painted on the surface of the container beforehand. Since mercury is a very good thermal conductor, the temperature on the container can be assumed to be similar as that on the surface of the specimen. Figure 5-3 showed the temperature map of the container during a 700 Hz fatigue test with a maximum stress level of 439 MPa.

In Figure 5-4, the temperature evolution was plotted as a function of time. It was observed that as the cycling proceeds at 10 Hz and a stress amplitude of 263 MPa, the specimen temperature initially increased abruptly, and then began to increase more slowly until reaching the highest temperature of approximately 350 °C at the time when the specimen failed. During testing at 0.2 Hz, the specimen temperature remained at about 20 °C. Thus, the specimen temperature at 10 Hz was much greater than that at 0.2 Hz, and resulted in shorter fatigue lives at 10 Hz and a significant frequency effect. Table 1 shows that the material strength would have been decreased substantially at the higher temperature.
5.2. Stress versus Strain Curves

The stress versus strain curves of selected cycles for tests at 0.2 Hz and 10 Hz with the same stress amplitude of 263 MPa are shown in Figures 5-5, 5-6, and 5-7, respectively.

Figure 5-5 presents the stress-strain curve (a hysteresis loop) at 10 Hz with a stress amplitude of 263 MPa and a R ratio of -1 without cooling by nitrogen gas. The test results indicated that the inelastic strain increased rapidly with increasing cycles (Figure 5-5). At the beginning of the cyclic loading, the area under the hysteresis loop was relatively small. The area of the hysteresis loop generally increased as fatigue loading continued. As shown in Figure 5-5, the differences between hysteresis loops at the 32nd and 1,024th fatigue cycles were significant. There was still a large amount of plastic strain building up during fatigue, which can be seen by comparing the relative positions of the hysteresis loops at the 32nd and 1,024th cycles. The accumulation of the irreversible plastic strain continued until the end of the test. The shape of hysteresis loops indicate a softening process throughout fatigue tests.

In contrast, the hysteresis loops for tests performed at 10 Hz with nitrogen cooling (Figure 5-6) showed a relatively slower increase of the inelastic strain with increasing fatigue cycles. The shape of hysteresis loops at the 32nd and 1,024th cycles didn’t change much, and the relative positions of the hysteresis loops at the 32nd and 1024th cycles remain almost the same, indicating a softening process is minimal. Similarly, the
hysteresis loops for fatigue tests at 0.2 Hz (Figure 5-7) exhibited a slowly increasing inelastic strain.

The uniaxial tension curve of 316 LN SS showed significant strain hardening behavior, which is contrary to the result shown in Figure 5-5. At a higher temperature of 300 °C, the yield strength is lower than at ambient temperature, as shown in Figure 5-8. The lower yield strengths at higher temperatures obviously compromised the strain hardening behavior, and lead to a softening process during fatigue, as shown in Figure 5-5.

A comparison of the areas of hysteresis loops corresponding to different fatigue cycles at 0.2 Hz and 10 Hz with a stress amplitude of 263 MPa and a R ratio of -1 is shown in Figure 5-9. The areas of hysteresis loops became larger with increasing fatigue cycles at both 0.2 Hz and 10 Hz. However, the areas of hysteresis loops for fatigue tests at 10 Hz in air without nitrogen cooling increased much faster than those with nitrogen cooling in air and those at 0.2 Hz. The areas of hysteresis loops at 10 Hz with nitrogen cooling and at 0.2 Hz are comparable; they are generally two to three times smaller than those at 10 Hz in air without nitrogen cooling. Increasing the number of cycles increases the difference in the areas of hysteresis loops at 10 Hz with nitrogen cooling (or at 0.2 Hz) and at 10 Hz without nitrogen cooling.
5.3. Theoretical Modeling

Fatigue of materials, as a complicated process of gradual accumulation of damage, is mainly controlled by the amplitude of the plastic deformation. Especially, at high cyclic stresses, plastic strain is the predominant cause of energy dissipation. It is well known that the stored energy, such as the energy stored in the dislocations and the elastic energy caused by the interaction of dislocations, is generally only a small portion of the energy during fatigue.\textsuperscript{[10]} The stored energy is the integration of the increase of the internal energy over all the elements in the plastic zone. Compared with the dissipation of thermal energy, it is only a small part of the total mechanical energy consumed during the fatigue process, which can be measured from the stress-strain hysteresis loop.\textsuperscript{[10]}

In materials undergoing cyclic loading, most of the dissipated energy due to the hysteresis effects manifests itself as heat, and the heat is removed from the material by heat transfer. Heat can be transferred by three processes: conduction, convection, and radiation. Conduction is the transfer of heat along a solid object. Convection transfers heat through the exchange of hot and cold molecules, e.g., air, water, etc. Radiation is the transfer of heat via electromagnetic (usually infrared) radiation. If the fatigue experiment is rapid enough, which is generally true for high-cycle fatigue testing, the temperature rise can be surprisingly high. For fatigue tests at 700 Hz, for example, the temperature may rise to 270 °C, depending on test materials and specimen geometries.
The temperature of a specimen changes during fatigue, because of the thermoelastic effect and the inelastic effect. However, the temperature evolution during fatigue has not been well examined. When a material is subjected to cyclic deformation, mechanically-induced strain energy dissipation is strongly dependent on the magnitude and history of the cyclic stress, and on the coupling between mechanical and thermal effects. Most of the dissipated strain energy is converted into heat, manifested by the change in temperature. The temperature evolution is complicated, and the data are hard to interpret not only due to the interrelated effects of thermal and mechanical coupling, strain amplitudes, and loading histories, but also owing to multiple modes of heat transfer from the material to the environment.

Materials tend to have different mechanical behavior with varying temperatures, regardless of the heat source. A theoretical study on the temperature evolution of the specimen during fatigue is necessary to investigate fatigue behavior of materials further. Several factors that influence the specimen temperature during fatigue are the energy-deposition rate due to inelastic processes, the energy-storage rate in materials, and the heat-dissipation rate to the surrounding environment. A model combining these factors is discussed in the following. The temperature evolution of the specimen was predicted and compared with the experimental results.

To study the temperature evolution of a homogeneous round-bar specimen, the following assumptions have been made.
a) The specimen is subjected to a one-dimensional uniaxial stress. The deformation is limited to the gage section of the test sample.

b) The heat-conducting part of the specimen has a uniform conductivity.

The coordinate system for heat transfer in the specimen is shown in Figure 5-10 with the x-axis along the length of the specimen. The end of the gage section corresponds to a point, x = 0 (Figure 5-10). The cross-hatched area indicates the half-length of the gage section.

To derive an equation for the temperature variation with time in the gage-section area, two kinds of heat-transfer effects were considered. One effect is the heat flow by conduction from the gage-section area into the other part of the specimen and the grips of the machine. Another effect is the heat convection with air from the surface of the gage-section area of the specimen.

The gage section is assumed to have a uniform temperature (Biot modulus = 0.02 < 0.1).\(^{[11]}\) Note that Biot modulus = \( \frac{hV}{Ak} \), where \( h \) is the convective heat coefficient of 15 w/m\(^2\)K of 316 LN SS, \( k \) is the thermal conductivity of 16.2714 w/mK, \( V \) is the volume of the gage section of 926.85 mm\(^3\), and \( A \) is the surface area of the gage section of 45.61 mm\(^2\). A small value for Biot modulus indicates that the controlling heat transfer phenomenon is convection, and temperature gradients within the medium are quite small. Thus, to simplify the problem, the gage section can be assumed to have a uniform temperture.
temperature, since the temperature gradient between center of the specimen and the surface is quite small.

The heat balance in the gage section can be expressed as follows:\textsuperscript{12}\textsuperscript{\[12\]}

\[ q = q_c + q_d + q_s \] (5-1)

\( q \) is the heat-generation rate due to the inelastic deformation of the specimen;
\( q_c \) is the heat-convection rate with air from the surface of the gage-section area;
\( q_d \) is the heat-flow rate by conduction from the gage-section area into the other part of the specimen and the grips of the machine;
\( q_s \) is the heat-storage rate in the specimen.

In the tension-tension, high-cycle fatigue test, the temperature evolution corresponded to the change of the stress-strain state. It has been experimentally found that the stored energy is only a small amount of dissipated energy.\textsuperscript{10}\textsuperscript{\[10\]} Hence, almost all the irreversible mechanical energy due to the inelastic deformation will be converted into heat. The mechanical energy dissipated in each cycle can be represented by the area of the hysteresis loop. The increment of the mean temperature was proportional to the area of hysteresis loop. From the first law of thermodynamics, conservation of energy, assuming the specimen was stressed in an adiabatic condition, and ignoring the energy stored inside the material,
\[ \rho C_v \Delta T = \int_{\varepsilon_1}^{\varepsilon_2} \sigma_u d\varepsilon - \int_{\varepsilon_1}^{\varepsilon_2} \sigma_l d\varepsilon \]  

(5-2)

where \( \varepsilon_1 \) and \( \varepsilon_2 \) are the minimum and maximum strains of the hysteresis loop, and \( \sigma_u \) and \( \sigma_l \) refer to the local stresses in the upper and lower curves of the hysteresis loop, respectively.

As stated in Equation (5-2), the increment of the mean temperature per cycle is proportional to the area of the hysteresis loop. As shown in Figures 5-5, initially the area of the hysteresis loop increased slowly, and, thus, the temperature increment was relatively low. However, the rate of the increment of the mean temperature per cycle increased as the fatigue process progressed due to the increasing plastic deformation.

The heat-generation rate can be expressed as

\[ q = \frac{dG}{dt} = \frac{V \int \sigma d\varepsilon}{\Delta t} \]  

(5-3)

where \( G \) is the total energy generation in a fatigue cycle, \( V \) is the volume of the gage section of the specimen, \( \int \sigma d\varepsilon \) is the area of the hysteresis loop in a cycle, and \( \Delta t \) is the period of time in a cycle.

The heat-convection rate is \(^{[12]}\)
\[ q_e = 2\pi r_0 L \bar{h}[T - T_0] \]  \hspace{1cm} (5-4)

where \( r_0 \) is the radius of the gage-section area, \( L \) is the length of the gage section, \( \bar{h} \) is the average convective heat-transfer coefficient, \( T \) is the temperature of the gage-section area of the specimen, and \( T_0 \) is room temperature.

The heat-conduction rate is \(^{[12]}\)

\[ q_d = -kA \left. \frac{\partial T}{\partial x} \right|_{x=0} \]  \hspace{1cm} (5-5)

where \( k \) is the thermal conductivity of the specimen, and \( A \) is the cross-sectional area of the gage section.

Using Equation (5-5), \( q_d \) can be determined from the solution, \( \frac{\partial T}{\partial x} \), to the following unsteady state heat-conduction equation, \(^{[12]}\) given the initial condition of \( t = 0, T = T_0 \), and the boundary conditions of \( x = L_c, T = T_0; x = 0, T = T_g \).

\[ \frac{\partial^2 T}{\partial x^2} = m \frac{\partial T}{\partial t} \]  \hspace{1cm} (5-6)
where \( m = \rho c / k \), \( \rho \) is the density of the material, \( c \) is the specific heat of the material, \( L_c \) is the theoretical length of the heat-conduction part, which can be expressed as \( K_c(L_t - L)/2 \). \( L_c \) represents the distance from position \( x = 0 \) to the place where the temperature of the specimen reaches room temperature. It is a value to be fitted with one of the experimental curves. \( K_c \) is a specimen-shape factor that can be determined by fitting a predicted temperature curve with one of the experimental curves, \( L_t \) is the total length of the specimen, and \( T_g \) is the temperature of the gage section at \( x = 0 \).

The boundary condition is not enough to solve Equation (5-6). Assuming that the temperature gradient at \( x = 0 \) can be expressed as,

\[
\frac{\partial T}{\partial x}
\bigg|_{x=0} = \frac{T_0 - T}{L_c} \tag{5-7}
\]

then

\[
q_a = -kA \frac{\partial T}{\partial x}
\bigg|_{x=0} = kA \frac{T - T_0}{L_c} \tag{5-8}
\]

The heat-storage rate is \(^{[12]}\)

\[
q_s = \rho V_c \frac{dT}{dt} = \rho V_c \frac{\Delta T}{\Delta t} \tag{5-9}
\]
Combining Equations (2), (3), (4), (8), and (9), the heat-balance equation becomes

\[
\frac{V}{\Delta t} \int \sigma \varnothing \, d\epsilon = 2\pi r_0 L_\pi \left[ T - T_0 \right] + k A \frac{T - T_0}{L_c} + \rho V c \frac{\Delta T}{\Delta t}
\]

(5-10)

The constants used in the Equations (5-2) - (5-10) are listed below:

L:  Gage length of the specimen, 20.32 mm
L_t:  Total length of the specimen, 228.6 mm
A:  Cross-sectional area of the gage section, 45.61 mm²
V:  Volume of the gage section, 926.85 mm³
\( \rho \):  Density of 316 LN SS, 7.8 g/cm³
\( c \):  Specific heat of 316 LN SS, 0.46 J/g°C
k:  Thermal conductivity of 316 LN SS, 16.2714 w/mK
\( K_c \):  Specimen-shape factor, 4.67 x 10⁻⁶
\( L_c \):  Theoretical length of the heat-conduction part, 4.86 x 10⁻⁷ m
\( h \):  Convective heat coefficient of 316 LN SS, 15 w/m²K

The fitted value of \( L_c \) is very small, which indicates a very large temperature gradient from the end of the gage section to the other part of the specimen. Physically, this is not likely to be a good representation of the temperature distribution near the end of the gage section during a fatigue test. Thus, the approximation of assuming a uniform temperature
in the gage section is too rough to accurately describe the temperature profile in this part. A physical model using a finite element method or a two-dimensional numerical solution could provide a better solution to the problem. The temperature evolution can be solved using Equation (5-10) as a function of time by inputting the area of each hysteresis loop, expressed as $\int \sigma d\epsilon$, which is dependent upon time during fatigue. Figure 5-11 shows the predicted and measured temperature evolutions of 316 LN SS tested at 10 Hz in air with a stress amplitude of 287 MPa and a R ratio of -1. The predicted and experimental temperature-evolution curves are in good agreement.

The change of temperature during fatigue was related to the dissipated energy given by the hysteresis loop. If the boundary conditions are known, the variation of the mean temperature from cycle to cycle can be determined from the dissipated energy. The temperature evolution during high-cycle fatigue is related to the testing frequency, specimen geometry, and loading conditions, etc.
5.4. Discussion

Because of the inelastic effect caused by plastic deformation of the specimen during fatigue tests, the specimen temperature tends to increase.\cite{1-9} At higher frequencies, the deformation rate of the specimen is greater than at lower frequencies, and so is the energy-generation rate. Furthermore, the hysteresis-loop areas and the inelastic strains in air at 10 Hz are much greater than those at 0.2 Hz (Figures 5-9), which induces more heat for each cycle at 10 Hz than at 0.2 Hz. When the heat-loss rate to the surrounding environment is not high enough to compensate for the higher energy-generation rate with increasing frequency, the energy-storage rate in the specimen will be greater, which leads to a higher temperature of the specimen at a greater frequency.

It can also be seen from the stress-strain curves (hysteresis loops) of the test results that there are a larger maximum inelastic strain and greater hysteresis-loop areas for fatigue tests at 10 Hz without nitrogen cooling due to the specimen self-heating, compared with the test results at 0.2 Hz (Figure 5-6, 5-7, and 5-9). In Figure 5-5, 5-6, and 5-9, there are much greater inelastic strains and hysteresis-loop areas at 10 Hz without nitrogen cooling than with nitrogen cooling, which correlates with the significant self-heating in the specimen without nitrogen cooling. The inelastic strains and hysteresis-loop areas for tests performed at 10 Hz with nitrogen cooling (Figure 5-6) and at 0.2 Hz (Figure 5-7) are comparable at the same number of fatigue cycles (Figure 5-9). Therefore, the higher temperature during fatigue at 10 Hz without nitrogen cooling induced a larger plastic
deformation of the material due to the smaller yield strength, which leads to a shorter fatigue life at 10 Hz than at 0.2 Hz. Consistently, the fatigue lives at 10 Hz without nitrogen cooling were shorter than those with nitrogen cooling. Moreover, the fatigue lives at 10 Hz with nitrogen cooling and at 0.2 Hz were comparable, which resulted from comparable inelastic strains, hysteresis-loop areas, and specimen temperatures at both frequencies.
5.5. Conclusion

It is well known that a material changes its temperature because of the mechanical deformation. Different means, such as thermocouples and thermography techniques, have been employed to monitor the temperature changes during mechanical tests. Most of the energy manifests itself as heat. The generated heat is mainly removed from the material by conductive and convective heat transfer. It has been of interest in the past to study the relationship between heat generation and fatigue behavior. In air, without cooling the specimen, increasing the test frequency from 0.2 Hz to 10 Hz at a R ratio of -1 decreased the fatigue life. The effect is attributed to the specimen self-heating at the higher frequency. Fatigue results showed that by holding the specimen temperature in the range from room temperature to about 70 °C with nitrogen gas cooling at 10 Hz, there was no decrease in fatigue life compared with 0.2 Hz tests. Theoretical modeling to predict the specimen-temperature evolution during fatigue tests was carried out. The predicted and measured temperature evolutions were comparable.
PART 6

EFFECTS OF FREQUENCY ON FATIGUE BEHAVIOR OF TYPE 316 LN STAINLESS STEEL IN AIR AND MERCURY
6.1. Introduction

Fatigue behavior is strongly affected by the environment, material, and loading conditions.\cite{1} As a candidate target-container material of the Spallation Neutron Source (SNS) being designed and constructed at the Oak Ridge National Laboratory, Type 316 low-carbon, nitrogen-added (LN) stainless steel (SS) is required to have good fatigue resistance under severe working environments. The radiation damage to the container is caused by both the incoming high-energy protons and the spallation neutrons produced in the mercury by the proton beam. The SNS will operate in a pulsed mode, with a proton beam pulse frequency of 60 Hz. These pulsed fluxes also lead to pressure waves in the mercury created by beam heating, which in turn produce time varying stresses that cause fatigue loading of the vessel at different frequencies and loading modes.\cite{2}

The stress mode varies at different parts of the container. Two of the most common loading conditions are the tension-tension and tension-compression stress modes. Thus, it is important to study the fatigue behavior of Type 316 LN SS under these stress modes. However, most mechanical data about 316 LN SS have been gathered in monotonic loading conditions, and relatively less information for fatigue loading conditions has been obtained.\cite{3,4} Most of the investigations on the frequency effect have been done at relatively low frequencies using strain-controlled fatigue tests, and those studies at high frequencies using ultrasonic fatigue machines have been focusing on the problem of elastic cyclic loading.\cite{5-7} The typical specimens used in the study of the frequency effect
on the fatigue life are compact-tension specimens, and relatively few investigations have been performed using smooth-bar specimens.\cite{8-18} In addition, a variety of materials were used in the study of the frequency effect including stainless steel.\cite{5-7,10-19} However, there is almost no information about the influence of frequency on the fatigue life of 316 LN SS in mercury, which is a central question for the SNS target container.\cite{2}

This part discussed the fatigue behavior of 316 LN SS in air and mercury at test frequencies of 0.2 Hz and 10 Hz with a R ratio of -1, and at 10 Hz and 700 Hz with a R ratio of 0.1. Microstructural characteristics of fracture surfaces, such as striation line spacing, were studied carefully to understand the high-cycle fatigue behavior of 316 LN SS at different frequencies.
6.2. Cooling Methods

The high-cycle fatigue tests were basically performed the same way as described in the previous PARTs. However, as mentioned in the previous PART, the self-heating effect of specimens during certain test conditions are quite significant. Therefore, the more complicated job was to efficiently control the specimen temperature during fatigue tests. For some fatigue tests in air at high frequencies, a cooling apparatus that used nitrogen gas was employed. When the specimens were exposed to the air environment, a cooling media can be directly applied to the surface of the specimen. Thus, the compressed nitrogen gas was employed as a cooling media, considering its inert property and the feasibility. To enhance the cooling efficiency, the nitrogen gas was cooled before it was applied to the specimen. A copper coil was immersed under the liquid nitrogen contained in a dewar, and nitrogen gas was cooled down by passing through the coil. The gas pressure had to be controlled manually to keep a constant specimen temperature during the fatigue process. Two thermocouples were used, one attached on the specimen gage section and one positioned near the notch of the cooling ring, to observe the specimen temperature and the gas temperature when it came out of the dewar.

A water-cooling line was employed to provide active cooling for fatigue tests in mercury. Since the specimen was surrounded by the body of mercury, an indirect cooling approach had to be used. A copper coil was wound around the wall of the mercury container, and
water circulation was used to remove heat from the container during fatigue tests, which reduced the sample temperature.
6.3. Fatigue Behavior in Air

At 0.2 Hz and 10 Hz with a R Ratio of -1

Fatigue tests with a R ratio of -1 were conducted to observe the frequency effect in air at 0.2 Hz and 10 Hz. As shown in Figure 6-1, the fatigue life at 10 Hz was shorter than that at 0.2 Hz for a given stress level. A greater fatigue-endurance limit was observed at 0.2 Hz than 10 Hz. The fatigue-endurance limits of 316 LN SS were approximately 220 MPa and 200 MPa at 0.2 Hz and 10 Hz, respectively.

The temperature measurement showed that the highest temperature for a fatigue test at 10 Hz in air with a stress amplitude of 263 MPa and a R ratio of -1 was about 350 °C (Figure 6-2). The specimen temperature went up abruptly at the beginning of the test, and then increased gradually until it reached the maximal value when the specimen failed (Figure 6-2). For a test conducted at 0.2 Hz, the specimen temperature was about 15 °C throughout the experiment (Figure 6-2). Thus, the specimen temperature at 10 Hz was significantly greater than that at 0.2 Hz, which resulted in lower strength and shorter fatigue lives at 10 Hz (Figure 6-1). Note that increasing the temperature from 24 °C to 500 °C significantly decreases the strength level of 316 LN SS.

To study the strain-rate effect, the specimens were cooled by flowing nitrogen gas and kept at about 20 °C - 70 °C during fatigue tests at 10 Hz. As presented in Figure 6-1, the
fatigue results at 10 Hz and 0.2 Hz in air were comparable after controlling the specimen temperature at 10 Hz. There was no significant strain-rate effect. Therefore, the difference in the fatigue life in air at 10 Hz and 0.2 Hz was primarily caused by the temperature effect at 10 Hz.

**At 10 Hz and 700 Hz with a R Ratio of 0.1**

The fatigue results at 10 and 700 Hz are presented in Figure 6-3. At the stress amplitude levels above ~ 160 MPa, the fatigue life at 700 Hz was shorter than that at 10 Hz for a given stress level. In addition, the difference in the fatigue life between 10 Hz and 700 Hz appears to decrease with decreasing stress amplitude. The fatigue-endurance limits at 10 Hz and 700 Hz are comparable at approximately 150 MPa in Figure 6-3. Thus, the test frequency appears to be an important factor that influences the fatigue life at stresses above the fatigue-endurance limit.

Figure 4-5 shows how the specimen temperature varied during load-controlled fatigue tests at different frequencies with a stress amplitude level of 198 MPa. The specimen temperature increased linearly with the test frequency. In air, the specimen temperature was about 245 °C higher at 700 Hz than that at 10 Hz. The specimen self-heating was the dominant factor that reduced the fatigue life as will be shown below, because the specimen temperature in air reached 270 °C during fatigue tests at 700 Hz. Tensile tests of 316 LN SS at different temperatures showed a significant decrease of the yield
strength with increasing temperatures from 24 °C to 500 °C (Table 3). This result is consistent with the different fatigue lives of 316 LN SS at 10 Hz and 700 Hz observed here.

To prove the above hypothesis, fatigue tests were performed at high frequencies with the specimen being cooled. The cooling media was flowing nitrogen gas. As shown in Figure 4-5, by cooling with nitrogen gas, the specimen temperature was kept between 20 and 70 °C during 700 Hz fatigue tests. The fatigue lives of specimens in air were generally longer for the tests conducted at 700 Hz with cooling of nitrogen gas, as compared to those without cooling, and were comparable with those at 10 Hz in air (Figure 6-3). However, there are still some differences between fatigue lives at 700 Hz with cooling and those at 10 Hz, which are believed to be due to small temperature differences caused by insufficient cooling at 700 Hz.
6.4. Fatigue Behavior in Mercury

At 0.2 Hz and 10 Hz with a R Ratio of -1

Figure 6-4 shows the S-N curves in mercury at 0.2 Hz and 10 Hz. The difference in fatigue life at 0.2 Hz and 10 Hz is negligible in mercury, which is in contrast with the air-environment results shown in Figure 6-1. This trend is associated with the fact that the specimen temperature during 10 Hz fatigue tests in mercury was reduced to 130 °C, relative to 350 °C observed during 10 Hz fatigue tests in air (Figure 6-2). Although there were only minimal differences in fatigue lives at 10 Hz and 0.2 Hz, a shorter fatigue life was still observed at 10 Hz than 0.2 Hz for higher stress amplitudes (= 230 MPa), which is due to the different temperatures (130 °C and 15 °C) at 10 Hz and 0.2 Hz, respectively (Figure 6-2). By further controlling the specimen temperature to 20 °C - 40 °C in mercury at 10 Hz using water cooling, fatigue lives at 10 Hz are further improved in comparison with those without water cooling (Figure 6-4). For stress amplitudes less than 270 MPa, fatigue lives at 0.2 Hz still seem to be somewhat shorter than at 10 Hz.

At 10 Hz and 700 Hz with a R Ratio of 0.1

Figure 6-5 shows the S-N curve of fatigue tests in mercury at 10 Hz and 700 Hz. The difference in fatigue life at 10 Hz and 700 Hz is smaller in mercury, compared to the air-environment results shown in Figure 6-3. This trend is associated with the fact that the...
specimen temperature at the stress amplitude of 198 MPa during 700 Hz fatigue tests in mercury was reduced to 78 °C, relative to 270 °C observed during 700 Hz fatigue tests in air (Figure 4-5). The mercury around the specimen served as a good heat sink and decreased the specimen temperature. In Figure 6-5, the influence of frequency on the specimen temperature in mercury is much less than that in air because of the much better heat sink provided by mercury than air.

During the 700 Hz fatigue tests in mercury, the specimen temperature at 700 Hz only reached 78 °C because the presence of mercury, which is comparable with that at 10 Hz, no cooling procedure was conducted.
6.5. Fractographs in Air

At 0.2 Hz and 10 Hz with a R Ratio of -1

Figure 6-6 presented the initiation and propagation areas of the fracture surfaces for specimens tested in air at 0.2 Hz and 10 Hz, respectively. The specimens exhibited transgranular fracture, with the crack initiation site on the specimen surface. Figure 6-7 shows the typical striation lines in the propagation areas of the fracture surfaces for specimens tested in air at 0.2 Hz and 10 Hz. At both frequencies, the striation lines are clear and uniform throughout the fracture surfaces of specimens. Specimens tested at 10 Hz in air with and without cooling showed same morphology of the fracture surface.

At 10 Hz and 700 Hz with a R Ratio of 0.1

Figure 6-8 presents the initiation and propagation areas of the fracture surfaces for specimens tested in air at 10 Hz and 700 Hz, respectively. The specimen tested in air showed typical transgranular (TG) cracking throughout the fracture surface, and the crack initiation site is on the surface of the specimen. The specimens tested at 10 Hz exhibited a rougher surface than those at 700 Hz due to the branching of cracks. Figure 6-9 shows micrographs of the typical crack-propagation regions for air tests at 10 Hz and 700 Hz, respectively. The fracture surface at 10 Hz showed clearer striation lines than at 700 Hz.
Specimens tested at 700 Hz with and without cooling showed same fracture modes on the fracture surface.
6.6. Fractographs in Mercury

At 0.2 Hz and 10 Hz with a R Ratio of -1

Figure 6-10 presents the initiation and propagation areas of the fracture surfaces for specimens tested in mercury at 0.2 Hz and 10 Hz, respectively. The surfaces of fatigue specimens tested in mercury (Figure 6-10) exhibited a more brittle appearance than those in air (Figure 6-6). Figure 6-11 shows the small intergranular-like facets in the propagation areas of the fracture surfaces for specimens tested in mercury at 0.2 Hz and 10 Hz. Specimens tested at 10 Hz in mercury with and without cooling showed the same morphology of the fracture surface. Figure 6-12 shows striation lines in the propagation areas of the fracture surfaces for specimens tested in mercury at 0.2 Hz and 10 Hz. The striations lines are not as clear and uniform as those observed in air (Figure 6-7).

At 10 Hz and 700 Hz with a R Ratio of 0.1

Figures 6-13 presents the initiation and propagation areas of the fracture surfaces for specimens tested in mercury at 10 Hz and 700 Hz, respectively. Figure 6-14 shows micrographs of the crack-propagation regions for mercury tests at 10 Hz and 700 Hz, respectively. The specimens tested in mercury showed typical intergranular (IG) cracking on the fracture surface at 10 Hz. The specimens tested at 700 Hz exhibited a brittle surface.
6.7. Discussion

Studies of frequency effects on the high-cycle fatigue behavior of materials have been conducted by a number of researchers.\textsuperscript{[21-25]} The influence of frequency on the fatigue-crack propagation behavior of materials has also been studied under various test conditions.\textsuperscript{[26-30]} Increasing test frequency has been reported to both increase and decrease lifetimes, as well as to have negligible effects on the fatigue lives of materials.

Thus, the apparent inconsistencies arise in previous works regarding frequency effects on fatigue behavior. Reasons for the inconsistencies can be due to different test conditions and materials. However, it is important to note that when considering the frequency effect, two factors are important, specimen self-heating and strain-rate effects. Self-heating of materials will produce different specimen temperatures at various frequencies. An efficient technique of holding the specimen at the same temperature is necessary to investigate solely the strain-rate effect that is associateded with the term, $f$, in Equations (2) by eliminating the temperature effect. Ignoring the specimen self-heating during high-cycle fatigue tests may lead to entirely different test results and conclusions of the frequency effect.

The present work took into account of specimen self-heating, and studied the frequency effect in air and mercury at 0.2 Hz and 10 Hz with a $R$ ratio of -1, and at 10 and 700 Hz with a $R$ ratio of 0.1.
Fatigue Behavior In Air

At 0.2 Hz and 10 Hz with a R ratio of -1

A significant increase in the specimen temperature was found for fatigue tests at 10 Hz with a R ratio of -1 in air, which was associated with the inelastic effect during cyclic loading.\textsuperscript{[31-41]} The direct result was a shorter fatigue life at 10 Hz than at 0.2 Hz with specimen temperatures, at a stress amplitude of 263 MPa, being 350 °C and 15 °C at 10 Hz and 0.2 Hz, respectively, which decreased material strength and, thus, fatigue resistance due to the greater specimen temperature at 10 Hz (Figure 6-2). By controlling the specimen temperature at 20 °C - 70 °C using cooled nitrogen gas, comparable fatigue lives were measured at 0.2 Hz and 10 Hz (Figure 6-1). The large frequency effect was primarily caused by significant self-heating of specimens at 10 Hz than at 0.2 Hz. With similar specimen temperatures at 10 Hz by nitrogen cooling and at 0.2 Hz, the frequency effect was minimal (Figure 6-1).

At 10 Hz and 700 Hz with a R ratio of 0.1

The specimen self-heating effect has also been observed during 700 Hz tests in air with a R ratio of 0.1. For the fatigue test at 700 Hz and $\sigma_a = 198$ MPa with a R ratio of 0.1, the specimen temperature in air was much greater than at 10 Hz during tension-tension fatigue tests, approximately 270 °C versus 20 °C (Figure 4-5). After cooling the specimen in air, fatigue lives were greatly improved at 700 Hz (Figure 6-3). However, relatively shorter fatigue lives at 700 Hz with cooling than those at 10 Hz can still be
observed (Figure 6-3), which could be caused by a higher specimen temperature at 700 Hz (max. 70 °C) due to an insufficient cooling.

**Fatigue Behavior In Mercury**

*At 0.2 Hz and 10 Hz with a R ratio of -1*

Due to the inelastic effect, an increase in the specimen temperature was also found for fatigue tests at 10 Hz with a R ratio of -1 in mercury (Figure 6-2). With the presence of the mercury pool in the container working as a heat sink, however, the temperature increase (130 °C) in the specimen was not as significant as in air (350 °C) at 10 Hz (Figure 6-2). As a result, the temperature rise is not so great at 10 Hz in mercury, which gives decreased frequency effects in mercury than in air (Figures 6-1 and 6-4). The shorter fatigue lives in mercury at 10 Hz than 0.2 Hz were only found at higher stress levels (= 230 MPa) induced by the temperature effect (130 °C at 10 Hz versus 15 °C at 0.2 Hz). However, at lower stress levels (< 230 MPa), the temperature increase is generally lower, and, thus, there were comparable temperatures at 10 Hz and 0.2 Hz, which resulted in similar fatigue lives at lower stresses (Figure 6-4).

By further controlling the specimen temperature to a range of 20 °C - 40 °C in mercury using water, fatigue lives at 10 Hz are further improved in comparison with those without water cooling, resulting from a decreased temperature effects (Figure 6-4). For stress amplitudes less than 270 MPa, fatigue lives at 0.2 Hz still seem to be somewhat shorter.
than at 10 Hz, which is due to a greater environmental effect at 0.2 Hz. At 0.2 Hz, the
time-dependent environmental effect is the dominant factor that determines the fatigue
life rather than the cycle-dependent mechanical damage, since the time between each
cycle during fatigue is longer than at 10 Hz. Thus, for lower stress levels (< 270 MPa),
mercury at 0.2 Hz has longer time to stay in the open crack on the surface of the
specimen and wet the crack tip than at 10 Hz, which results in the more significant
environmental effect [i.e., the liquid-metal embrittlement effect (LME)] and shorter
fatigue lives at 0.2 Hz (Figure 6-4). However, at high stress levels around 270 MPa,
fatigue lives at 0.2 Hz and 10 Hz are so short that the environmental effect does not have
enough time to take place before the specimen fails, which results in comparable fatigue
lives at 0.2 Hz and 10 Hz. Note that the LME effect is evidenced by the presence of the
intergranular fracture in Figure 6-11.

At 10 Hz and 700 Hz with a R ratio of 0.1
Shorter fatigue lives in air were observed at 700 Hz than at 10 Hz, which was primarily
caused by the higher temperature at 700 Hz (Figure 6-5). However, the differences in
specimen temperatures at 700 Hz (78 °C) and 10 Hz (20 °C) in mercury are not very
significant (Figure 4-5). Consistently, there is a minimal frequency effect at 10 Hz and
700 Hz in mercury (Figure 6-5), which is in contrast with the significant frequency effect
in air (Figure 6-3). This trend results from the fact that mercury is a good cooling media,
which reduces the specimen temperature at 700 Hz.
Fractographs in Air

The fracture morphologies in air at 0.2 Hz, 10 Hz, and 700 Hz and in mercury at 0.2 Hz and 10 Hz exhibit the presence of striations (Figure 6-7, 6-9, and 6-12). The striation spacing was measured using specimens tested at 0.2 Hz, 10 Hz, and 700 Hz in air and mercury. The striation spacings were determined by averaging striation spacings at a specific depth from the crack-initiation site. The stress-intensity factor at a specific depth from the crack-initiation site was calculated using the Forman and Shivakumar equation,[30]

\[
K_i = \sigma \sqrt{aF(\lambda)}
\]  

(3)

where \( \lambda = a/D \), \( a \) = the crack depth, \( D \) = the gage diameter of the specimen, and \( F \) = the boundary-correction factor, defined by

\[
F(\lambda) = g(\lambda)\left\{0.752 + 2.02\lambda + 0.37[1 - \sin(\pi\lambda/2)]^3\right\}
\]

(4)

where

\[
g(\lambda) = \frac{1.84}{\pi \cos(\pi\lambda)} \sqrt{\tan(\pi\lambda/2)} \]

(5)
Therefore, the striation spacing can be plotted as a function of $\Delta K$ in air and mercury at different frequencies and R ratios, as described below.

At 0.2 Hz and 10 Hz with a R ratio of -1

There was no obvious difference in the fracture mode in air at 0.2 Hz and 10 Hz. Fracture surfaces also showed a similar morphology at both frequencies (Figures 6-6 and 6-7). The results of striation spacing versus $\Delta K$ are shown in Figure 6-15. The calculated stress-intensity-factor range at the beginning of the crack propagation region from the high-cycle fatigue test at 10 Hz in air is consistent with the measured fatigue crack growth threshold from the FCG test (Figure 3-3), which can shed a light on the fatigue initiation behavior at very low $\Delta K$ levels for high-cycle fatigue tests in air and mercury. The striation spacing representing the crack-growth rate at 10 Hz in air is generally greater than that at 0.2 Hz for a given $\Delta K$. In particular, the difference in the striation spacing (or the crack-growth rate) in air at 0.2 Hz and 10 Hz increases with increasing $\Delta K$ (Figure 6-15). This trend is consistent with the greater specimen temperature in air at 10 Hz than at 0.2 Hz, which results in faster crack-growth rates and shorter fatigue lives at 10 Hz (Figure 6-1). After cooling the specimens using nitrogen gas, the striation spacing in air at 10 Hz is comparable with that at 0.2 Hz (Figure 6-15), which results in comparable crack-growth rates and fatigue lives (Figure 6-1) at 10 Hz with nitrogen cooling and 0.2 Hz in air.
At 10 Hz and 700 Hz with a R ratio of 0.1

The striation spacing at 700 Hz in air seemed to be greater than that at 10 Hz for a certain ΔK (Figure 6-16). This trend can be explained with the influence of the specimen temperature at different test frequencies. For fatigue tests at 700 Hz in air, the specimen temperature is much higher than that at 10 Hz, approximately 270 °C at 700 Hz versus 20 °C at 10 Hz (Figure 4-5). Therefore, the greater specimen temperature in air at 700 Hz than 10 Hz could lead to the larger striation spacing (faster crack-growth rates) at 700 Hz, which could contribute to the shorter fatigue life at 700 Hz (Figure 6-3). The measurement of striation spacing at 700 Hz with nitrogen cooling was attempted. However, the striation spacing on the fracture surface is not well defined for the measurement.

Fractographs In Mercury

At 0.2 Hz and 10 Hz with a R ratio of -1

The striation spacing at 10 Hz in mercury, however, is the same as that at 0.2 Hz (Figure 6-15). This trend, as described previously, can be explained with the influence of the specimen temperature at different test frequencies. For fatigue tests conducted in mercury, the temperature difference of the specimens at 0.2 Hz and 10 Hz was not significant. For the fatigue test in mercury at 10 Hz and σₘₐₓ = 230 MPa with a R ratio of -1, the specimen temperature was about 70 °C at 10 Hz versus 15 °C at 0.2 Hz. With a minimal temperature effect, the striation spacings were comparable at 10 Hz and 0.2 Hz.
in mercury (Figure 6-15), which results in approximately the same fatigue lives at both frequencies (Figure 6-4).

At 10 and 700 Hz with a R ratio of 0.1

The striation spacings at 10 Hz and 700 Hz in mercury were not compared because there were no clear striation lines on the fracture surfaces at 700 Hz.

Thus, the fracture morphologies seem to be closely related to the fatigue lives in air and mercury at different frequencies.
6.8. Conclusions

1. The effects of frequency are summarized in Table 5. In air, without cooling the specimen, increasing the test frequency from 0.2 Hz to 10 Hz at a R ratio of -1 decreased the fatigue life. The effect is attributed to specimen self-heating at the higher frequency of 10 Hz. Fatigue results in air showed that by holding the specimen temperature at about room temperature with cooled nitrogen gas at 10 Hz, the frequency effect was negligible at 10 Hz and 0.2 Hz in air.

2. Specimen self-heating is observed at the high frequency of 700 Hz in air with a R ratio of 0.1, which decreased the fatigue life at 700 Hz, relative to that at 10 Hz. By keeping the specimen temperature at about room temperature with cooled nitrogen, the frequency effect was minimized. The observable difference between the fatigue lives at 700 Hz with cooling and at 10 Hz is caused by insufficient cooling at 700 Hz.

3. For fatigue tests in mercury without cooling, the frequency effect on the fatigue lives at 0.2 Hz and 10 Hz with a R ratio of -1 was much decreased relative to that in air. However, fatigue lives were shorter at 10 Hz than 0.2 Hz in mercury at high stress amplitudes (= 230 MPa) due to the greater specimen temperature at 10 Hz. By cooling the specimen in mercury to about room temperature with water at 10 Hz, the frequency effect was negligible. However, at low stress amplitudes (< 270 MPa), the fatigue life at
0.2 Hz in mercury was still shorter than that at 10 Hz with water cooling, which resulted from liquid-metal embrittlement.

4. For fatigue tests in mercury, the frequency effect was negligible at 700 Hz and 10 Hz with a R ratio of 0.1, because mercury around the specimen served as a good heat sink and decreased the specimen temperature at 700 Hz.

5. The striation spacing in air at 10 Hz was greater than at 0.2 Hz with a R ratio of -1, which is consistent with the greater specimen temperature at 10 Hz than at 0.2 Hz, and resulted in shorter fatigue lives at 10 Hz. The striation spacing in mercury at 0.2 Hz was approximately the same as that at 10 Hz in the range of $\Delta K$ levels investigated, which indicates comparable fatigue lives at 0.2 Hz and 10 Hz in mercury.

6. The striation spacing in air at 700 Hz was greater than at 10 Hz with a R ratio of 0.1, which is consistent with the greater specimen temperature in air at 700 Hz than at 10 Hz, and resulted in shorter fatigue lives at 700 Hz.
PART 7

ULTRAHIGH-CYCLE FATIGUE BEHAVIOR
7.1. Introduction

This part focuses on the study of fatigue behavior of 316 LN stainless steel in the ultra-high-cycle fatigue region. Issues, such as the location of crack initiation in UHCF and the existence of primary and secondary plateaus are discussed.

Failures of structural components such as turbine engines due to the high-frequency, low-amplitude, ultra-high-cycle fatigue has always been a concern in the industrial applications. In the SNS applications, the target is subjected to constant bombardment of the proton beam. The resultant vibration of the target container demands a good fatigue resistance of target container material under cyclic loading in a severe environment for the target to perform safely. The high-frequency ultrahigh-cycle fatigue of components can result in essentially unpredictable failures of the entire system. Due to the intrinsic cyclic service condition and a large amount of loading cycles imposed on the target container, it is difficult to study the fatigue property and achieve the desired high-frequency ultrahigh cycles using conventional fatigue testing machines. The ultrasonic fatigue is most useful in a practical sense when the material being tested is eventually exposed in service to frequencies near the test frequency. Over the past decade, new types of material test systems have been developed, making it possible to perform ultrahigh frequency (~20kHz) fatigue tests in a reasonably short period of time on structural materials. To simulate the high-frequency condition and study the frequency effect on the fatigue behavior of materials were two of the reasons, which led to the
development of high-frequency material test systems. Topics regarding ultrasonic fatigue include equipment development, frequency effects on fatigue, ultrasonic fatigue test results with and without aggressive environments present.

Mason is considered to be the first to use piezoelectric crystal transducers to investigate the fatigue properties of metals at an ultrasonic frequency, on the order of 20 kHz.\(^{[1]}\) He contemplated using magnetostrictive and piezoelectric transducers for developing high-power sound waves to fracture materials in fatigue. The source of sound energy is usually some form of electrical-energy converter, which takes an oscillating electrical signal and converts it to strain energy. This strain energy is transmitted by a waveguide, called a horn, to the specimen. Nowadays, a great portion of the ultra-high-frequency test systems still follows Mason’s original design.\(^{[1-4]}\) In principle, they were resonance systems in which each component was designed to have a resonant frequency of about 20 kHz. Piezoelectric and magnetostrictive types of the transducer were used to transfer the longitudinal ultrasonic waves to the specimen, and the whole system operated at the resonant frequency. Using this type of material test system, it was claimed that the ultrasonic frequency caused a pronounced increase in the fatigue life of aluminum alloys,\(^{[2]}\) had a mild effect on the fatigue life of body-centered-cubic (BCC) materials and no effect on that of face-centered-cubic (FCC) materials,\(^{[3]}\) or had various effects depending on the types of test materials.\(^{[4]}\) It was found that the fatigue behavior at high frequencies had similar characteristics to that of low-frequency fatigue, except that persistent slip bands were rather more localized in the high-frequency case.\(^{[2-4]}\)
A high-frequency (1,000 Hz) material test system capable of relatively high actuator displacements (+/- 0.17 mm) and dynamic loads (+/- 20 kN) at 1,000 Hz was employed to run ultrahigh cycle tests. In this part, the ultrahigh cycle fatigue properties of annealed and cold-worked 316 LN SS were studied, and issues such as the existence of primary and secondary plateau and the location of crack initiation are discussed.
7.2. Primary and Secondary Plateaus

The stress vs. fatigue life (S-N) curve of 20%-cold-worked specimens in terms of the number of cycles to failure obtained at 700 Hz using the MTS Model 810 system in air at room temperature is shown in Figure 7-1. Similar curves of annealed specimens were shown in PART 4 and PART 6. Similar to the fatigue data of annealed specimens, there was no secondary plateau region on the S-N curve that was observed by some researchers during UHCF fatigue. As shown in Figure 7-1, the fatigue endurance limit of cold-worked specimens at 700 Hz is about 250 MPa.

Although many researchers found fatigue failures in the UHCF region at stresses below the conventional fatigue limit, the fatigue result of 316 LN stainless steels showed no failure up to $10^8$ cycles at stress levels below the primary plateau (Figures 6-3 and 7-1). One explanation for this is that there are almost no internal inclusions inside this material or the internal inclusions are so small in size that they will not become the crack initiation site below the stress level of the primary plateau. As a result, the second plateau disappears because there are no internal failure mechanisms in 316 LN stainless steels at the stress levels below the primary plateau.

This result is a direct proof to the argument proposed by Mughrabi [6, 7] about Type I and Type II materials. Type I material is “clean” inside, and the failure mechanism, no matter in the HCF or UHCF regime, is always due to the formation of PSB or irreversible
slip on the specimen surface. Therefore, the S-N curve of the material will show a conventional asymptote. Type II material has internal inclusions of various sizes, and the failure of the materials depend on the size of the inclusions. If the size of the inclusions is very large, materials could fail at very low numbers of cycles with the cracks originated from internal inclusions. If the inclusions are small, they could play a role in the failure over the UHCF regime, when the PSB or irreversible slip couldn’t form on the surface below a certain stress level, and the initiation from the internal inclusions becomes easier than the surface. Then, the secondary plateau could form due to this mechanism.

Many researchers found that high-strength steels and surface-hardened steels are more likely to exhibit the stepwise S-N curve [8, 9]. Sakai, Takeda, and Oguma [9] proposed that the fatigue property of metallic materials in the UHCF region is governed by the static strength level. Their results on high-strength steels of SUJ2 and SNCM439 exhibit duplex S-N property for surface induced fracture and interior inclusion induced fracture. In contrast, the UHCF result of 316 LN stainless steel, which is a low-strength steel, only shows surface induced fracture in the entire region of S-N curve. However, the S-N property of UHCF could be more complex than only depending on the strength of materials, the material production method, microstructure, even the testing method can influence the shape of the S-N curve in a long time fatigue tests.
7.3. Temperature Evolution

It was found that the temperature of the specimen subjected to cyclic deformation rose due to the heat generated by energy dissipation, and a great increase in the temperature at the high frequency of 700 Hz was observed. In the present study, the temperature increase was measured by the thermocouples.

The study on Type 316 LN stainless steel indicated that the specimen self-heating at a high frequency during UHCF could be an important factor. Shorter fatigue lives in air were observed at 700 Hz than at 10 Hz, which was primarily caused by the higher temperature at 700 Hz. It was observed that the specimen temperature reached a maximum of 270 °C during the 700 Hz test, compared to 20 °C during the 10 Hz test. The elevated temperature decreased the strength of the material, thereby reducing the fatigue life. By cooling the specimen using nitrogen gas during fatigue tests in air at 700 Hz, the difference in fatigue lives at 700 Hz and 10 Hz were decreased. The remaining difference seems to be caused by the small difference in the specimen temperatures at 10 and 700 Hz (Figure 6-3).
7.4. Location of Fatigue Crack Initiation

The fracture surfaces of the failed specimens were examined by SEM. Figure 7-2 presents the fracture surface of a specimen fatigued at 700 Hz with a R ratio of 0.1 in air and at room temperature. For 700 Hz tests, the fracture surface showed similar appearance as that at 10 Hz. The topography of the fracture surface was found to have three distinctive regions. As presented in Figure 7-2, they are (a) the crack-initiation region, (b) the crack-propagation with striations, and (c) the dimpled-overload region. The crack-initiation sites were identified by tracing back along the radiation lines, which originated from the point of the initiation. The fatigue-crack-initiation site was at the specimen surface, as shown in Figures 7-2 and 7-3. Generally, the size of the crack-initiation sites was on the order of the grain size, ranging from 100 to 200 µm.

One of the most important factors that determine the surface initiation or external initiation could be the size of the inclusions. The internal initiations of cracks in the UHCF regime are usually caused by the failure or the de-bounding of inclusions. When the sizes of inclusions are small enough to overcome the internal failure mechanism in UHCF, the crack initiation sites will always be on the surface.

The fatigue-crack initiation occurs on the specimen surface of 316 LN SS as a result of the irreversible process of extrusion and intrusion formation through slip deformation. Typically, internal fatigue-crack-initiation origins have been attributed to the presence of
inclusions in some materials. Since Type 316 LN SS is a single phase materials and is “clean” with no inclusions inside, it does not form internal crack-initiation site in specimens undergoing high-cycle fatigue subjected to either low or high peak stresses.
7.5. Conclusions

1. There was no secondary plateaus in the S-N curves of annealed and cold-worked 316 LN SS at 700 Hz. The fatigue endurance limit for UHCF occurred at the stress amplitude of 260 MPa for cold-worked specimens. Persistent slip bands were the mechanism for the crack-initiation process.

2. The fractographic studies of all specimens showed surface crack initiation, and no internal initiation was found in the material.
PART 8

CHARACTERIZATION OF DISLOCATION STRUCTURES IN
TYPE 316 LN STAINLESS STEEL AFTER FATIGUE
8.1. Introduction

In an attempt to investigate residual stresses in 316 LN SS samples after fatigue, dislocation structures forming in 316 LN SS after fully-reverse-loading fatigue were examined. It was found that most dislocations in the specimens were of an edge type.

The 316 LN SS under fatigue loading at room temperature in air usually fails by crack initiation and propagation originated from the surface. The microstructural study and EDS analysis on the fatigue specimens showed no inclusions at the crack-initiation sites. The fracture surfaces showed a transgranular feature throughout the crack path. Both indicate a crack initiation mechanism due to the intrusion and extrusion activities of dislocations on the specimen surface during fatigue. Hence, an understanding of the dislocation mechanism in 316 LN SS undergoing uniaxial fatigue loading is necessary. Many researchers have discussed the dislocation structures in Type 316 SS, \cite{1-5} but little has been done on 316 LN SS, which is unique in its chemical composition and critical to the SNS application.

An investigation on the substructure in 316 L SS under thermal fatigue has been conducted by Robertson et. al.\cite{6} The dislocation arrangements after thermal fatigue from 27 °C up to 377 °C were found to be ladder-like in the fatigue samples.
TEM studies of 316 SS by Zielinski et al.\[7\] showed a macroscopically nonuniform spatial distribution of dislocations with a high dislocation density in the vicinity of the surface of the deformed specimens, indicating a higher work-hardening rate and the presence of the active dislocation sources near the free surface.

The comparative study on the fatigue crack-growth behavior of 316 L and 316 LN SS by Maeng and Kim\[8\] suggested that the better fatigue crack-growth resistance of 316 LN SS is related to the planar slip characteristic in the plastic zone rather than the martensitic transformation at the crack tip or crack closure. The planar structure of dislocations were often reported from the specimens with a low stacking fault energy, because the cross slip difficulty increases, as the stacking fault energy decreases. When the cross slip is difficult, the dislocations are observed along their slip planes, and they do not form bands of tangled dislocations and cell structures.

The planar dislocation structure and the enhancement in the fatigue properties of 316 LN were also explained by the decrease of the stacking fault energy with the addition of nitrogen and the interaction between interstitial atoms. In the study of the combined effect of molybdenum and nitrogen on the fatigued microstructure of Type 316 austenitic stainless steel by Murayama\[5\], specimens with three different composition of molybdenum and nitrogen content were investigated, i.e. 2.2Mo-0.0N, 0.0Mo-0.2N, and 2.2Mo-0.2N. A typical dislocation cell structure was the feature for 2.2Mo-0.0N specimens after low-cycle fatigue test at the total strain amplitude of $\Delta\varepsilon_{ta} = 0.6\%$ and
1.25%. In the other two types of specimens, cell structures were not developed. Specifically, in the 0.0Mo-0.2N specimen, dislocations form bands. In contrast, the microstructure of the 2.2Mo-0.2N specimen showed a planar array of dislocations. The author suggested that the stacking fault energy is believed to be low in the 0.0Mo-0.2N and 2.2Mo-0.2N specimens, but the planar array of dislocations is only evident in the 2.2Mo-0.2N specimen and not in the 0.0Mo-0.2N specimen. This trend suggests that the stacking fault energy may not be the only reason for the planar dislocation structure.

Chen et. al.[9] studied the microstructural evolution of fatigue damage in SA533-B1 steel and concluded that there is no correlation between the fatigue damage and the misorientation changes of subgrain structures. However, significant changes of dislocation structures were observed in the fatigued samples of Type 316L stainless steel. Dislocations of the fatigued 316L stainless steel specimens tend to arrange themselves on \{111\} slip planes at room temperature. At 300 ºC, the dislocations tend to move from their slip planes into subgrain boundaries (i.e., the dynamic recovery).

The previous researchers have addressed various aspects of the dislocation structure, morphology, and mechanisms, but few have discussed the basic characteristic of an individual dislocation after fatigue deformation, such as the type of dislocations, which can help explain the dislocation morphology in 316 LN SS after fatigue. This Part concentrates on the experimental study regarding the dislocation structure of dislocations in 316 LN SS after tension-compression fatigue loading in air.
8.2. Experiment

Sample Preparation

Samples were disks with 3 mm in diameter. Most samples were cut, perpendicular to the longitudinal direction of the specimen from the gage section near the fracture surface. Some samples were cut along an angle of 45° to the longitudinal direction of the specimen. Samples that were cut this way are suitable for the dislocation analyses, since their surfaces are somehow parallel to the shear plane with the highest deformation. Dislocations in these directions tend to be relatively long and straight.

A Gillings HAMCO thin sectioning machine was employed to slice thin disks off the specimen. The thickness of disks after sectioning from the fatigue specimens were in the range of 0.25 ~ 0.3 mm. These thin disks were then cut into small disk samples of 3 mm in diameter using a hand punch. To obtain thin samples with more observable areas, disks were mechanically thinned and polished using sand papers of 300, 400, 600, and 800 grit. The final thickness of samples were in the range of 0.15 ~ 0.18 mm. The final step was electro-polishing, using a Struers Tenupol-3 dual-jet electro-polisher.

To obtain a good polishing result suitable for TEM observations and dislocation studies, many polishing parameters had to be properly selected and controlled. These parameters include the polishing current, electrolyte flow rate, photo sensitivity, and polishing time.
Choosing a good electrolyte usually gave a better result than controlling other parameters. The electrolyte for polishing 316 LN SS was composed of 5% perchloric acid and 95% methanol. The electrolyte reservoir had to be cooled to preserve the viscosity of the electrolyte. The common method was a water-cooling coil around the polishing unit.

The optimal polishing voltage was 40 V, and the polishing current was about 0.35 ~ 0.5 A. This voltage shouldn’t be too low or too high. In the previous case, there was no electro-polishing effect during the polishing process, with the corrosion effect being the dominant factor. Samples usually showed a large hole at the center, and thick edges and corrosion pits on the surface. On the other hand, if the voltage is too large, the polishing process would be too fast to be well controlled.

The photo sensitivity will determine the size of the central hole. Experience showed that a small hole usually showed a thinner edge than that of a larger hole. Therefore, the photo sensitivity should be adjusted to the maximum. Sometimes, switching the side of the sample in the holder could help develop a better polishing result. However, the result was not always consistent.

The flow rate of the revolution of the electrolyte through the jets in the polishing chamber is controlled by the sensitivity screw, which was adjusted to the minimum. The flow rate was set to 5.5, which proves to be the optimal value. After examining the polished
sample, it was found that the flow rate should be adjusted to the optimal value to obtain a good polishing condition. If the flow rate is too low, the electrolyte will not have enough pressure to form a jet effect. The sample generally formed thick edges around irregular holes, which made the subsequent TEM observation difficult. On the other hand, when the flow rate was too high, the polishing surfaces of samples were uniform.

Generally, the optimal polishing time was about 20~30 sec, depending on the thickness of sample.

Operation of TEM in Observing the Dislocation Structure

The first thing to be considered is the specimen characteristics in a TEM study of dislocations in the material. The grain size of the material and the thickness of the specimen are both important factors to determine if the specimen is suitable for a dislocation characterization under TEM. The grain size of an annealed 316 LN SS is about 50 µm on the average, which is appropriate for selective area diffraction (SAD) analyses. The disk sample after double jet electro-polishing has both thin areas (< 100 nm to 300 nm thick, depending on the material) to observe the morphology of dislocations and areas that are sufficiently thick for Kikuchi lines to be visible.

To determine the orientation of the region of the specimen illuminated by the beam, a stereographic projection and a Wulff net are needed. A stereographic projection is a
good method for visualizing the orientation of grains. It is a method of projecting three-dimensional crystal orientations onto a plane, and presenting the angles between crystal planes on the projection. A detailed introduction can be found in any introductory crystallography text. The Wulff net shows 90 great circles all passing from the north pole to the south pole and another around the equator: a great circle always passes through opposite ends of a diameter in the projection. Circles on the sphere that do not contain the center of sphere are small circles. All distances on the net are proportional to angles in a real space but only correspond exactly to angles around the primitive great circle. Therefore, the angles between any two crystal-direction can be read from the net after certain operation.

The method to determine the diffraction, $g$, is by using both the Kukuchi lines and diffraction patterns. Using a diffraction pattern, the orientation of the beam, relative to the crystal, can be estimated to an accuracy of $\sim 3^\circ$. Kikuchi patterns can increase the accuracy to $\sim 0.1^\circ$.

The TEM employed in the analyses of dislocations is a Philips CM30, whose accelerating voltage of the electrons is 300 kV with a wavelength $\lambda = 0.0197 \, \text{Å}$.\[^{10}\]

The fundamental relationship in a diffraction pattern is

$$ Rd = L\lambda $$  \hspace{1cm} (8–1)
R: spacing diffraction spots  
d_{hkl}: the interplanar spacing  
L: the camera length, L = 500 mm (Mag: 42 kx, 300 kV)  
\lambda: the wavelength

The 316 LN SS fcc \( \gamma \)-Fe, whose crystal cube edge, \( a = 3.5698 \, \text{Å} \), and the relationship of \( a \) 
and \( d_{hkl} \) is

\[
2 \sum_{ijkl} = \left( \frac{8}{l^2+k^2+t^2} \right)^{1/2} 
\]

Therefore, the ratio of any two R values gives that of the d-spacings. Knowing the lattice parameter of 316 LN SS, the d-spacings can be determined by checking with the allowed reflections and the ratios of any two d-spacings. Once the tentative values of \( g \) vectors are identified, the answer is cross-checked with the angles between \( g \) vectors. The diffraction pattern can then be determined.

In practical measurements, SAD is not the most accurate method for determining the spacing of lattice planes or angles between them. It fails when the difference between the two patterns is a 180° rotation because they look the same in SAD. In this case, Kikuchi lines can be used to remove the 180° confusion. Another thing that needs the attention is
that the specimen has to be tilted so that excitation error, $s$, is set to 0 for the SAD reflections. When $s$ is set to 0, using a higher-order reflection, can be more accurate than a low-index reflection. The method to set $s$ to 0 is to keep the selected $g$ strongly excited, and let the corresponding Kikuchi lines pass through the diffraction spots. Two or more diffraction patterns are needed to determine the orientation relationship.

After identifying the diffraction, $g$, the diffraction vector can be indicated in the TEM image of the corresponding area. Due to the magnetic rotation of the electron beam of TEM, there is a rotation angle between the TEM image and its diffraction pattern. The rotation angle of Philips CM30 at 300 kV is clockwise (CW) 260º. The angle is the rotation of the SAD required to make pattern collinear with the image. The emulsion sides of negatives of SAD photos are kept up. Positive angles are CW.

Kikuchi patterns are formed by the incoherently scattered electrons, which travel in all directions. These electrons can be Bragg diffracted by the planes. Those diffusely scattered electrons that travel close to the direction of the incident beam have higher energy than those away from the incident beam direction, and, thus, the former ones form a bright line and the later ones form a dark line on the image. The reason that they form two lines on the image can be found in most TEM text. Since the scattered electrons are traveling in all directions, the diffracted beam will lie on one of two cones. In other words, we see cones of diffracted electrons rather than well-defined beams, because there is a range of incident beam directions rather than a single direction. Since the screen is
flat and nearly normal to the incident beam, the intersection of two cones with the screen form two parabolas. Because only a small portion of the two parabolas is observed on the screen, they look like two parallel lines, which are the pair of Kikuchi lines.

If the sample is tilted through a very small angle, the Kikuchi lines will move but the intensities of the diffraction spots will hardly change, and the positions of the spots will not change. This property makes the Kikuchi lines a unique tool to determine the exact orientation of the electron beam, relative to the crystal.

The distance in the reciprocal space between a pair of Kikuchi lines is \( g \). The spacing of two Kikuchi lines can be measured and used to calculate the interplanar spacing using equation 8-1. The crystal planes can, then, be identified, and checked by measuring the angles between the traces of planes on the Kikuchi map.

**Developing Negatives and Prints**

The exposure time when taking TEM pictures varies, depending on the intensity of the bright or dark field image. The developing steps include four minutes in developing the solution, five minutes in the stop bath, and two minutes in the fixer, with fifteen seconds of cleansing between steps.

The exposure time of prints varies, depending on the aperture size and filter paper. The developing procedure includes immersing prints in three solutions, which are the
developing solution (1 part of Dektol developer plus 2 parts of water), stop bath (1 liter water plus a small amount of indicator), and fixer. Time in each solutions are, respectively, 30 ~ 60 seconds, 10 ~ 15 seconds, and 5 minutes.
8.3. Analyses of Types of Dislocations in 316 LN SS After Fatigue

Ideally, for quantitative analyses, it’s better to choose long, nearly straight dislocations. The 316 samples were cut along 45° from the longitudinal direction of the tested specimen to obtain relatively long dislocations.

The burger’s vectors of dislocations can be determined by examining the visibilities of dislocations at different diffractions. For the total dislocation in the fcc material, possible \( \mathbf{b} \) vectors were shown in Table 6. A schematic Kikuchi map for a f.c.c. crystal was shown in Figure 8-1. A total of five diffractions at –30°, -20°, -12°, 15°, and 30° to the foil normal direction (FN) were chosen to take TEM images and their corresponding SADs. A dislocation morphology was taken at the foil normal direction, which was shown in Figure 8-2. Four relatively long dislocations, a, b, c, and d, were analyzed, whose corresponding positions on Figure 8-2 were illustrated in Figure 8-3. The selected orientations of the beam and the diffraction of each orientation were indicated on the schematic Kikuchi map. To obtain the selected orientations, a double-tilt specimen holder was employed. The possible \( \mathbf{g} \cdot \mathbf{b} \) values for \( \mathbf{b} = \mathbf{a}/2 <110> \) dislocations in fcc metals were constructed in Table 6.

For a total dislocation in 316 LN SS, the dislocation will lose its contrast when \( \mathbf{g} \cdot \mathbf{b} = 0 \). From Figures 8-4 to 8-8, the visibility of dislocations a - d can be identified. The result was shown in Table 7. By comparing the visibility of each dislocation under different
diffractions with that in Table 6, their burger’s vectors can be determined (Table 7). The burger’s vectors of dislocations were then plotted on the images. Since the burger’s vectors were not in the plane of the image, their projections on the image plane were plotted, and the angle between the burger’s vectors and their projection were indicated in the caption. The line directions of dislocations a - d were, although not ideally, in the plane of the image. The method of determining the line direction of a dislocation is described below.

Take two images of the same dislocation with different beam orientations. From the Kikuchi map, identify the orientation of the beam on one of the maps, and indicate the direction of the beam orientation on the standard projection. Find and plot out the big circle of the plane, perpendicular to the beam direction, which represents the plane orientation of the first image. Indicate the diffraction vector, \( \mathbf{g} \), on the big circle. Measure the angle between the line direction of the dislocation and \( \mathbf{g} \), and plot out the position of the line direction on the big circle. Next, connect the beam position and the line position using a big circle. This is the big circle of the projection plane of the dislocation on the first image. Do the same on another image, and draw another big circle connecting the line direction and the beam direction. The intersection of these two big circles is the actual line direction of the dislocation. The measured line directions of dislocations in 316 LN SS samples are very close to those shown on the images, with an error less than 5º.
The direction of \( b \) and dislocation line can then be reconstructed, and they were basically perpendicular to each other for the dislocations studied, as shown from Figures 8-4 to 8-8, which indicated that they are edge-type dislocations.

The morphologies of dislocations in the 316 LN stainless steel after fatigue were shown in Figures 8-9 to 8-16. Figure 8-9 showed grain structures of the material. The dark lines were bending contours and thickness contours in the film sample, and their mixed effects. The typical dislocation cell structures were found in the specimen. Due to the small stacking fault energy of austenitic steels, which is about 20 mJm\(^{-2}\)[11], cell structures only occurred locally (as shown by the arrows in Figures 8-10 and 8-11) in the grains where the grain underwent severe deformation. Besides the dislocation cell structures, twin lamellas were also evident in the specimen as shown in Figure 8-11. Figure 8-12 gave a better view of the cell structure of dislocations at a higher magnification, formed by cell walls composed of low angle edge-type dislocations. In addition, persistent lüder band (PLB) structures were found in some areas in the samples after fatigue, which prevented the formation of cell structures (Figure 8-13). Other features like stacking faults (Figure 8-14) and dislocation pile-ups were found near the grain boundaries (Figure 8-15). Figure 8-16 showed the typical irregular pile up of glissile dislocations at a grain boundary in the 316 LN stainless steel showing the zig-zag feature of edge dislocations due to the slipping of dislocations at different crystal planes.
8.4. Conclusions

1. The dislocations in 316 LN stainless steel after fatigue at 0.2 Hz with a R ratio of -1 are mostly of an edge type.

2. The typical dislocation morphology in 316 LN stainless steel after tension-compression fatigue consists of a cell structure and pile-up dislocations near the grain boundary.
PART 9

CONCLUSIONS
The present research was intended to provide both a fundamental scientific and engineering understanding of fatigue behavior of Type 316 LN stainless steel in the air and mercury environments, dealing with the effects of frequency and environment on the fatigue-crack initiation and propagation behavior, and the fatigue life of the material.

A fundamental methodology was employed to integrate the fatigue studies, including the high-cycle, ultrahigh-cycle fatigue testing and microstructural characterizations in light of the optical and electron microscopy.

A mechanistic understanding of the crack propagation was achieved by indirectly measuring the striation spacing of specimens after high-cycle fatigue as a function of the stress-intensity-factor range. Mercury was found to have a detrimental effect on the resistance to fatigue crack propagation at low frequencies (0.2 Hz and 10 Hz). At high frequencies (700 Hz), this effect is minimal due to the time-dependant environmental process in mercury. Another observation is that the environmental effect is only significant at high stress levels due to the fact that a larger crack opening displacement is needed for a better wettability between the mercury and the crack tip.

The frequency effect on the fatigue life was largely contributed by the self-rising of specimen temperature during the high-frequency fatigue process. With a comparable specimen temperature, fatigue lives at different frequencies are comparable, indicating a minimal strain rate effect. A theoretical calculation of the specimen temperature was
performed numerically using the basic theory of thermo-inelastic, heat conduction and convection effects.

Ultrahigh cycle fatigue was conducted and its distinct features, such as the location of crack initiation site and the shape of S-N curve, were discussed. No failure below the primary fatigue endurance limit was found with fatigue lives extending over $10^9$ cycles. Cold-worked samples simulating the mechanical property of the annealed 316 LN stainless steel subjected to the radiation effect was tested in air and mercury environments with a controlled specimen temperature. It was found that the environmental effect on the fatigue life of the cold-worked 316 LN stainless steel was minimal.

The present project was the first attempt to systematically study the fatigue behavior of Type 316 LN stainless steel in mercury. The elevated temperatures were identified and characterized, and their effects on the fatigue performance were investigated in detail. The understanding of fatigue mechanisms of Type 316 LN stainless steel will initiate new opportunities for structural applications, and provide pertinent information for material processing and applications. In addition, a large amount of fatigue data of 316 LN stainless steel was accumulated during this investigation, which will be a good reference for the related research and engineering activities in the applications of the material for the nuclear and accelerator-based neutron source facilities.
PART 10

FUTURE WORK
The present study helps understand the fatigue resistance of Type 316 LN SS in air and mercury and at high frequencies. In the future work, well-controlled fatigue crack propagation (FCP) tests and a feasible method of measuring the crack length in mercury are of great interest. Investigations of the interaction of mercury with Type 316 LN SS and the chemical process during fatigue will help understand the time-dependent environment factor that contributes to the intergranular fatigue damage of the material.

The fatigue-damage mechanisms of 316 LN SS in air are influenced by the thermo-inelastic phenomenon during the fatigue process. The IR thermography technology was utilized to observe the temperature evolution and obtain the temperature map on the specimen during fatigue. In the future investigation, the finite element modeling can be conducted to further study temperature evolutions and temperature profile of the specimen during fatigue. A study on the dynamic thermal-mechanical coupling effect during fatigue is crucial to characterize the fatigue damage and predict the fatigue life of 316 LN SS in an environment without the temperature control. In an environment where the specimen temperature is well controlled, the temperature gradient from interior of the specimen to the surface could still be a factor that influences the fatigue life of the specimen, especially during a high-frequency fatigue test. Highly efficient cooling methods are needed to effectively control the specimen temperature during fatigue when the fatigue results at various test conditions are to be compared.
In the present study, the crack closure effect due to the penetration of mercury into the crack tip was not considered, which could be a factor that affects the fatigue life of 316 LN SS in mercury. Comparative study of this effect using inert oil could be performed to characterize the liquid-induced crack closure effect.

A detailed microstructure characterization, such as the evolution of the dislocation structures, and a comparison of the material under tension and fatigue conditions can be of critical importance to understand the fatigue-damage mechanisms of Type 316 LN SS.

To combine techniques such as mechanical testing, microstructural characterization, nondestructive evaluation, fatigue and fracture mechanics, and life prediction would be a good methodology to the further investigation on the frequency and environmental effects on the fatigue behavior of Type 316 LN SS.
REFERENCES
Part 1.


Part 2.


Part 3.


Part 4.


Part 5.


Part 6.


Part 7.


Part 8.


Table 1. Chemical composition of Type 316 low-carbon, nitrogen-added (LN) stainless steel (SS) (wt%, weight percent)\[1\]

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt%</td>
<td>0.009</td>
<td>1.75</td>
<td>0.029</td>
<td>0.002</td>
<td>0.39</td>
<td>10.2</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Element</th>
<th>Cr</th>
<th>Mo</th>
<th>Co</th>
<th>Cu</th>
<th>N</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt%</td>
<td>16.31</td>
<td>2.07</td>
<td>0.16</td>
<td>0.23</td>
<td>0.11</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

Table 2. Mechanical characteristics of Type 316 stainless steels at 27 and -196 °C [1]

<table>
<thead>
<tr>
<th>Materials</th>
<th>Test Temperature (°C)</th>
<th>$\sigma_y$ (MPa)</th>
<th>$\sigma_u$ (MPa)</th>
<th>A (Pct)</th>
<th>S (Pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L</td>
<td>27</td>
<td>262</td>
<td>574</td>
<td>56.4</td>
<td>75.9</td>
</tr>
<tr>
<td></td>
<td>-196</td>
<td>402</td>
<td>1156</td>
<td>56.8</td>
<td>69.6</td>
</tr>
<tr>
<td>316LN</td>
<td>27</td>
<td>328</td>
<td>697</td>
<td>47.3</td>
<td>71.7</td>
</tr>
<tr>
<td></td>
<td>-196</td>
<td>902</td>
<td>1415</td>
<td>45.6</td>
<td>63.7</td>
</tr>
</tbody>
</table>

Table 3. Tensile properties of 316 LN SS at a strain rate of 0.001 1/s

<table>
<thead>
<tr>
<th>Specimen Condition</th>
<th>Test Temperature (°C)</th>
<th>$\sigma_{0.2}$ (MPa)</th>
<th>$\sigma_{uts}$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Annealed</td>
<td>24</td>
<td>289</td>
<td>600</td>
</tr>
<tr>
<td></td>
<td>300</td>
<td>173</td>
<td>460</td>
</tr>
<tr>
<td></td>
<td>500*</td>
<td>133</td>
<td>350</td>
</tr>
</tbody>
</table>

$\sigma_{0.2}$ = 0.2 % offset yield strength
$\sigma_{uts}$ = ultimate tensile strength
Table 4. Environmental effects on the fatigue behavior of 316 LN SS

<table>
<thead>
<tr>
<th>Effects</th>
<th>Condition</th>
<th>Phenomenon</th>
<th>Mechanism</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.2 Hz</td>
<td>Shorter fatigue lives in mercury than in air</td>
<td>Liquid-metal-embrittlement (LME) effect</td>
</tr>
<tr>
<td>R = -1</td>
<td>10 Hz</td>
<td>Significant shorter fatigue lives in air than in mercury</td>
<td>Greater specimen temperature in air than in mercury; Mercury acting as a coolant, reducing the specimen temperature</td>
</tr>
<tr>
<td></td>
<td>10 Hz</td>
<td>Shorter fatigue lives in mercury than in air</td>
<td>LME effect; Better wetting between the mercury and steel</td>
</tr>
<tr>
<td>R = 0.1</td>
<td>700 Hz</td>
<td>Longer fatigue lives in mercury than in air</td>
<td>Insignificant LME effect; Greater specimen temperature in air than in mercury</td>
</tr>
</tbody>
</table>
Table 5. Frequency effects on the fatigue behavior of 316 LN SS

<table>
<thead>
<tr>
<th>Environment</th>
<th>R Ratio</th>
<th>Test Condition</th>
<th>Phenomenon</th>
<th>Mechanism</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air</td>
<td>-1</td>
<td>Without cooling</td>
<td>Significant shorter fatigue lives at 0.2 Hz than at 10 Hz</td>
<td>Increase of the specimen temperature at the higher frequency</td>
</tr>
<tr>
<td></td>
<td></td>
<td>With cooling</td>
<td>Insignificant difference in fatigue lives at 0.2 Hz and 10 Hz</td>
<td>Comparable specimen temperature at 10 Hz with nitrogen cooling and at 0.2 Hz, and negligible frequency effect</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>Without cooling</td>
<td>Significant shorter fatigue lives at 10 Hz than at 700 Hz</td>
<td>Increase of the specimen temperature at the higher frequency</td>
</tr>
<tr>
<td></td>
<td></td>
<td>With cooling</td>
<td>Small difference in fatigue lives at 10 Hz and 700 Hz</td>
<td>A somewhat higher specimen temperature at 700 Hz than at 10 Hz due to insufficient nitrogen cooling</td>
</tr>
<tr>
<td>Mercury</td>
<td>-1</td>
<td>Without cooling</td>
<td>Comparable fatigue lives at 0.2 Hz and 10 Hz</td>
<td>Mercury acting as a coolant, reducing the specimen temperature at 10 Hz</td>
</tr>
<tr>
<td></td>
<td></td>
<td>With cooling</td>
<td>Somewhat shorter fatigue lives at 0.2 Hz than at 10 Hz</td>
<td>A more significant environmental effect at 0.2 Hz than at 10 Hz due to longer wetting time for mercury in cracks</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>Without cooling</td>
<td>Comparable fatigue lives at 10 Hz and 700 Hz</td>
<td>Mercury acting as a coolant, reducing the specimen temperature at 700 Hz</td>
</tr>
<tr>
<td></td>
<td></td>
<td>With cooling</td>
<td>No cooling procedure is used since mercury itself acts as a coolant</td>
<td></td>
</tr>
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</table>
Table 6. Possible $\mathbf{g} \cdot \mathbf{b}$ values for $\mathbf{b} = a/2 \langle 110 \rangle$ dislocations in fcc metals

<table>
<thead>
<tr>
<th></th>
<th>101</th>
<th>011</th>
<th>110</th>
<th>(\bar{1})01</th>
<th>01(\bar{1})</th>
<th>1(\bar{1})0</th>
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<tbody>
<tr>
<td>0 2 0</td>
<td>0</td>
<td>2</td>
<td>2</td>
<td>0</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>(\bar{1})11</td>
<td>0</td>
<td>2</td>
<td>0</td>
<td>2</td>
<td>0</td>
<td>(\bar{2})</td>
</tr>
<tr>
<td>11(\bar{1})</td>
<td>0</td>
<td>0</td>
<td>2</td>
<td>(\bar{2})</td>
<td>2</td>
<td>0</td>
</tr>
<tr>
<td>2 2 0</td>
<td>2</td>
<td>2</td>
<td>0</td>
<td>(\bar{2})</td>
<td>(\bar{2})</td>
<td>4</td>
</tr>
<tr>
<td>2 0 0</td>
<td>(\bar{2})</td>
<td>0</td>
<td>(\bar{2})</td>
<td>2</td>
<td>0</td>
<td>(\bar{2})</td>
</tr>
</tbody>
</table>
Table 7. Visability of dislocations at different $g$

<table>
<thead>
<tr>
<th>Beam direction</th>
<th>$g$</th>
<th>Dislocation</th>
<th>a</th>
<th>b</th>
<th>c</th>
<th>d</th>
</tr>
</thead>
<tbody>
<tr>
<td>-30°</td>
<td>0 2 0</td>
<td></td>
<td>visible</td>
<td>visible</td>
<td>invisible</td>
<td>invisible</td>
</tr>
<tr>
<td>-20°</td>
<td>1 1 1</td>
<td></td>
<td>visible</td>
<td>visible</td>
<td>visible</td>
<td>invisible</td>
</tr>
<tr>
<td>-12°</td>
<td>1 1 1</td>
<td></td>
<td>invisible</td>
<td>invisible</td>
<td>visible</td>
<td>invisible</td>
</tr>
<tr>
<td>15°</td>
<td>2 2 0</td>
<td></td>
<td>visible</td>
<td>visible</td>
<td>visible</td>
<td>visible</td>
</tr>
<tr>
<td>30°</td>
<td>2 0 0</td>
<td></td>
<td>invisible</td>
<td>invisible</td>
<td>visible</td>
<td>visible</td>
</tr>
</tbody>
</table>
APPENDIX B

FIGURES
Figure 1-1. Facility of the Spallation Neutron Source (SNS)[*]

Figure 1-2. Illustration of the target container of the Spallation Neutron Source[^1]

Figure 2-1. The mechanism of a competition between LME and concurrent crack tip oxidation: a schematic illustration by D. A. Wheeler, 1989 [*]

Figure 3-1. Cylindrical specimens used for high-cycle fatigue tests at R ratios of (a) -1 and (b) 0.1
Figure 3-2. Compact-tension specimen of 316 LN SS with a width (W) of 50.8 mm, a thickness (B) of 6.35 mm, a notch length (a₀) of 10.16 mm, and an initial crack length (a) of 11.43 mm
Figure 3-3. Fatigue crack-propagation rates for the compact-tension specimen of 316 LN SS
Figure 4-1. Environmental effect on the fatigue life of 316 LN SS at 0.2 Hz and a R ratio of -1
Figure 4-2. Environmental effect on the fatigue life of 316 LN SS at 10 Hz and a R ratio of -1 with nitrogen and water cooling.
Figure 4-3. Environmental effect on the fatigue life of 316 LN SS at 10 Hz and a R ratio of 0.1
During 700 Hz fatigue tests, the specimen temperature can reach as high as 270 °C and 78 °C in air and mercury, respectively.

\[ \sigma_a = -54.29 \log(N_f) + 488.41 \]  

\[ \sigma_a = -37.12 \log(N_f) + 382.82 \]

Figure 4-4. Environmental effect on the fatigue life of 316 LN SS at 700 Hz and a R ratio of 0.1
Figure 4-5. Variation of the specimen temperature with the test frequency during fatigue experiments of 316 LN SS in air and mercury at a stress amplitude of 198 MPa and a R ratio of 0.1
During 700 Hz fatigue tests, the specimen temperature can reach as high as 270 °C and 78 °C in air and mercury, respectively. N\textsubscript{2} cools the sample to 20 °C - 70 °C during 700 Hz fatigue tests in air.

Figure 4-6. Environmental effect on the fatigue life of 316 LN SS at 700 Hz and a R ratio of 0.1
Figure 4-7. Material microstructure of 316 LN stainless steel (etched electrolytically using 10%wt oxalic acid) after failure. The sample was tested at 0.2 Hz in air with a R ratio of -1 and a stress amplitude of 287 MPa, and failed after 7,872 cycles.
Figure 4-8. Material microstructure of 316 LN stainless steel showing a growing surface crack. The sample was tested at 0.2 Hz in air with a R ratio of –1 and a stress amplitude of 287 MPa, and was suspended after 5,159 cycles.
Figure 4-9. Material microstructure of 316 LN stainless steel showing the morphology of the specimen surface (border of the bright and dark fields in the upper part of the photo).
Figure 4-10. Material microstructure of 316 LN stainless steel showing the initiation of surface cracks. The sample was tested at 0.2 Hz in air with a R ratio of -1 and a stress amplitude of 287 MPa, and was suspended after 5,000 cycles.
Figure 4-11. Material microstructure of 316 LN stainless steel showing a growing surface crack after fatigue testing in air. The sample was tested at 0.2 Hz in air with a R ratio of -1 and a stress amplitude of 287 MPa, and was suspended after 5,506 cycles.
Figure 4-12. Material microstructure of 316 LN stainless steel showing a growing surface crack after fatigue testing in mercury. The sample was tested at 10 Hz in mercury with a R ratio of -1 and a stress amplitude of 263 MPa, and was suspended after 4,361 cycles.
Figure 4-13. Fracture surface showing the transgranular fracture in the crack-propagation area for the specimen of 316 LN SS tested in air at 0.2 Hz with a stress amplitude of 260 MPa and a R ratio of -1.
Figure 4-14. Fracture surface showing the intergranular fracture in the crack-propagation area for the specimen of 316 LN SS tested in mercury at 0.2 Hz with a stress amplitude of 260 MPa and a R ratio of -1.
Figure 4-15. Fracture surface showing the transgranular fracture in the crack-propagation area for the specimen of 316 LN SS tested in air at 10 Hz with a stress amplitude of 260 MPa and a R ratio of -1.
Figure 4-16. Fracture surface showing the intergranular fracture in the crack-propagation area for the specimen of 316 LN SS tested in mercury at 10 Hz with a stress amplitude of 260 MPa and a R ratio of -1.
Figure 4.17. Fracture surface showing the typical fracture mode in the crack-propagation area for the specimen of 316 LN SS tested in air at 10 Hz with a stress amplitude of 232 MPa and a R ratio of 0.1.
Figure 4-18. Fracture surface showing the intergranular fracture in the crack-propagation area for the specimen of 316 LN SS tested in mercury at 10 Hz with a stress amplitude of 232 MPa and a R ratio of 0.1.
Figure 4-19. Stress vs. strain curves for uniaxial loading tests of 20% cold-worked 316 LN SS in air at ambient temperature.
Figure 4-20. Environmental effect on the fatigue life of 20% cold-worked 316 LN SS at 700 Hz and a R ratio of 0.1
Figure 5-1. 256 x 256 pixels focal plane array (InSb: 3-5 µm), snap-shot mode, temperature resolution: 0.015 °C, spatial resolution: 5.4 µm (microscope attachment), high speed: full frame at 140 Hz, window at 6,100 Hz
Figure 5-2. Temperature change during 700 Hz fatigue test of Type 316 LN stainless steel in air
Figure 5-3. Temperature change during the 700 Hz fatigue test of Type 316 LN stainless steel in mercury
Figure 5-4. Specimen temperature evolution of 316 LN SS in air at 0.2 Hz and 10 Hz with a stress amplitude of 263 MPa, and a R ratio of -1
Figure 5-5. Stress vs. strain curves corresponding to different fatigue cycles of 316 LN SS in air at 10 Hz with a stress amplitude of 263 MPa and a R ratio of -1.
Figure 5-6. Stress vs. strain curves corresponding to different fatigue cycles of 316 LN SS in air with nitrogen cooling at 10 Hz with a stress amplitude of 263 MPa and a R ratio of -1.
Figure 5-7. Stress vs. strain curves corresponding to different fatigue cycles of 316 LN SS in air at 0.2 Hz with a stress amplitude of 263 MPa and a R ratio of -1.
Figure 5-8. Stress vs. strain curves for uniaxial loading tests of 316 LN SS in air at (a) ambient temperature and (b) 300 °C
Figure 5-9. Areas of hysteresis loops corresponding to different fatigue cycles in air with and without nitrogen cooling at 0.2 Hz and 10 Hz with a stress amplitude of 263 MPa and a R ratio of -1
Figure 5-10. Coordinate system for modeling the temperature evolution of the gage-section area of the specimen during fatigue tests
Figure 5-11. Predicted and measured results of specimen-temperature evolutions of 316 LN SS in air at 10 Hz with a stress amplitude of 287 MPa and a R ratio of -1.
Figure 6-1. Frequency effect on the fatigue life of 316 LN SS in air at 0.2 Hz and 10 Hz with a R ratio of -1.
Figure 6-2. Specimen-temperature evolution in air and mercury at 0.2 Hz and 10 Hz with a stress amplitude of 263 MPa and a R ratio of -1.
The specimen temperature can reach as high as 270 °C during 700 Hz fatigue tests in air. N₂ cools the sample to 20 °C - 70 °C during 700 Hz fatigue tests.

Figure 6-3. Frequency effect on the fatigue life of 316 LN SS in air at 10 Hz and 700 Hz with a R ratio of 0.1
Figure 6-4. Frequency effect on the fatigue life of 316 LN SS in mercury at 0.2 Hz and 10 Hz with a R ratio of -1
The specimen temperature can reach 78 °C during 700 Hz fatigue tests in mercury.

Figure 6-5. Frequency effect on the fatigue life of 316 LN SS in mercury at 10 Hz and 700 Hz with a R ratio of 0.1
Figure 6-6. Microstructure of the fracture surface in the crack-initiation area for the specimen of 316 LN SS tested in air at 0.2 Hz and 10 Hz with a stress amplitude of 230 MPa and a R ratio of -1.
Figure 6-7. Fracture surface showing the striations in the crack-propagation area for the specimen of 316 LN SS tested in air at 0.2 Hz and 10 Hz with a stress amplitude of 230 MPa and a R ratio of -1.
Figure 6-8. Fracture surface showing the crack initiation and propagation area for the specimen of 316 LN SS tested in air at 10 Hz and 700 Hz with a stress amplitude of 198 MPa and a R ratio of 0.1.
Figure 6-9. Fracture surface showing the striations in the crack-propagation area for the specimen of 316 LN SS tested in air at 10 Hz and 700 Hz with a stress amplitude of 198 MPa and a R ratio of 0.1.
Figure 6-10. Microstructure of the fracture surface in the crack-initiation area for the specimen of 316 LN SS tested in mercury at 0.2 Hz and 10 Hz with a stress amplitude of 230 MPa and a R ratio of -1.
Figure 6-11. Microstructure of the fracture surface in the crack-propagation area for the specimen of 316 LN SS tested in mercury at 0.2 Hz and 10 Hz with a stress amplitude of 230 MPa and a R ratio of -1.
(a) At 0.2 Hz  
(b) At 10 Hz

Figure 6-12. Fracture surface showing the striations in the crack-propagation area for the specimen of 316 LN SS tested in mercury at 0.2 Hz and 10 Hz with a stress amplitude of 230 MPa and a R ratio of -1.
Figure 6-13. Microstructure of the fracture surface in the crack-initiation area for the specimen of 316 LN SS tested in mercury at 10 Hz and 700 Hz with a stress amplitude of 198 MPa and a R ratio of 0.1.
Figure 6-14. Microstructure of the fracture surface in the crack-propagation area for the specimen of 316 LN SS tested in mercury at 10 Hz and 700 Hz with a stress amplitude of 198 MPa and a R ratio of 0.1.
Figure 6-15. Striation spacing versus $\Delta K$ for specimens of 316 LN SS tested in air and mercury at 0.2 Hz and 10 Hz with a stress amplitude of 230 MPa and a R ratio of -1.
Figure 6-16. Striation spacing versus $\Delta K$ for specimens of 316 LN SS tested in air at 10 Hz and 700 Hz with a stress amplitude of 198 MPa and a R ratio of 0.1.
Figure 7-1. Comparison of the S-N curves with and without secondary plateau: (a) S-N curve of 20%-cold-worked 316 LN SS in air at 700 Hz, (b) a schematic of a typical S-N curve with a HCF plateau around $10^6$ to $10^8$ cycles and a UHCF plateau above about $10^9$ cycles

Figure 7-2. Typical macrostructure of fracture surface of 316 LN SS tested at 700 Hz
Figure 7-3. Micro-crack initiated on the surface of 316 LN SS tested at 700 Hz
Figure 8-1. A schematic Kikuchi map for a f.c.c. crystal

Figure 8-2. Dislocation image and the selected area diffraction (SAD) pattern showing Kikuchi lines at the corresponding areas: (a) dislocations and (b) SAD at the foil normal (FN) direction
Figure 8-3. Illustration of measurements of burger’s vectors of dislocations
Figure 8-4. Dislocation image and the selected area diffraction (SAD) pattern showing Kikuchi lines at the corresponding areas: (a) dislocations and (b) SAD at a beam direction of -30°, \( g [0 \overline{2} 0] \)
Figure 8-5. Dislocation image and the selected area diffraction (SAD) pattern showing Kikuchi lines at the corresponding areas: (a) dislocations and (b) SAD at the beam direction of -20°, \( g [\bar{1} 1 1] \), the angle between the \( b \) and plane is 31°
Figure 8-6. Dislocation image and the selected area diffraction (SAD) pattern showing Kikuchi lines at the corresponding areas: (a) dislocations and (b) SAD at the beam direction of \(-12^\circ\), \(g\ [11\bar{1}]\), the angle between the \(b\) and plane is \(4^\circ\).
Figure 8-7. Dislocation image and the selected area diffraction (SAD) pattern showing Kikuchi lines at the corresponding areas: (a) dislocations and (b) SAD at the beam direction of $15^\circ$, $g\ [2\ 2\ 0]\]
Figure 8-8. Dislocation image and the selected area diffraction (SAD) pattern showing Kikuchi lines at the corresponding areas: (a) dislocations and (b) SAD at the beam direction of 30º, **g** [\(2\overline{2}00\)], the angle between \(\mathbf{b} 1/2(101)\) and plane is 40º, the angle between the \(\mathbf{b} 1/2(\overline{1}01)\) and plane is 42º.
Figure 8-9. Grain boundaries and many bending and thickness contours
Figure 8-10. Grain boundaries and dislocation-cell structures
Figure 8-11. Dislocation-cell structures and twin lamellas
Figure 8-12. Dislocations forming cell structures after fatigue at 0.2 Hz with a R ratio of -1
Figure 8-13. Persistent Lüder bands structures in 316 LN SS after fatigue
Figure 8-14. Stacking faults in 316 LN SS
Figure 8-15. Electron micrographs illustrating dislocation pile-ups near the grain boundary
Figure 8-16. Pile-up dislocations exhibiting a zig-zag feature due to slipping on different crystal planes
APPENDIX C

PROGRAM TO COMPUTE TEMPERATURE EVOLUTION
// Calculate the area of stress-strain curve of fatigue test.
// Data file should have a format that cycles are delimited by some text.
// Data are acquired starting from time 0 of each cycle
// Force is control signal, Strain is reference signal, Displacement is additional signal
// Data file must be raw file, not the file converted from excel, not unicode file

#include <iostream.h>
#include <stdlib.h>
#include <fstream.h>
#include <iomanip.h>
#include <string.h>
#include <math.h>
#include "InputLine.h"
// A header file for inputting fields delimited by blanks from the raw file
#define Gagelength (0.8*25.4*0.001)  // Gage length of specimen 0.8 inch (m)
#define Diameter (0.3*25.4*0.001)  // Diameter of specimen 0.3 in, (m)
#define Crossarea (3.1415926*0.15*0.15*25.4*25.4*0.000001)
// Area of cross section of specimen (m2)
#define PI 3.1415926
// Transformation of units from square inch to square m
#define GageVolume Crossarea*Gagelength
#define Density (7.8*1000000)  // Density of material 7.8 g/cm3 (g/m3)
#define SpecificHeat 0.46  // Specific heat of 316 LN steel 0.46 J/gdegreeC
#define ThermalConductivity 16.2714
// Thermal conductivity (16.2714 w/mK, w-watt, w = J/sec)
#define HeatCoefficient 15  // Convective heat coefficient, w/m2K, or J/m2Ksec
#define RoomTemp 20 //Room temperature 20 degree C
#define HeatConductionFactor 0.00000467
// Depend on heat conducting condition, material, shape, ambient temperature
#define Conductlength HeatConductionFactor*4.1*25.4*0.001
// Length of heat conduction part, m, 2 ends for conduction
// Specimne length 9 inch, (9-0.8)/2 = 4.1 inch on each side, 1.45 inch clamping part

to int main(int argc, char **argv){
    double stress1, stress2, strain1, strain2, force1, force2, deltae, firststress, firststrain,
    firstforce;
    // stress is stress, calculated from force and Crossarea
    // strain is strain, if not provided, can use disp/Gagelength ,
    // disp is displacement, force is force

double Area, AreaIncr, startarea, CalcArea, T1, T2, MaxTemp, CycleCompensator, n;
// Area is the area of hysterisis loop per cycle, T1 and T2 are temperatures

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double Frequency, cycletime;
// Frequency is the test frequency, cycletime is the time period of a cycle
int i, nf, cyclecount, startcount;
// cyclecount is the number of cycles read from data file,
// startcount is the cycle number of last recorded cycle
int ForceField, DispField, StrainField;

if(argc!=2){
    fprintf(stderr,"usage: parttemp inputfile\n");
    exit(1);
}

ofstream outClientFile("temp.txt", ios::out); // Can change to append mode if needed
    cout << "Output file: temp.txt" << endl;
if ( !outClientFile){
    cerr << "Output file could not be opened\n"
    exit(1);
}

CycleCompensator = 1.001; //Due to the cyclic softening of material
MaxTemp = 0;
T1 = RoomTemp;
startarea = 0;
startcount = 0;
cyclecount = 0;
n = 0.33;
InputLine il(argv[1]);

// Suppose the data file have four columns: force (KN), strain(mm/mm),
// displacement (mm), and time (sec)
// The order of these fields doesn't matter, but units does

outClientFile << setiosflags(ios::left) <<setw(11) << "Cycles" << "  " <<setw(10) << "Area(Pa)" << "  " <<setw(10) << "Tmp.(C)" << "  " <<setw(12) << "Time(s)" <<endl;

cout << "Calculating......." << endl;
while(il.get_line()>=0){
    // Process each cycle and calculate area
    if(il.get_NF()<= 3 && strcmp(il.get_Field(0), "Cycle")==0 && strcmp(il.get_Field(1), "Number=")==0){
        cyclecount = atoi(il.get_Field(2)); // cyclecount is the real number of cycles
        if (cyclecount ==0 ){

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fprintf(stderr, "Cycle count error\n");
exit(1);
}

nf = il.get_NF();
if(strcmp(il.get_Field(0), "Dimension=")==0)
  // Determine the field number of stress, strain and displacement
  // Number of fields are consistent backwards, not forwards
  for(i=1; i<nf;i++){
    if(strcmp(il.get_Field(i), "Force")==0)
      ForceField = nf-i;
    }
    if(strcmp(il.get_Field(i), "Length")==0)
      DispField = nf-i;
    }
    if(strcmp(il.get_Field(i), "Strain")==0)
      StrainField = nf-i;
    }
  }
}

if(il.get_NF()>=3 && strcmp(il.get_Field(1), "Frequency=")==0)
  // Determine the test frequency
  Frequency = atof(il.get_Field(2));
cycletime = 1/Frequency;
if(Frequency==0)
  fprintf(stderr, "Frequency reading error\n");
exit(1);
}

if(il.get_NF()>=4 && strcmp(il.get_Field(il.get_NF()-1), "0") == 0)
  // new cycle starts from time 0
  // ignore text lines between last point of previous cycle and new cycle
  // data file should have 4 fields of doubles
  Area = 0; // Area of each hysteresis loop of each cycle
  nf = il.get_NF();
  // The input line takes care of the order of inputing fields,
  // which can be rearranged.
  if(nf >= 4) { // get the data of the first cycle.
    force1= atof(il.get_Field(nf-ForceField));
    strain1 = atof(il.get_Field(nf-StrainField));
    //disp1 = atof(il.get_Field(nf-2));
    stress1 = force1*1000/Crossarea;
    //unit of force is KN, CrosArea is square mm, stress is MPa
    firststrain = strain1;
firstforce = force1;
firststress = stress1;

} else {
    fprintf(stderr, "Error in data file. Less than 4 fields(strain, force, disp,
time)\n");
    exit(1);
}

if(il.get_line() >= 0 && atof(il.get_Field(0)) != 0) {
    // The second record after the start cycle should be a record
    // with 4 fields. If EOF or text in the first field appears,
    // cycle isn't finished

    nf = il.get_NF(); // nf is the number of fields in this cycle
    if(nf >= 4) {
        // get the data of the second cycle
        force2 = atof(il.get_Field(nf-ForceField));
        strain2 = atof(il.get_Field(nf-StrainField));
        // disp2 = atof(il.get_Field(nf-2));
        stress2 = force2 * 1000 / Crossarea;
        deltae = strain2 - strain1;
        Area += deltae * (stress1 + stress2) / 2;
        // small area of tetragonal using mean of stress 1 and stress2
        // as height of rectangular (area of tetragonal)
    } else {
        fprintf(stderr, "Error in data file. Less than 4 fields(strain, force, disp,
time)\n");
        exit(1);
    }

} while(il.get_line() >= 0 && il.get_NF() >= 4) {
    // get other data in a cycle consecutively.
    // Drops out when cycle number=i line appears
    force1 = force2;
    strain1 = strain2;
    // disp1 = disp2;
    stress1 = stress2;

    // unit of force is KN, CrosArea is square mm, stress is MPa
    nf = il.get_NF();
    if(nf >= 4) {
        force2 = atof(il.get_Field(nf-ForceField));
        strain2 = atof(il.get_Field(nf-StrainField));
        // disp2 = atof(il.get_Field(nf-2));
        stress2 = force2 * 1000 / Crossarea;

    } 269
deltae = strain2-strain1;
Area += deltae*(stress1+stress2)/2;
// small area of tetragonal using mean of stress 1 and stress2
// as height of rectangular (area of tetragonal)
}
else{
  // may because cycle isn't complete, or data error with less fields than 4
  fprintf(stderr, "Error in data file. Less than 4 fields(strain, force, disp, time)\n ");
  fprintf(stderr, "Or Cycle %d wasn't complete\n", cyclecount);
  exit(1);
}
}

// Calculate the area between the first point in a cycle and the last point
deltae = firststrain - strain2;
Area += deltae*(firststress + stress2)/2;
}
else{
  fprintf(stderr, "Cycle %d wasn't complete\n", cyclecount);
  exit(1);
}

// After processing one cycle, calculate temperature increase in each cycle
// Keep a record of 1. Time; 2. cycle number 3. area 4. temperature
if (Area <=startarea){
  Area = startarea*CycleCompensator;
}
if ((cyclecount - startcount) == 1){
  // If cycles are continuous, which means
  // the data file records every cycle

  T2 = (Density*GageVolume*SpecificHeat*T1 + GageVolume*Area -
  2*ThermalConductivity*PI*Diameter/2*Diameter/2*\n  pow((T1 - RoomTemp),n)/Conductlength*cycletime -
  2*PI*Diameter/2*Gagelength*HeatCoefficient*(T1 -
  RoomTemp)*cycletime)/(Density*GageVolume*SpecificHeat);

  T1 = T2;

  if (MaxTemp < T1){
    MaxTemp = T1;
  }
}

// Fixed width of data column can be read by excel
outClientFile << setiosflags(ios::left|ios::showpoint|ios::fixed) <<
  setw(11) << cyclecount << " 
" << setw(10) << setprecision(5) << Area << " 
" // Area of the cycle, in Pa

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<< setw(10) << T1 << " " // Temperature after the cycle
<< setw(12) << cyclecount/Frequency
// can divide into smaller time units
<< endl;

} else {
    if (startarea != 0) {
        AreaIncr = ( Area - startarea)/(cyclecount-startcount);
        for(i=startcount+1;i<= cyclecount;i++){

            CalcArea = startarea + AreaIncr * (i-startcount);
            T2 = (Density*GageVolume*SpecificHeat*T1 \ 
                + GageVolume*CalcArea\ 
                -2*ThermalConductivity*PI*Diameter/2*Diameter/2*pow((T1 \ 
                -RoomTemp),n)/Conductlength*cycletime\ 
                -2*PI*Diameter/2*Gagelength*HeatCoefficient*(T1 \ 
                -RoomTemp)*cycletime)/(Density*GageVolume*SpecificHeat);

            T1 = T2;

            if (MaxTemp < T1){
                MaxTemp = T1;
            }

            // Fixed width of data column can be read by excel
            outClientFile << setiosflags(ios::left|ios::showpoint|ios::fixed)
                << setw(11) << i << " "
                << setw(10) << setprecision(5) << CalcArea << " "
                // Area of the cycle, in Pa
                >> setw(10)<< T1 << " " // Temperature after the cycle
                >> setw(12) << i/Frequency // can divide into smaller time units
                >> endl;
        }
    } else {
        CalcArea = Area;
        for(i=startcount+1;i<= cyclecount;i++){

            T2 = (Density*GageVolume*SpecificHeat*T1 \ 
                + GageVolume*CalcArea\ 
                -2*ThermalConductivity*PI*Diameter/2*Diameter/2*pow((T1 \ 
                -RoomTemp),n)/Conductlength*cycletime\ 
                -2*PI*Diameter/2*Gagelength*HeatCoefficient*(T1 \ 
                -RoomTemp)*cycletime)/(Density*GageVolume*SpecificHeat);

            T1 = T2;

            if (MaxTemp < T1){
                MaxTemp = T1;
            }

            // Fixed width of data column can be read by excel
            outClientFile << setiosflags(ios::left|ios::showpoint|ios::fixed)
                << setw(11) << i << " "
                << setw(10) << setprecision(5) << CalcArea << " "
                // Area of the cycle, in Pa
                >> setw(10)<< T1 << " " // Temperature after the cycle
                >> setw(12) << i/Frequency // can divide into smaller time units
                >> endl;
        }
    }
}
T1 = T2;

if (MaxTemp < T1)
    MaxTemp = T1;

// Fixed width of data column can be read by excel
outClientFile << setiosflags(ios::left|ios::showpoint|ios::fixed)
    << setw(11) << i << " "
    << setw(10) << setprecision(5) << CalcArea << " "
    // Area of the cycle, in Pa
    << setw(10) << T1 << " " // Temperature after the cycle
    << setw(12) << i/Frequency // can divide into smaller time units
    << endl;

startcount = cyclecount;
startarea = Area;
//end if,end new cycle
//end while
    cout << "Maximum temperature is " << MaxTemp << " degree C" << endl;
return 0;
}
VITA

Hongbo Tian was born in Shenyang, Liaoning Province, People's Republic of China (PRC). He attended schools in Shenyang, where he graduated from Shenyang Second High School in August 1992. During September 1992 to August 1996, he was pursuing his undergraduate study at the University of Science and Technology, Beijing (USTB), PRC, where he received his Bachelor of Science degree in Materials Science. In September 1996, he entered the Master's program in Materials Science and Engineering at USTB, where he obtained the Master's degree in June 1999. He came to the University of Tennessee and enrolled in the doctoral program in Materials Science and Engineering department in 1999. The Doctor of Philosophy degree was received in December 2003.