MULTI-SCALE COMPUTATIONAL MODELING OF NI-BASE SUPERALLOY BRAZED JOINTS FOR GAS TURBINE APPLICATIONS

DISSERTATION

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ABSTRACT

Brazed joints are commonly used in the manufacture and repair of aerospace components including high temperature gas turbine components made of Ni-base superalloys. For such critical applications, it is becoming increasingly important to account for the mechanical strength and reliability of the brazed joint. However, material properties of brazed joints are not readily available and methods for evaluating joint strength such as those listed in AWS C3.2 have inherent challenges compared with testing bulk materials. In addition, joint strength can be strongly influenced by the degree of interaction between the filler metal (FM) and the base metal (BM), the joint design, and presence of flaws or defects. As a result, there is interest in the development of a multi-scale computational model to predict the overall mechanical behavior and fitness-for-service of brazed joints. Therefore, the aim of this investigation was to generate data and methodology to support such a model for Ni-base superalloy brazed joints with conventional Ni-Cr-B based FMs.

Based on a review of the technical literature a multi-scale modeling approach was proposed to predict the overall performance of brazed joints by relating mechanical properties to the brazed joint microstructure. This approach incorporates metallurgical characterization, thermodynamic/kinetic simulations, mechanical testing, fracture mechanics and finite element analysis (FEA) modeling to estimate joint properties based
on the initial BM/FM composition and brazing process parameters. Experimental work was carried out in each of these areas to validate the multi-scale approach and develop improved techniques for quantifying brazed joint properties.

Two Ni-base superalloys often used in gas turbine applications, Inconel 718 and CMSX-4, were selected for study and vacuum furnace brazed using two common FMs, BNi-2 and BNi-9. Metallurgical characterization of these brazed joints showed two primary microstructural regions; a soft, ductile α-Ni phase that formed at the joint interface and a hard, brittle multi-phase centerline eutectic. CrB and Ni$_3$B type borides were identified in the eutectic region via electron probe micro-analysis, and a boron diffusion gradient was observed in the BM adjacent to the joint. The volume fraction of centerline eutectic was found to be highly dependent on the extent of the boron diffusion that occurred during brazing and therefore a function of the primary process parameters; hold time, temperature, FM/BM composition, and joint gap.

Thermo-Calc$^\text{TM}$ and DICTRA$^\text{TM}$ simulations were used to model the BM dissolution, isothermal solidification and phase transformations that occurred during brazing to predict the final joint microstructure based on these process parameters. Good agreement was found between experimental and simulated joint microstructures at various joint gaps demonstrating the application of these simulations for brazed joints. However, thermodynamic/kinetic databases available for brazing FMs were limited.

A variety of mechanical testing was performed to determine the mechanical properties of CMSX-4/BNi-2 and IN718/BNi-2 brazed joints including small-scale tensile tests, standard-size butt joints and lap shear tests. Small-scale tensile testing
provided a novel method for studying microstructure-property relationships in brazed joints and indicated that both joint strength and ductility decrease significantly with an increase in the volume fraction of centerline eutectic. In-situ observations during small-scale testing also showed strain localization and crack initiation occurs around the hard, eutectic phases in the joint microstructure during loading. The average tensile strength for standard-size IN718/BNi-2 butt joints containing a low volume fraction of centerline eutectic was found to be 152.8 ksi approximately 90% of the BM yield strength (~170 ksi). The average lap shear FM stress was found to decrease from 70 to 20 ksi for IN718/BNi-2 joints and from 50 to 15 ksi for CMSX-4/BNi-2 as the overlap was increased from 1T to 5T due to non-uniform stress/strain distribution across the joint.

Digital image correlation techniques and FEA models of the lap shear brazed joints were developed to assess the strain distributions across the overlap. Results were used to validate the use of damage zone models for predicting the load carrying capacity of lap shear brazed joints and suggest that the damage zone is independent of the overlap length.

To account for the presence of flaws and defects in fitness-for-service assessments of brazed joints determination of the average fracture toughness ($K_{IC}$) is necessary. Currently no standard exists to measure the $K_{IC}$ for brazed joints, so three test methods were evaluated in this investigation on IN718/BNi-2 brazed joints. The compact tension and double cantilever beam test methods were found to give the most conservative $K_{IC}$ values of 16.42 and 14.42 ksi√in respectively. Linear-elastic FEA models of the test specimens were used to validate the calculated $K_{IC}$ values. Similar to
joint strength the fracture toughness appeared to be strongly influenced by the volume fraction of centerline eutectic phases.

The data and methodology generated in this initial study provides validation for the proposed multi-scale computational model by demonstrating microstructure-property relationships in brazed joints and the ability to predict joint microstructure using simulation tools. Furthermore, an experimental framework and new techniques including small-scale tensile testing, digital image correlation and fracture mechanics were established to assist in future modeling efforts. Ultimately, successful development and implementation of multi-scale computational models for brazed joints will allow for the optimization of BM/FM compositions, brazing process optimization, improved reliability of brazed joints, and more efficient design and analysis of brazed components by accounting for the properties of the joint. In addition, the overall multi-scale modeling approach demonstrated in this investigation may also be applied for dissimilar joints in general, for example dissimilar metal welds used in oil and gas, petrochemical, nuclear and power generation industries.
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CHAPTER 1

1. INTRODUCTION

The gas turbine engine industry is experiencing tremendous growth in both aerospace and land-based power generation applications due to the worldwide increasing demand for commercial/military aircraft and electricity correspondingly [1, 2]. Due to competitive pressure within the market tremendous advancements in gas turbine technology have been made in the past few decades to improve the overall efficiency of the gas turbine engine.

This has been primarily accomplished by increasing the turbine entry temperature (TET). As shown in Figure 1.1 from Reed et al. [3] the TET has increased nearly 800°C over the past 60 years from approximately 700°C in 1940 to over 1400°C in recent years. This has largely been made possible by developing advanced cooling designs for the turbine blades and more recently using of thermal barrier coatings (TBC) to insulate the underlying blade material from the exhaust heat. Aside from enhancing the component design, continuous improvement of heat resistant materials for engine components such as nickel-based superalloys in directionally solidified (DS) and single crystal (SC) form has also significantly contributed to the increase in efficiency.
Successful and cost-effective implementation of heat resistant materials in the gas turbine engine requires the ability to join these materials effectively. Precipitate strengthened Ni-base superalloys are inherently difficult to join through conventional fusion joining processes, such as gas tungsten arc welding (GTAW), due to their tendency for hot cracking at the weld. As a result, high temperature brazing is often used to join these materials and in some cases is the only applicable joining method to avoid cracking.

In brazing a filler metal (FM) preferentially melts and flows in a narrow gap between two base materials (BM) to create a strong metallurgical bond on solidification
Due to the low joining temperatures brazing minimizes cracking and also produces low distortion and residual stress within the component in many cases eliminating the need for post-weld heat treatments. In addition, brazing can be used to join a wide range of similar and dissimilar materials such as steel, aluminum, nickel, and titanium alloys as well as non-metallic materials such as graphite or ceramics to provide material combinations for lightweight in aerospace turbine engines [5, 6, 7, 8].

For such critical applications, it is important to account for the brazed joint strength and mechanical behavior in the design of brazed components. Despite the long history and success of high temperature brazing there is still limited information on the mechanical behavior of braze joints. Conventional test methods for obtaining material properties such as elastic modulus, YS, or UTS for bulk materials or welded joints are not straightforward applicable for brazed joints. This is primarily due to the inherent narrow joint gaps and brittle micro-constituents produced in brazing.

As a result, there is a general lack of data on brazed joint properties with no databases or standard guidelines available for design engineers to use like the Metallic materials properties development and standardization (MMPDS) document for bulk materials [9]. In addition, joint properties have been shown to be strongly influenced by the joint clearance (gap), the degree of interaction between the BM and FM, the presence of flaws or defects and joint design [4]. The cost and time required to develop a similar database for brazed joints with such a wide range of materials and processing conditions available is not practical if possible at all. Therefore, the mechanical strength of brazed joints is often evaluated on a joint-to-joint basis depending on the application primarily.
by the component manufacturer. This can be very expensive when dealing with high-cost materials such as Ni-base superalloys, so test results are often kept proprietary and rarely published in the technical literature.

Instead it would be of great benefit to develop a multi-scale computational model based on the physical metallurgy and brazing parameters that is capable of predicting brazed joint mechanical behavior and fitness-for-service. The aim of this project, therefore, is to develop data and methodology for such a model through experimentation on Ni-base superalloy brazed joints with Ni-Cr-B based FMs. This is a well-known brazing system and has been used successfully in gas turbine applications for over 60 years. Based on a review of the relevant literature a multi-scale modeling approach is proposed these brazed joints that incorporates metallurgical interactions, thermodynamic/kinetic simulations, mechanical testing, fracture mechanics and FEA modeling to predict the overall strength of the joint. Experimental techniques in these areas were then developed and carried out to provide a framework and validation of the multi-scale modeling approach. This document is organized into 10 chapters.

Chapter 2 begins with a review of the relevant technical literature on high temperature brazing used in gas turbine applications. This includes a brief introduction of the gas turbine engine and nickel-based superalloy materials used for high temperature gas turbine components. A background on the specific FMs, wetting behavior, metallurgy and typical joint microstructure observed when brazing these materials is provided. These basic principles have been well established for brazing Ni-base superalloys and are important to understand before attempting to model the process. Previous work on
analytical and numerical modeling of brazed joint microstructure for these alloys will also be discussed.

Chapter 3 provides a summary of relevant literature on the mechanical behavior and methods for evaluating strength and toughness of brazed joints. Historical data and typical results using the two most common test methods, the butt and lap joint, will be discussed along with the inherent issues associated with these geometries. Methods developed for measuring fracture toughness of brazed joints and application of fitness-for-service assessments for brazed joints are also introduced. Studies using finite element models to predict stresses and strains at the joint and the challenges in using this technique for brazed joints will also be discussed.

Based on the background covered in Chapters 2 and 3, a multi-scale modeling approach is outlined in Chapter 4 along with the specific research objectives and tasks for this work. The tasks are designed to develop data and methodology for the components of the multi-scale model and be applicable for other braze alloy systems.

Chapters 5 through 8 provide the results and discussion of the experimental work performed in this study. Chapter 5 focuses on metallurgical characterizations of brazed joints using two nickel-based superalloys, Inconel 718 and Alloy CMSX-4, with two commonly used FMs BNi-2 and BNi-9. Compositional analysis, micro-hardness, and dispersion parameters of micro-constituents in these brazed joints will be analyzed. In addition, the influence of process parameters (hold time, joint gap, brazing temperature, and FM/BM composition) on joint microstructure will be evaluated.
In Chapter 6, Thermo-Calc and DICTRA software is used to predict the joint microstructure observed in Chapter 5 by calculating dissolution, isothermal solidification, and equilibrium solidification. Additional, experimental brazing was carried out to further validate the simulations. In Chapter 7, the mechanical behavior of brazed joints is investigated using standard-size butt joints, small-scale tensile tests and lap shear test methods. Several new approaches including in-situ small-scale testing, digital image correlation, and damage zone models are introduced for predicting brazed joint failures. In Chapter 8, an evaluation of fracture mechanics test methods for measuring the fracture toughness (KIC) of brazed joints is performed using IN718/BNi-2 joints to provide a basis for fitness-for-service FFS assessments. In addition, FEA models of selected test specimens in both Chapter 7 and 8 will be generated to compare with experimental results.

Based on results and discussion in Chapters 5-8, important findings and conclusions are provided in Chapter 9 along with a summary of how these methods can be incorporated in a multi-scale model. Finally, suggestions for future work and modeling efforts for brazed joints are provided in Chapter 10.
CHAPTER 2

2. BRAZING BACKGROUND

2.1 Introduction

By definition brazing comprises a group of joining processes in which coalescence is produced by heating to temperatures above 450°C (940°F) and using a FM that must have a liquidus temperature above 450°C and below the solidus temperature of the BM [10]. On heating the FM preferentially melts and flows between two BM surfaces in a narrow gap (typically between 0.001 – 0.004 inch) by capillary force. Diffusion and metallurgical interactions between the liquid FM and solid BM create a strong metallurgical bond between the two parts on cooling and solidification of the FM.

For brazing superalloys, Ni-base FMs containing melting point depressant (MPB) elements such as boron or silicon are used. For these FMs, the extent of metallurgical interaction between the BM and FM can produce a wide range of joint microstructures that may contain brittle or ductile phases depending on the process parameters and FM composition. The brittle micro-constituents that form in the joint are often deleterious to the mechanical strength and ductility of the joint. Therefore, when modeling and designing brazed joints it is important to account for these micro-constituents, for their properties and for properties across the joint.
2.2 Nickel-based Superalloys

Ni-base superalloys have been used in the hot sections of turbine engines for over 75 years due to their excellent high temperature strength and corrosion resistance [11]. A superalloy is defined as a material that can successfully operate as high as 25% lower than its melting point, while being able to sustain adequate mechanical strength. For example, the ultimate tensile strength of a Ni-base superalloy at a temperature of 850°C was approximately 1200 MPa (~175 ksi) [12, 13]. The remarkable high temperature properties of superalloys can be directly related to their microstructure which consists of an austenitic face-center cubic (FCC) crystal structure (γ) with a high volume fraction (~0.7) of coherent γ’ (Ni₃(Al,Ti)) or γ” (Ni₃Nb) strengthening precipitates [11]. Full solution and age heat treatments are required after casting to form these strengthening precipitates. Other intermetallic compounds such as carbides, borides or silicides may also be observed in Ni-base superalloys and in small quantities can be beneficial by preventing dislocation movement and grain boundary sliding. However, large quantities of these phases or undesirable morphologies may cause material embrittlement.

Advancements in superalloy compositions, heat treatments, and casting techniques over the years have led to a transition from conventionally cast wrought alloys, to directionally solidified (DS) and single crystal (SC) superalloys for improved material properties and temperature capabilities [14, 15, 16]. The advantage of SC alloys over wrought or DS alloys is improved creep life due to the absence of grain boundaries [17]. However, advanced casting methods using grain selectors and carefully controlled
solidification is required to achieve the SC form. Gell et al. provide an excellent overview of the development of DS and SC superalloys turbine blades [18]. [19, 20, 21, 22]

While superalloys are often required for the high stress and temperature environment in gas turbine engines they are inherently difficult to join. The excessive heating and melting/re-solidification involved in conventional fusion joining processes, such as gas tungsten arc welding (GTAW) and laser or electron beam welding, often leads to solidification cracking in the weld metal or liquation cracking in the heat affected zone (HAZ). In addition, the high heat input leads to dissolution or overaging of \( \gamma' \) or \( \gamma'' \) strengthening precipitates creating heat affected zone softening [23, 24, 25].

Figure 2.1, shows solidification cracks and HAZ damage observed in GTA welds of Inconel 718. In addition, welding often produces high distortions and residual stress. After welding a full solution and age, post-weld heat treatment is typically required to regain the superalloy properties which can be expensive and lead to other complications such as strain age cracking or over aging in the BM [26]. Due to these weldability issues, high temperature brazing has been a successful and cost-effective method for joining these materials. Since brazing occurs at temperatures below the BM’s melting temperature, typically from 800°C-1100°C for Ni-base superalloys, the negative effects of fusion joining are avoided. In some cases brazing can be performed below the \( \gamma' \) or \( \gamma'' \) solvus temperature to avoid the need for a post-braze heat treatment or the brazing cycle can be combined in one heat treatment to save costs.
Figure 2.1: Weldability issues with nickel-based superalloys include solidification cracking (left) and heat affected zone softening (right) [25].

2.3 Brazing Fundamentals

Nickel-based brazing FMs have been widely used for the manufacture and repair of gas turbine engines since they were first introduced by the late Robert L. Peaslee in the 1950’s [27]. Some commonly brazed gas turbine components, shown in Figure 2.2, include honeycomb seals (left), exhaust plugs (center) and nozzles (right) [28, 29, 30]. Wide gap brazing is also frequently used in the repair of nozzles and vanes that have cracked due to thermal-mechanical fatigue during service [31]. Due to the extensive use in these applications a number of groups have studied the wetting properties, FM compositions, metallurgy and microstructures involved in brazing with Ni-base FMs.
2.3.1 Wetting

Wetting and spreading of the liquid FM in contact with the solid BM material is critical for successful brazing [33]. Due to high surface energy in most solids the thermodynamic driving force for wetting to occur is the reduction of free surface energy by replacing the solid/gas interface with a solid/liquid interface [34]. The classic model for wetting is based on the behavior of a drop of liquid on a solid with a flat surface in a bulk, vapor phase as shown in Figure 2.3. According to Young’s equation (Eqn. 2.1) there is a relationship between the contact angle ($\theta$), the surface tension of the liquid ($\gamma_{lv}$), the interfacial tension between the liquid and solid ($\gamma_{ls}$), and the surface free energy of the solid ($\gamma_{sv}$) where:

$$\gamma_{sv} = \gamma_{sl} + \gamma_{lv} \cos \theta$$  \hspace{1cm} (2.1)

For wetting to occur the contact angle should be less than $90^\circ$. If the liquid droplet is between a narrow gap (typically between 0.001 -0.006 inch) the combination of surface
tension and adhesive forces between the solid and liquid result in capillary motion of the liquid to flow into the joint. Both capillary action and wetting can be strongly influenced by the surface roughness, cleanliness, oxide layers and temperature or hold time [34, 35].

![Diagram of wetting behavior](image)

Figure 2.3: Wetting behavior of liquid droplet on solid surface can be described by Young's equation and balance between liquid, solid and vapor surface tensions.

Brazing of non-oxidizing metals like copper, nickel, steel and precious metals is relatively easy as liquid FM films easily wet and spread on the surface of these materials. However, ceramics, titanium, aluminum and other materials that form surface oxides have lower surface energies and are often difficult to braze due to poor wetting characteristics. This includes highly alloyed Ni-base superalloys that have Al, Ti or Cr added to their composition. For these materials, fluxes or controlled atmospheres are used during brazing to remove or prevent the formation of surface oxides to enable the FM to wet and flow on the BM. Brazing of Ni-base superalloys is typically performed under high vacuum less than $10^{-5}$ torr to avoid oxidation and flux entrapment. Other techniques such as nickel plating, grit blasting or metallization of ceramics also assist in wetting and spreading of the FM. While wetting is an important consideration in many brazing
operations, it was not a primary concern in this study as vacuum furnace brazing was used and wetting properties of the FMs investigated have been well established. For other BMs and/or FMs wetting tests involving measurements of contact angle or spreading area may be necessary prior to brazing test specimens or actual components.

2.3.2 Filler Metals

There are a large number of brazing FMs available and new FMs are continuously being developed for specific applications. The AWS A5.8 Specification for Filler Metals for Brazing provides a list of common FM groups including; silver, gold, aluminum, magnesium, nickel, cobalt and copper based FMs [36]. While some FMs are simply pure metal, most are alloyed with additional melting point depressant (MPD) elements for near eutectic compositions to lower the melting temperature and allow for quick, uniform melting. For Ni-base FMs, MPD elements are typically boron, silicon or phosphorus, that result in a eutectic reaction with nickel during FM solidification as illustrated in the Ni-rich portion of the Ni-B binary phase diagram in Figure 2.4 from Liao et al. [37].
Figure 2.4: Eutectic reaction in Ni-rich portion of Ni-B phase diagram is utilized for brazing Ni-base superalloys [37].

At a composition of approximately 3.6 wt% boron (~17 at%) the alloy melting temperature is 1093°C compared to the 1455°C for pure nickel. Si and P give similar reactions with Ni which allow for brazing temperatures well below the BM solidus. Industry standard nickel-based braze alloys (BNi-), their liquidus/solidus temperatures and recommended brazing temperature ranges from AWS A5.8 are listed in Table 2.1 [36]. The recommended brazing temperatures for these FMs range from 927-1204°C which are often considered as ‘high temperature’ brazing FMs and brazing is performed 50-100°C above the liquidus temperature to ensure complete melting and capillary flow of the FM [4]. Chromium and iron are often added for additional strength or corrosion resistance and molybdenum or tungsten can also be included for high temperature properties. Brazing FM is available in pre-alloyed powder, wire, foil or tape form. Powder is typically mixed with a water or organic based binder to create a braze paste
that can be easily applied in the joint area. Two of the most common nickel-based FMs, BNi-2 and BNi-9, will be used in both paste and foil forms in this study.

Table 2.1: Chemical composition, liquidus/solidus temperatures and recommended brazing temperature range from AWS A5.8 for Ni-based FMs used for brazing Ni-base superalloys [36].

<table>
<thead>
<tr>
<th>AWS Classification</th>
<th>Ni</th>
<th>Cr</th>
<th>B</th>
<th>Si</th>
<th>Fe</th>
<th>P</th>
<th>W</th>
<th>Liquidus (°C)</th>
<th>Solidus (°C)</th>
<th>Brazing Temp. Range (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BNi-1</td>
<td>bal.</td>
<td>14</td>
<td>3.125</td>
<td>4.5</td>
<td>4.5</td>
<td>0.02</td>
<td>-</td>
<td>977</td>
<td>1038</td>
<td>1066-1204</td>
</tr>
<tr>
<td>BNi-2</td>
<td>bal.</td>
<td>7</td>
<td>3.125</td>
<td>4.5</td>
<td>4</td>
<td>0.02</td>
<td>-</td>
<td>971</td>
<td>999</td>
<td>1010-1177</td>
</tr>
<tr>
<td>BNi-5</td>
<td>bal.</td>
<td>19</td>
<td>0.03</td>
<td>10.1</td>
<td>-</td>
<td>0.02</td>
<td>-</td>
<td>1079</td>
<td>1135</td>
<td>1149-1204</td>
</tr>
<tr>
<td>BNi-6</td>
<td>bal.</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>11</td>
<td>877</td>
<td>877</td>
<td>927-1093</td>
</tr>
<tr>
<td>BNi-8</td>
<td>bal.</td>
<td>-</td>
<td>7</td>
<td>-</td>
<td>-</td>
<td>0.02</td>
<td>-</td>
<td>982</td>
<td>1010</td>
<td>1010-1093</td>
</tr>
<tr>
<td>BNi-9</td>
<td>bal.</td>
<td>15</td>
<td>3.6</td>
<td>-</td>
<td>1.5</td>
<td>0.02</td>
<td>-</td>
<td>1055</td>
<td>1055</td>
<td>1066-1204</td>
</tr>
<tr>
<td>BNi-10</td>
<td>bal.</td>
<td>11.5</td>
<td>2.5</td>
<td>3.5</td>
<td>3.5</td>
<td>0.02</td>
<td>16</td>
<td>970</td>
<td>1105</td>
<td>1149-1204</td>
</tr>
</tbody>
</table>

2.3.3 Heating Methods

A number of heating methods can be utilized for brazing including torch, induction, furnace, resistance, lasers or electron beam heat sources. Furnace brazing is most widely used for brazing Ni-base superalloys for two primary advantages. First, the atmosphere can be carefully regulated inside the furnace to protect the parts from oxidation during heating by utilizing high-purity gases or a high vacuum. Second, it provides the ability to control the heating and cooling cycles with computerized instrumentation [4]. This allows for good repeatability of the brazing cycle and yields the highest quality brazed joints. An example of a batch-type furnace for brazing is shown in Figure 2.5A [38]. Typical heat cycles used for furnace brazing often consist of several hold times at different temperatures as shown schematically in Figure 2.5B.
Figure 2.5: (A) Batch-type vacuum furnaces often used for brazing Ni-base superalloys [38]. (B) Typical brazing heat cycles include several hold times; 1. outgas of organic contaminants, 2. homogenize part temperature before brazing and 3. hold time at brazing temperature.

The first hold at low temperatures allows for organics in the binder or paste to outgas from the braze deposit [39]. The second hold just below the FM solidus temperature is used to ensure uniform heating throughout the part before heating quickly to the brazing temperature. The last hold is at the brazing temperature and typically ranges from 0-60 minutes to allow sufficient time for the FM to wet the BM and flow into the joint. The primary process parameters that need to be selected in a brazing cycle are the FM, joint gap, brazing temperature and hold time. Heating and cooling rates of the brazing cycles are selected to control distortion, residual stress and meet the required metallurgical properties and production needs [40].

Additional diffusion, homogenization and/or aging steps following the brazing cycle are often used in transient liquid phase (TLP) bonding or diffusion brazing to allow for complete diffusion of BM and FM elements across the joint [41]. However, TLP
bonding or diffusion brazing require long heat cycles that can be well over 20 hours and are typically only utilized for highly stressed joints. For conventional brazing, hold times are much shorter to reduce cost and avoid damaging the BM properties. The cooling rates of the brazing cycles are selected to control distortion, residual stress and meet the required metallurgical properties and production needs [40].

2.3.3 Brazing Metallurgy

Due to their wide use in gas turbine applications, a number of research groups have studied the metallurgical steps that occur when brazing with nickel-based FMs [42, 43, 44, 41]. Upon melting of the FM a metallurgical bond is created in three primary stages; dissolution of the BM, diffusion of MPD elements across the solid/liquid interface and subsequent solidification of the FM either isothermally or upon cooling.

Figure 2.6, adopted from Gale and Butts [41], shows a schematic of these metallurgical stages for a typical braze joint with a substrate of material ‘A’ bonded with a brazing FM (shaded) consisting of A and MPD elements ‘B’. The initial composition of the liquid FM is chosen to be near the eutectic composition \( C_0 \) at the bonding temperature and the initial composition of the solid BM \( C_A \) is free of MPD elements. Once the liquid FM has wet both substrates (Figure 2.6A) the initial compositions are in a non-equilibrium condition at the brazing temperature \( T_b \). As shown in Figure 2.6B, partial dissolution of the substrate material is required to adjust the composition of the liquid to \( C_L \). This effectively widens the joint gap until the composition of the solid BM adjacent to the liquid is brought to \( C_a \). At this point equilibrium is achieved and dissolution will stop. The dissolution stage does not require long range diffusion and the
activation energy is usually low, so this stage happens quickly at $T_b$ [44]. After the
dissolution process is complete, MPD elements (B) in the FM continue to diffuse into the
solid substrate material as shown in Figure 2.6C. Local equilibrium is assumed to be
maintained at the solid/liquid interface, i.e. the composition of the liquid and solid are
fixed at $C_L$ and $C_s$, so as B diffuses into the substrate the liquid region must narrow as the
interface moves to maintain equilibrium at $T_b$. This effect is known as isothermal
solidification and results in the epitaxial growth of a soft, ductile phase from the $\gamma/\gamma'$
substrate. This phase continues to grow into the liquid region while holding above the
FM liquidus temperature due to long range diffusion of MPD elements from the liquid
into the solid BM. The kinetics of this process are relatively slow compared to the
dissolution stage, however given enough time the joint may completely solidify at the
brazing temperature due to this phenomenon. For most brazing cycles however
isothermal solidification does not complete due to the relatively short hold times and
some liquid FM will remain in the joint. On cooling from $T_b$, equilibrium will be
maintained in the liquid and solid until the eutectic temperature ($T_e$) is reached as shown
in Figure 2.6D. As cooling continues below $T_e$, any remaining FM liquid will form a
eutectic constituent along the joint centerline consisting of A and A$_3$B$_7$ intermetallic
phases.
Figure 2.6: Metallurgical stages of nickel based brazing for a BM with composition $C_A$ and FM with initial composition $C_0$. Non-equilibrium initial condition (A) leads to BM dissolution and widening of the interlayer (B). Holding at $T_b$ results in MPD diffusion of B elements into the BM (C) and subsequent isothermal solidification. Cooling and subsequent solidification of the remaining liquid FM remaining forms centerline eutectic consisting of A and $A_xB_y$ intermetallic phases (D).

2.3.4 Joint Microstructure

These metallurgical stages produce a relatively complex joint microstructure that can play an important role on the overall properties. Several examples of brazed joint
microstructures using Ni-base FMs taken from the literature are shown in Figure 2.7 [45, 43, 46]. Three distinct microstructural regions are typically identified in brazed joints with Ni-base FMs described below moving from the BM into the brazed joint [47]:

1. **Diffusion-affected zone (DAZ)**, a region of the BM enriched in MPD elements that can either dissolve in the BM solid solution or react to form precipitates. This region is a result of the MPD diffusion in the solid at elevated temperatures.

2. **Isothermally solidified zone (ISZ)**, is a single phase region that forms along the joint interface and extends into the joint gap due to depletion of MPDs in the liquid FM during holding at the brazing temperature. The driving force for the formation of this region is the compositional gradient (chemical potential) between the liquid FM and BM. The width of this region depends on the extent of MPD element diffusion that occurs while above the FM liquidus temperature.

3. **Athermally solidified zone (ASZ) or centerline eutectic**, consists of brittle intermetallic phases that form along the joint centerline due to insufficient time for isothermal completion of the solidification process, with cooling as the main driving force.

Most FMs containing MPD elements will consist of these three microstructural regions, however pure metals like silver or gold are used as FMs the joint may only consist of a single ductile phase as has been reported by Flom et al [48]. For nickel-based FMs, the intermetallic phases in the ASZ are typically hard, brittle borides, silicides or phosphides that can be detrimental to the overall joint properties [43, 6]. Joints that contain these eutectic constituents exhibit lower shear and ultimate tensile strength as
well as reduced fracture toughness [49, 50]. Therefore, it is important to account for the formation of these phases and their properties when modeling and designing brazed joints.

Figure 2.7: Examples of brazed joint microstructures with Ni-base FMs. ASZ and ISZ regions are observed when brazing with these materials [46].

Most research efforts in high temperature brazing with Ni-base superalloys have been focused on reducing or eliminating these detrimental phases from the joint using transient liquid phase (TLP) bonding or diffusion brazing. These techniques utilize long hold times (2-20 hours) at or near $T_b$ to allow for complete isothermal solidification of the joint to avoid the formation of these constituents [51, 52]. With complete isothermal solidification and additional post-braise homogenization/aging heat treatments there may
be no apparent joint visible in the material and joint strength can reach 90% or higher of the BM strength \([44, 49, 53]\). Pouranvari et al. discuss the differences in joint microstructure and properties between normal brazing and TLP bonding \([54]\). Despite the high joint strength associated with TLP bonding or diffusion brazing these processes require long hold times at high temperatures and are typically only applied for highly stressed joints. In conventional brazing shorter brazing cycles are used to reduce the time and cost of operation and avoid excessive heating of the BM.

Therefore, isothermal solidification begins during the brazing process, but may not complete. The time required for complete isothermal solidification to occur \(t_{iso}\) depends on the solubility and diffusivity of the MPD elements in the BM and can vary from minutes to hours depending on the BM/FM composition, interlayer thickness (joint gap), brazing temperature or hold time \([55, 56, 57]\). For example, the influence of joint gap on microstructure can be observed in the lower image in Figure 2.7. At the internal joint area where the joint gap is narrow complete isothermal solidification has occurred, whereas the fillet regions with large joint gaps contain the ASZ eutectic phases.

However, even if isothermal solidification does not completed as is the case for most brazed joints, the volume fraction of eutectic constituents can also influence the joint strength, fracture toughness and fatigue life. \([50, 58]\). Yu et al. showed this for wide-gap brazed joints and described brazed joints as a composite structure in which joint strength \(\sigma_B\) is based on the rule of mixtures \([59]\):

\[
\sigma_B = (1 - f)\sigma_F + f\sigma_G + \sigma_i
\]  

\[ (2.2) \]
where $f$ is the volume fraction of the solid-solution, $\sigma_F$ and $\sigma_G$ are the strength of the eutectic and solid-solution phases, respectively, and $\sigma_i$ is the effect of interaction between the micro-constituents. In this case, the fraction of detrimental eutectic phases ($f_E$) can be taken as:

$$f_E = 1 - f$$

(2.3)

For brazing FMs $\sigma_F$ is typically significantly less than $\sigma_G$ therefore as shown by Eqn. 2.2 and 2.3, decreasing the fraction eutectic by allowing for more diffusion and growth of the solid-solution phase leads to an increase in the overall joint strength. Therefore, it is important to account for the size and distribution of these phases, for their properties and their influence on the overall joint properties. As a result, several groups have studied phase equilibria for braze alloy systems including binary Ni-B, Ni-Si, Ni-P and ternary Ni-Cr-B, Ni-Si-B, Ni-Al-B [60, 61, 62]. Vargas et al. identified the primary phases in BNi-2 FM for pure Ni/BNi-2 couples as Ni$_3$B and CrB type borides, $\gamma'$ Ni$_3$Si and $\alpha$-Ni phase within a multi-component eutectic matrix [42]. Others have reported similar phases in actual joints between commercially available Ni-base superalloys or stainless steels [63, 64] for a range of different heating methods, various BMs and brazing parameters [56, 63]. In general, under normal brazing conditions the three primary microstructural regions should be expected in all joints brazed with Ni-base FMs.

While the formation of brittle, eutectic constituents is known to occur during high temperature brazing there have been very few attempts to quantify dispersion parameters such as volume fraction or average particle size and hardness properties of the individual
phases within the ASZ. This is likely due to the fact that most research has focused on eliminating these phases rather than accounting for them. In addition, the complex microstructure and narrow joints in brazing create a challenge for performing accurate metallurgical characterization throughout the joint. As a result, reliable data on dispersion parameters and properties of individual phases within the centerline eutectic in the literature is rare.

2.5 Thermodynamic and Kinetic Simulations

Since the mechanical properties of brazed joints have been shown to be directly related to their microstructure ($f_e$), the ability to predict joint microstructure becomes a critical component of an accurate multi-scale model. Typically brazed joint microstructure can be realized through extensive experimental brazing and destructive metallurgical characterizations [55, 65]. However, with an almost infinite number of materials and process parameters for brazing the time and cost of this approach is often impractical. Instead it would be useful to model and predict the joint microstructure based on the material compositions, joint design and brazing process.

This has primarily been accomplished by calculating the diffusion of MPD elements during the initial BM dissolution and subsequent isothermal solidification stages [66]. According, to Illingworth et al. diffusion in both the liquid and solid can be assumed to follow Fick’s second law [66]:

$$\frac{\partial C}{\partial t} = D_s \frac{\partial^2 C}{\partial x^2}$$  \hspace{1cm} (2.4)

where the change in concentration in a volume of either solid or liquid is proportional to the diffusivity of the diffusing elements and the second derivative of the concentration.
gradient. By applying appropriate boundary conditions, analytical solutions for this equation can be developed to estimate the diffusion distance and time required for dissolution or isothermal solidification. For example the initial dissolution step the general solution for the interface displacement (x) follows a square root law for time (t) in the form of [45]:

\[ x = K_1 \sqrt{4D_L t} \]  \hspace{1cm} (2.5)

Where \( D_L \) is the BM solute diffusion in the liquid and \( K_1 \) is a temperature dependent dissolution parameter that depends on the solidus and liquidus compositions of the alloy system. These equations were derived for a pure metal FM and assume the solute diffusivity in the liquid phase \( D_L \) is the controlling mechanism. From Eqn. 2.5 the time required for complete dissolution \( (t_{diss}) \) for an initial joint gap \( (w_0) \) can then be calculated as [67]:

\[ t_{diss} = \frac{W_0^2}{16K_1^2D_L} \]  \hspace{1cm} (2.6)

For the Ni-B system the time required for dissolution is in the range of seconds and can be considered to occur immediately once the FM has wet the BM. Once the BM dissolution has occurred and the composition of the solid/liquid interface is at equilibrium the interface begins to move in the opposite direction as now MPD elements in the FM begin to diffuse into the BM beginning the isothermal solidification process. Similar equations can be used to describe the diffusion of MPD elements in the solid assuming unidirectional diffusion, equilibrium is maintained at the interface, a constant diffusion
coefficient, constant solid-liquid interface area and static liquid region. The displacement of the interface can be estimated as:

\[ x = K_2 \sqrt{4D_s t} \]  \hspace{1cm} (2.7)

where \( K_2 \) is a constant related to the solidification characteristics and \( D_s \) is the diffusion coefficient of the MPD element in the solid BM. \( K_2 \) can be calculated by numerical methods, but basically accounts for the solubility of MPD elements in the solid. Using mass conservation and Fick’s second law assuming the density of the BM and FM are equal the time for complete isothermal solidification can be estimated as [45]:

\[ t_{iso} = \frac{\pi W_0^2}{16D_s} \left( \frac{c_f}{c_{al}} \right)^2 \]  \hspace{1cm} (2.8)

Despite the relative simplicity, these analytical solutions assume steady-state conditions for a binary system with a constant solute concentration. In reality, the dissolution and isothermal solidification process are transient due to the moving solid/liquid interface and changes in solute concentration during the process. In addition, most brazing systems are multi-component and the phases that form on solidification of the liquid FM are not captured in these diffusion equations.

To address these issues commercial software such as Thermo-Calc can be utilized. Thermo-Calc uses CALPHAD-based thermodynamic and kinetic databases that have been developed over the past few decades to model and design a wide range of alloy systems. [68, 69]. Using these databases and built-in algorithms the software can calculate various thermodynamic properties, stable/metastable phase equilibria, phase transformations and solve multi-component diffusion equations to ultimately accelerate
alloy design and improve understanding of existing alloys [70]. Small et al. provides an excellent example of how CALPHAD-based software has been used in the development of turbine disk alloys [71]. Similar modeling approaches have also been used to describe the microstructure evolution during joining of metals and alloys, including high temperature brazing of nickel-based superalloys [72, 73].

For brazing, numerical diffusion models to address the moving interface problem were developed by Ohsasa et al. and Ramirez et al. to calculate dissolution, isothermal solidification and homogenization during TLP bonding of Ni using Ni-P and Ni-B FMs [74, 75]. Ohsasa et al. combined thermodynamic calculations in Thermo-Calc with diffusion analysis by a finite difference method and provided a basis for calculating diffusion controlled transformations in what’s known as the DICTRA module in Thermo-Calc. Campbell et al. used Ohsasa’s model in DICTRA to model TLP bonding of a Ni-10Al alloy with a Ni-10B alloy [73]. Results shown in Figure 2.8 predicted variations in boron composition during isothermal solidification for various hold times. The movement of the S/L interface is coupled with the penetration of boron into the BM to maintain equilibrium at the interface. Boride formation in the DAZ found after TLP bonding was a result of this increased boron concentration. Schnell et al. used a similar DICTRA model based on the Ni-B binary system to estimate dissolution and isothermal solidification times in diffusion brazing of the SC superalloy CMSX-4 with a Ni-Cr,Co,Al,Ta,B braze alloy [45].
Figure 2.8: DICTRA model results from Campbell et al. used to predict boron diffusion and isothermal solidification during brazing of pure nickel BM with a Ni-10B FM.

To predict the phases that formed during solidification of the FM, Vargas et al. used Thermo-Calc to calculate the equilibrium phases of a BNi-2 FM composition and compared this to the composition and volume fraction of intermetallic phases identified in brazing with BNi-2 on a pure nickel substrate [42]. Arafin et al. performed similar equilibrium calculations and found good agreement between Thermo-Calc and experimental investigations for actual brazed joints of Inconel 718 and Inconel 625 with BNi-2 FM [76].

Based on these results, it appears that the combination of both thermodynamic and diffusion (kinetic) calculations using Thermo-Calc/DICTRA software could be utilized to estimate the final brazed joint microstructure and the total volume fraction of solid-solution present in the final joint. This in turn could be directly related to
mechanical properties using Eqn. 2.2. Additional studies to quantify the microstructure-property relationships would also be necessary to validate this approach.

2.6 Summary

This Chapter provided background on high temperature brazing of Ni-base superalloys for gas turbine applications. The basic principles of brazing including wetting, FMs, heating methods, metallurgy, and microstructure for these materials were covered. Ni-base FMs containing MPD elements produce three primary microstructural regions the ASZ (centerline eutectic), ISZ, and DAZ. The volume fraction of centerline eutectic has a strong influence on mechanical properties and should be evaluated when assessing brazed joint strength. Brazing parameters including FM composition, hold time, joint gap, and brazing temperature play a strong role on the volume fraction of centerline eutectic by affecting the extent of MPD diffusion during brazing. Analytical and numerical calculations have been developed to predict isothermal solidification and dissolution stages of the brazing process by modeling the diffusion of MPD elements. Simulation software such as Thermo-Calc can be utilized to perform these calculations and calculate equilibrium phases to ultimately predict the final joint microstructure based on the process parameters selected. Both thermodynamic and kinetic simulations should be incorporated in the multi-scale modeling approach.
CHAPTER 3

3. MECHANICAL PROPERTIES OF BRAZED JOINTS - BACKGROUND

3.1 Introduction

Mechanical properties of brazed joints are often inherently difficult to ascertain due to the narrow joints and complex microstructure produced in the brazing process. High constraint by the surrounding BM leads to a high tri-axial stress in brazed joint material and low strain to failure even for ductile FMs. As a result, brazed joints do not follow conventional material yielding behavior and failures appear brittle in nature. In addition, joint strength has been shown to be influenced by the joint gap, the degree of interaction between the BM and FM, presence of discontinuities or defects and the joint design [4]. These factors, particularly the degree of interaction between the FM/BM as shown in Chapter 2, will also depend on the brazing process parameters such as brazing temperature, hold time or the FM composition.

The American Welding Society C3 committee has recommended several standard methods for evaluating brazed joint strength in AWS C3.2 [77]. The two most commonly used methods from AWS C3.2 are the butt joint (tensile) and lap shear test shown in Figure 3.1 with the shaded area representing the brazed joint. These geometries are used to determine the average tensile and shear strength of the brazed joint respectively. This
Chapter will begin with a discussion of typical test results obtained from these standard test methods and their limitations.

![Diagram of butt and lap joints]

Figure 3.1: Butt (left) and lap shear (right) joint configurations are the two most commonly used for evaluating the strength of braze joints [78].

In most applications however, brazed joints are subjected to a combination of stresses such as shear, tensile and bending. Techniques to predict failure under combined loading conditions including failure assessment diagrams and finite element analysis (FEA) will also be covered in this Chapter. Finally, fitness-for-service assessments based on both fracture mechanics and joint strength have been proposed to predict failure loads for brazed joints containing flaws or defects similar to the API 579 or BS 7910 fitness-for-service assessments for welded joints [79, 80, 81]. While there are currently no standard methods for measuring fracture toughness of brazed joints this is an important material property for brazed joints due to the low strain to failure or brittle-like behavior and presence of defects. Therefore, a brief review of the FFS methodology and previous attempts to measure fracture toughness of brazed joints will be provided.

3.2 Butt Joint Tensile Specimen

Tensile testing is the most common method for determining material properties used in engineering analysis such as elastic modulus (E), yield strength (YS), ultimate
tensile strength (UTS), elongation and reduction in area. A comparison of stress/strain curves generated in tensile testing with bulk materials and with brazed joints is shown schematically in Figure 3.2 adopted from Flom et al. [82]. For homogeneous, ductile BMs like Ni-base superalloys (left in Figure 3.2) there will be an elastic and plastic portion to the curve from which material properties (E, YS, UTS, etc.) can be extracted from based on standard procedures such as ASTM E8 [83]. For example, yield strength can be determined using an extensometer to measure the stress at 0.2% strain offset. These material properties provide the basis for engineering and design of structural components and have been well established for common metals and alloys [9].

Figure 3.2: Uni-axial tensile stress-strain behavior of a homogeneous ductile BM (left) compared to brazed butt joint (right) [82].

For brazed joints these properties are not as well defined. When a brazed butt joint is located in the gauge section (right in Figure 3.2) the thin joint material is highly constrained or pinned by the surrounding BM resulting in a tri-axial stress state under...
tensile loading [84]. As a result, fracture in the braze joint occurs instead of necking and no significant plastic deformation occurs even if the FM is very ductile. Therefore, the yield strength and related material properties are not well defined by the stress/strain curve and the mechanical behavior is similar to a brittle material. In addition, since the FM interlayer cannot deform due to the BM constraint, the tri-axial stress state leads to higher joint strength than that of the bulk FM material. This is shown in Figure 3.3, for tensile tests of 304 stainless steel braze joints with a pure silver (Ag) FM. The tensile strength of the bulk pure silver FM was found to be approximately 18.5 ksi while the joint strength was greater than 35 ksi [48].

![Stress-strain curves of uni-axial tensile testing of 304SS BM and braze joints with pure Ag FM shows increase in joint strength from that of bulk Ag.](image)

Figure 3.3: Stress/strain curves of uni-axial tensile testing of 304SS BM and braze joints with pure Ag FM shows increase in joint strength from that of bulk Ag [48].

Most attempts to model brazed joint properties assume bulk FM properties can be applied to the joint material. However, as discussed in Chapter 2 metallurgical
interactions between the BM and FM can lead to quite different joint microstructures compared to bulk FM microstructure. As shown in Figure 3.4, a typical Ni-base superalloy braze joint microstructure with a BNi-2 FM contains DAZ, ISZ and ASZ regions centerline while the as-cast BNi-2 alloy produces a fine, uniform distribution of intermetallic phases. In addition, as previously discussed the volume fraction of these phases is highly dependent on the brazing process parameters and play a significant role on the tensile strength of the joint [85].

For example, Lugscheider et al. reported the tensile strength of butt joints for 316 SS brazed with BNi-2 and BNi-5 FMs significantly increased by 20-60 ksi with increasing hold time, brazing temperature and/or decreasing joint gap due to the reduction of centerline eutectic phases from the joint [50]. Similar results were reported by Lee et al. for butt joints of SUS304 with a BNi-2 FM who reported an increase in brazing temperature from 1010 to 1100°C resulted in an increase in tensile strength from approximately 430 (~62 ksi) to 500 MPa (~72.5 ksi) and an increase in brazing time from 10 to 120 minutes led to an increase from 350 (50.7 ksi) to 500 MPa (~72.5 ksi) [86]. Historically the joint gap has been shown to strongly influence brazed joint strength, with larger joint gaps resulting in lower tensile strength [4]. However, it is unclear whether this effect is due to a change in tri-axial stress as suggested by West et al. [87] or a result of increased brittle, intermetallic phases within the joint as suggested by Lugscheider et al. [50].

Differences between joint material properties and bulk properties are also observed in welding processes, typically to a lesser extent due to dilution and re-
solidification. To account for this in welding AWS D1.1 provides standard procedures for extracting tensile samples from weld metal [88]. The standard length for these weld metal test specimens is 2-4 inches. Braze joints are on the order of 0.002-0.004” in width and highly constrained, so obviously the AWS D1.1 approach cannot be applied. The ratio of the joint gap to the gauge length of a test specimen is often referred to as the joint aspect ratio. For standard size test specimens the joint aspect ratio is around 1:1000 or lower, so from a basic rule of mixtures methodology tensile tests will behave like the BM until failure occurs in the joint. Therefore, for all practical purposes the only useful property obtained from a butt joint tensile test is the ultimate tensile strength or fracture stress [89].

Figure 3.4: Microstructure of IN718/BNi-2 brazed joint (left) compared to the bulk BNi-2 FM microstructure in as-cast condition (right).

3.3 Lap Shear Test Method

The single lap shear test method was recommended in a comprehensive report by the American Welding Society (AWS) Committee on Brazing and Soldering in 1963 as a simple, relatively inexpensive and easily reproducible method for determining the
strength of brazed joints. This recommendation was based on a number of research papers published by Peaslee, Bredz and Miller with recommended geometries, manufacturing procedures and test results using the lap shear specimen for braze joint testing [90, 91, 92]. Lap joint configurations are often utilized in brazing because they offer the greatest possibility of obtaining joints with maximum efficiency and the greatest ease of fabrication [4]. The standard method developed and still used today involves brazing several lap joints at different overlaps and recording the breaking load and failure location. From the breaking load, the average shear stress in the FM and tensile stress in the BM can be calculated by the following equations [77]:

\[
\text{Avg. FM shear stress (FM)} = \frac{\text{Breaking Load}}{A \times W} \tag{3.1}
\]

\[
\text{Avg. BM tensile stress} = \frac{\text{Breaking Load}}{W \times T} \tag{3.2}
\]

where \(A\) is the brazed joint overlap length, \(W\) is the width of the BM and \(T\) is the thickness of the BM as shown in Figure 3.5.

![Diagram of Single Lap Shear Test Specimen Geometry](image-url)

Figure 3.5: Single lap shear test specimen geometry as defined in AWS C3.2 [77].
In general, as the overlap distance or braze joint area increases the breaking load also increases and if the overlap is large enough failure will occur in the BM rather than through the FM. Figure 3.6A, from AWS C3.2 shows typical results and illustrations of types of failures for lap shear tests [77]. Line 1 represents the average shear stress in the FM which decreases exponentially with increasing overlap distance. Line 2 gives the average BM tensile stress which increases linearly with increasing breaking load until failure in the BM occurs. Open data points indicate failure in the joint and filled points indicate failure occurred in the BM. Figure 3.6B, shows an example of actual AWS single lap shear test results taken from Lugschieder et al. for 316SS brazed joints with BNi-5 FM [93]. As shown, lap shear results give some indication as to what overlap is required to achieve failure in the BM, for Lugschieder’s results this overlap was found to be approximately 4T. However, similar to butt joint testing the lap shear strength will also depend on the extent of MPD diffusion that occurs during the process as demonstrated by Miyazawa et al. [94].
Figure 3.6: Schematic of typical lap shear tensile test results from AWS C3.2 [77] (A) and actual lap shear results for 316 SS brazed with BNi-5 FM for overlaps ranging from 0.5 to 6T (B) [93].

The primary issue with the lap shear test method is that the calculated FM shear stress is not constant and decreases exponentially with increasing overlap. For the 316SS/BNi-5 joints an average FM shear stress of about 55 ksi was calculated for 0.5T overlaps, but at 6T overlaps the FM shear stress was approximately 10 ksi. This creates an obvious problem from a design standpoint and makes it difficult to recommend a safe stress level. This occurs because the shear stress values calculated using Eqn.1 are average values assuming the shear stress is uniformly distributed across the joint, however it has been well established that this is not the case due to the peel stresses and stress concentrations that develop during shear-tensile loading [95].

Several research groups have attempted to address the issues with the single lap joint geometry in an effort to predict the failure load independent of the test specimen.
geometry (overlap) [90, 96, 97]. Lugscheider et al. used a von Mises criteria to calculate an equivalent stress based on the twisting angle at various overlaps for 316 SS brazed with BNi-2 FM [96]. Broughton et al. used finite element analysis (FEA) to calculate peel and shear strain for adhesive lap joints with various BMs [95]. Similarly, Flom et al. performed FEA to calculate the von Mises stress distribution across a range of overlaps for 347SS brazed joints with pure Ag FM [97]. Results from these studies indicate stress concentrations occur at the edge of the joints due to the inherent bending of the lap shear test specimen and change in cross-sectional area in this location. This effect is amplified for larger overlaps to the point where the central portion of the joint carries little to no load, thereby reducing the effective area in Eqn. 1. This was shown in a study by Flom et al. in which flaws introduced in the center of 5T overlap samples had little effect on the breaking load [98]. Based on these results, Flom et al. recommended 0.5T overlaps should be used to define the critical shear strength of the braze joint since the stress distribution is most uniform across the joint and suggested the use of a damage zone model for predicting failure loads at large overlaps.

3.3.1 Damage Zone Model

Damage zone models have been used to predict failure of adhesively bonded lap joints, but have not been widely implemented for brazed joints [99, 100, 101]. The basis for damage zone failure criteria is to evaluate the stress distribution surrounding a stress concentration and determine if the stress exceeds some sort of failure criteria such as von Mises yield criteria or maximum principal stress or strain. Any location that exceeds this stress or strain level is considered to be part of the damage zone and if the damage zone
reaches a critical size failure will occur. For adhesive and braze joints, in which the
failure occurs along the path of the joint, the size of the critical damage zone can be
considered as a length rather than an area in 2D.

Flom et al. introduced this concept for lap shear brazed joints using a von Mises
failure criteria in Figure 3.7. Based on FEA models of experimental lap shear testing at
various overlaps the calculated von Mises stress at the failure load was plotted across the
normalized overlap distance [97]. As shown, the calculated stresses at failure are very
high at the edge of the joints especially at large overlaps, and low at the center of the
joint. However, at a certain distance (X=0.05) from the edge of the overlap the stress
values appear to be about the same around 62 ksi (427 MPa). Therefore, this distance or
10% of the total overlap length was considered to be the critical damage zone length
assuming a threshold von Mises stress of 62 ksi (427 MPa). The FEA estimate based on a
10% damage zone failure criterion was also shown to give good agreement with the
classic work of Bredz and Miller who reported lap shear test results for a similar BM/FM
combination as was used by Flom et al. [91].
While the good agreement between the FEA models and lap shear test results using the damage zone model is encouraging, there have not been any attempts to experimentally measure or observe the actual damage zone during testing of lap shear brazed joints. In addition, FEA models require accurate FM material properties that are not readily available. For example, the approach used by Flom et al. to define pure Ag FM properties by conventional tensile testing may not be applicable for Ni-based FMs that have strong metallurgical interactions with the BM and produce significantly different joint microstructures from the bulk FM.

### 3.4 Alternative Test Methods

Due to the issues with single lap shear and butt joint test specimens, a number of other test methods have also been used to evaluate the strength of braze joints. Since it is difficult to avoid stress concentrations at joints, most of the “improved” specimen designs
are aimed at eliminating or minimizing specimen bending. AWS C3.2 includes a pin-loaded double-lap shear test specimen to minimize the bending moment of the single lap shear. Similarly Datta et al. used a support fixture to minimize the bending in single lap shear tests of Cu-P brazing foils. Spingarn et al. compared a pin-collar specimen with lap shear specimens (0.3T-2T overlaps) for A296 stainless steel braze joints with BAu-4 and BNI-2 FMs [102]. Test results showed good agreement between test methods for the average shear strength for a ductile BAu-4 FM, but not for the brittle acting BNI-2 FM.

Klausnitzer et al. and Shawki et al. utilized a punch and die method to measure the shear strength of stainless steel braze joints with several different high temperature FMs [103, 104]. The punch and die method allowed better control of the brazing gap without a fixture, easy testing at high temperatures and could rank the relative ductility of the braze joints by using a series of round dies at different diameter. Klausnitzer’s reported high shear strengths ranging from 65-75 ksi for the tested brazed joints with narrow joints gaps of 15 \( \mu \text{m} \). While these types of test methods may eliminate some of the issues with the standard butt or lap joint specimens published results using these geometries are scarce, the stress concentrations at the joint are not completely eliminated and in some cases the geometries are more difficult or expensive to manufacture.

3.5 Failure Assessment of Brazed Joints

In most applications brazed joints are under a combination of shear and tensile loading. Numerous failure criteria have been developed to predict failure of materials under complex loads including Tresca or von Mises yield criteria, maximum stress or maximum strain failure criterion, damage zone models (as previously discussed) and
fracture mechanics [105]. However, none of these have been well established to predict failure of brazed joints. Due to the tri-axial stress and constraint of the BM on the FM, conventional yielding failure criteria such as Tresca or von Mises yield criteria are difficult to apply since the joint material is under hydro-static pressure and doesn’t follow a ductile failure mechanism.

Flom et al. proposed a modified Coulomb-Mohr expression based on shear and tensile allowables measured from uni-axial butt joint tensile tests and lap shear testing to predict the load carrying capacity and provide margins of safety for brazed joints under combined loading conditions [106]. This approach involves creating a failure assessment diagram to determine if a brazed joint was ‘safe’ or ‘un-safe’ based on a conservative interaction equation between the shear and tensile stress ratios:

\[ R_\sigma + R_\tau = 1 \] (3.3)

Where \( R_\sigma \) is equal to the nominal tensile stress at a given location divided by the material allowable tensile strength and \( R_\tau \) is equal to the nominal shear stress divided by the material allowable shear strength. Based on this methodology if the sum of the stress ratios was kept below a value of one failure would not occur and the joint would be deemed ‘safe’ for operation. Flom et al. used this approach to generate a failure assessment diagrams for Ti-brazed joints and showed this interaction equation gives a conservative estimate of the safe static load carrying capacity for brazed T-joints [107].

Ghovanlou et al. proposed a similar stress based power law failure criterion for predicting biaxial loading in low carbon steel brazed joints with a copper FM where [108]:

\[ (R_\sigma)^\alpha + (R_\tau)^\alpha = 1 \] (3.4)
In this case, $\alpha$ was determined from experimental testing to be 1.85 from tensile/torsion tests for these brazed joints. This approach was less conservative than the one proposed by Flom et al. and showed good agreement with experimental results. However, both of these approaches assume a constant shear stress material allowable which has been shown to strongly depend on the overlap. Also, as shown by Flom et al. for lap joints the calculated stress at or near stress concentrations may greatly exceed the material allowable without causing joint failure [84]. In addition, these approaches assume defect free joints and joints containing flaws or defects such as lack of braze resulted in failures inside the defined safe region.

3.6 Fracture Mechanics

Fracture mechanics is another type of failure theory used to predict failure of materials containing pre-existing flaws and to characterize a material’s resistance to fracture. The basic concept is to compare the local stress distribution near the edge of the flaw or crack tip to critical values for the material. For example, the calculated stress intensity factor ($K_I$) or $J$ integral ($J$) are compared to the material properties of the critical stress intensity factor or fracture toughness ($K_{IC}$) and critical $J$ integral ($J_C$) to determine if the crack will propagate. In general, the stress intensity factor ($K_I$) for a crack of length ($a$) can be calculated from the applied stress ($\sigma_{nom}$) and a geometrical parameter $f(a/W)$ with units of ksi$\sqrt{\text{in}}$ (Pa$\sqrt{\text{m}}$) as [109]:

$$K_l = \sigma_{nom} \sqrt{\pi a} * f \left( \frac{a}{W} \right)$$  \hspace{1cm} (3.5)

In brazing, cracks are inherently found within the joints due to defects such as pores or lack of braze and may also form during fatigue or static loading in the hard, brittle
centerline eutectic regions, leading to crack growth and possible catastrophic failures. Figure 3.8 shows one such defect that formed as a result of incomplete gap filling in a steel brazed joint with Ag-based FM [79].

Figure 3.8: SEM image of void in a steel brazed joint with Ag-based FM that formed as a result of incomplete gap filling from Leinenbach et al. [79].

3.6.1 Fitness-For-Service Assessment

For homogenous materials, welded structures, composites, and adhesive joints, fracture mechanics has been found to yield the most consistent results and can account for geometrical variations and the presence of flaws and stress singularities [109]. As a result, this failure criteria has been widely implemented in industry to determine the structural integrity of components in-service that may contain a flaw or damage via fitness-for-service (FFS) assessments. The FFS approach was first defined by Irwin [110] and Wells [111] in the early 1960s and was introduced in the welding industry by the British Standards Institute in 1980 in the PD 6493 – Guidance and Methods for Assessing the Acceptability of Flaws in Fusion Welded Structures [112]. FFS assessments are now widely accepted by the API and ASME in the analysis of bulk materials and welded
structures containing discontinuities [80]. In a FFS assessment, failure assessment diagrams (FAD) similar to those described in the previous section are generated using material allowables to calculate stress ($S_r$) and fracture ($K_r$) ratios at a given load. These ratios are defined as:

$$S_r = \frac{\sigma_{nom}}{\sigma_{flow}}$$  \hspace{1cm} (3.6)$$

and

$$K_r = \frac{K_I}{K_{IC}}$$  \hspace{1cm} (3.7)$$

$\sigma_{nom} = \text{applied stress}$

$\sigma_{flow} = \frac{\sigma_{YS} + \sigma_{UTS}}{2} = \text{flow stress}$

$K_I = \text{calculated stress intensity factor}$

$K_{IC} = \text{critical stress intensity factor}$

Figure 3.9 shows a FAD for a Level 1 assessment according to BS 7910 where $\sigma_{flow}$ is assumed to be approximately $1.2\sigma_{YS}$ giving a $S_r$ of approximately 0.8 and a safety factor of 2 is applied for the maximum crack size resulting in a $K_r$ of approximately 0.7. According to this methodology if a material is operating with stress and fracture ratios below these values failure will not occur and the loading conditions are acceptable. If these values are exceeded the material is likely to fail and is unacceptable for service [113]. Level 2 and Level 3 assessments are more sophisticated approaches based on relationships between these two ratios such as the strip-yield model to predict their combined effects. These assessments incorporate FEA modeling to calculate $S_r$ and $K_r$ for actual defects observed during inspection of materials to account for their size and location in the material. Ultimately, FFS assessments provide a tool for determining...
maximum allowable flaw size, repair needs and life predictions. As a result, it would be useful to implement the same methodology that has been used successful in welding for brazed joints.

Figure 3.9: An example of a Level 1 failure assessment diagram (FAD) used in fitness-for-service assessments as per BS 7910 [81].

### 3.6.2 Fracture Mechanics Test Methods for Brazed Joints

The use of FFS assessments in industry has led to the development of standard fracture mechanics test methods to assess the critical stress intensity factor for bulk and welded metals like the compact tension specimen listed in ASTM E399 or single-edge bend test in ASTM 1820 [114, 115]. Standardized methods have also been developed for adhesive bonding such as the double cantilever beam listed in ASTM D3433 [114]. However, no such standards or FFS codes currently exist for brazed joints and only a few research groups have published results in this area. A summary of the publications found
in the technical literature evaluating the fracture toughness of brazed joints is provided in Table 3.1 listing the authors, materials, test method and calculated $K_{IC}$.

Table 3.1: List of publications with fracture mechanics methods and measured $K_{IC}$ values for brazed joints

<table>
<thead>
<tr>
<th>#</th>
<th>Author</th>
<th>BM</th>
<th>FM</th>
<th>Method</th>
<th>$K_{IC}$ (ksi*in$^{1/2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Leinenbach et al.</td>
<td>Martensitic stainless steel (X3CrNiMo13-4)</td>
<td>Au-18Ni</td>
<td>Double Cantilever Beam (DCB)</td>
<td>43.0 - 46.1</td>
</tr>
<tr>
<td>2</td>
<td>Weiss et al.</td>
<td>Inconel 718 (full HT)</td>
<td>BNi-5</td>
<td>Single Edge Notched Bend (SENB)</td>
<td>21.1 - 28.2</td>
</tr>
<tr>
<td>3</td>
<td>Moorhead et al.</td>
<td>Alumina, PSZ (ceramics), and Ti</td>
<td>Cu-44Ag-4Sn-4Ti</td>
<td>Modified DCB</td>
<td>2.5 - 10.7</td>
</tr>
<tr>
<td>4</td>
<td>Ghovanlou et al.</td>
<td>ASTM A36</td>
<td>Copper (BCu-1)</td>
<td>SENB (four point bend)</td>
<td>19.74</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Copper (BCu-1)</td>
<td>Single Edge Notched Tension (SENT)</td>
<td>42.32</td>
</tr>
<tr>
<td>5</td>
<td>Gan et al.</td>
<td>SS 304 and V-5Ti-5Cr</td>
<td>AuNi</td>
<td>SENT (V-Notch)</td>
<td>18.20</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>MBF80 (BNi-9)</td>
<td></td>
<td>1.55</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>AuNiPd</td>
<td></td>
<td>1.91</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Cu</td>
<td></td>
<td>10.01</td>
</tr>
</tbody>
</table>

As shown, a variety of test methods have been used all of which have been designed to test Mode I fracture with the braze joint located at the tip of a manufactured notch. Philips et al. used a wedge specimen to test Ni-P FMs with 304 stainless steel [116]. Moorhead et al. developed an applied moment double cantilever beam specimen to determine the fracture toughness of brazed joints with ceramic materials [117]. Gan et al, used a V-notch butt joint sample to evaluate the toughness of a vanadium alloy brazed to 304 stainless steel with several different FMs [118]. The notch specimens used in this study, shown in Figure 3.10, were reportedly easy to manufacture and compare to standard butt samples. An equation for $K_{IC}$ was provided for the V-notch configuration based on the peak tensile load and sample geometry. Gan et al. reported $K_{IC}$ values of 1.55, 10.01 and 18.20 ksi$\sqrt{\text{in}}$ for the MBF80 (BNi-9), Cu and AuNi FMs respectively.
The lower $K_{IC}$ value for the Ni-Cr-B FM was attributed to the presence of brittle intermetallic phases in the joint that were not observed in the AuNi and Cu joints. $K_{IC}$ of the AuNi joint (20 MPa√m) was found to be about one-fifth that of the vanadium BM (110 MPa√m) indicating even ductile FMs have limited fracture toughness. Weiss et al. tested a BNi-5 FM with Inconel 718 and reported significantly higher $K_{IC}$ values of 21-28 ksi√in for the nickel-based FM compared to Gan et al. indicating the sample geometry or BM may influence $K_{IC}$.

Figure 3.10: SS304/Au-Ni/V-5Ti-5Cr brazed joint V-Notch samples used for fracture toughness assessment and equation for $K_{IC}$ provided by Gan et al [118].

The work of both Ghovanlou et al. and Leinenbach et al. were the only two studies found in the literature that followed a FFS approach for brazed joints in which both fracture mechanics testing and mechanical testing (tensile and tensile/torsion) were used to generate FADs based on stress and fracture ratios. Leinenbach et al. utilized a modified double cantilever beam (DCB) test specimen shown in Figure 3.11 to calculate
$K_{IC}$ for soft martensitic stainless steel brazed joints and standard butt joints were used to
determine ultimate tensile strength allowable for the joints [79]. Good agreement was
found between the generated FADs and the experimental results for different samples
containing defects of various size. Ghovanlou et al. found similar agreement using the
FFS methodology for A36 brazed joints with BCu-1 FM and indicated that the sample
geometry played a strong role on the calculated $K_{IC}$ values as shown in Table 3.1. Using a
single edge notched tensile specimen similar to the V-Notch specimen gave a higher
average $K_{IC}$ value of 42.3 ksi√in compared to the single-edge notch bend test specimen
which gave a value of 19.7 ksi√in [119].

Figure 3.11: Double cantilever beam (DCB) specimen with dimensions used by
Leinenbach et al. for fracture mechanics testing of brazed joints [79].

As indicated by both Leinenbach et al. and Ghovanlou et al. the FFS methodology
can be very effective in predicting brazed joint failures for joints containing flaws or
defects. However, there is still a lack of data and more importantly standard procedures that include geometry, brazing, pre-cracking and testing considerations for brazed joints.

### 3.7 Finite Element Modeling of Brazed Joints

As previously mentioned, finite element analysis (FEA) has been used to model the stress and strain distribution in brazed joints and is also incorporated in FFS assessments of weldments to more accurately estimate $S_t$ and $K_r$. FEA is also used widely as a numerical tool for engineering analysis of mechanical systems to estimate stress, strain, and displacements in a component based on prescribed boundary conditions.

However, there have been relatively few attempts to apply FEA for brazed joints in the technical literature. For example, a bibliography of FEA and simulation for adhesive bonding, soldering and brazing publications from 1976-1996 indicated less than 24 papers were published during this time [120]. In addition, the primary focus in most FEA modeling work for brazed joints has been measuring residual or thermal stress between dissimilar BMs rather than structural analysis or fracture mechanics. As a result, design engineers do not typically account for brazed joints in FEA models of gas turbine components and instead assume BM properties across the joint with various safety factors applied based on the experience and recommendation of the brazing engineer. This creates obvious concerns from a design and reliability standpoint.

According to Flom et al. there are two primary ways to analyze brazed joints using FEA [121]. The first approach is to use FEA of the entire structure as a whole in which the brazed joint is simply a line with the same material properties as the BM. The distribution of stress in the braze plane is determined and the maximum value of shear
and tensile stresses are identified and compared to maximum shear/tensile stresses measured from lap or butt joint testing. The second approach is a more detailed FEA model of the brazed joint that accounts for the joint gap and material properties of the FM. This requires knowledge of the brazed joint elastic modulus, yield strength and stress/strain behavior which can be challenging to determine as previously discussed. In most cases, the bulk FM properties are used for FEA modeling for example in the lap shear models developed by Flom et al. to calculate the Mises stress distribution for 304SS with a pure Ag FM [89]. Ghovanlou et al. used a similar approach to define a BCu-1 FM properties for FEA calculations of the equivalent Mises stress in copper brazed joints with two different steel BMs [122]. Results from Ghovanlou et al. shown in Figure 3.12 indicated the highest Mises stress occurs at the FM/BM interface and is higher for BM with higher yield strength. Ghovanlou et al. also investigated the use of a cohesive zone model based on the energy release rate to predict fatigue failure using FEA models [123]. Leinenbach et al. used in-situ DIC via scanning electron microscopy to estimate the local stress/strain behavior of an Au-18Ni FM for FEA models to calculate the maximum stress level in soft, martensitic stainless steel brazed joints containing defects [79]. This was a unique approach because the stress/strain behavior of the actual joint material rather than the bulk FM was measured and applied to the FEA models.
Figure 3.12: FEA models of A36 and A108 steel brazed joints with Cu FM [108].

In general, current state-of-the-art FEA models of brazed joints assume perfect joints without defects and typically requires a refined mesh in the joint regions particularly in regions of stress concentration. FE modeling of joints with pure Ag, Au or Cu FMs is relatively easy as the elastic/plastic properties of the pure metal can be assigned to the homogeneous joint regions. When brazing with Ni-base FMs however, these properties are not easily identified due to the complex, multi-component microstructures that form within the joint as previously discussed. To model these eutectic forming FMs, the joint region is assumed to be a homogeneous material with average properties selected at the discretion of the engineer [124]. No attempts to incorporate the properties of more than one phase brazed joint region was found in the literature.
3.8 Summary

This Chapter introduced the inherent challenges in determining mechanical behavior of brazed joints and predicting brazed joint failures. The butt joint and lap shear test methods are most commonly used to evaluate joint strength. However stress/strain curves cannot be readily assessed with butt joints due to the tri-axial stress and low strain to failure. For the lap shear geometry, stress concentrations at the edge of the overlap due to peel stresses and reduction of area lead to inconsistent shear stress values depending on the overlap used. Despite the issues with these test methods, both are still used by industry to determine the maximum brazed joint allowable shear and tensile stress.

Several methods for predicting joint failure were introduced including failure assessment diagrams based on the shear/tensile allowables using modified Coulomb-Mohr or power law interaction equations to predict safe or unsafe stress levels for operation. However, these types of failure assessments do not consider the presence of defects or flaws which have been shown to dramatically decrease the load carrying capacity of brazed joints. Instead fitness-for-service assessments similar to those used for welded structures have been proposed to assess safe levels of operation for defect-containing brazed joints. The FFS assessment incorporates both stress ratios ($S_r$) and fracture toughness ratios ($K_r$) based on fracture mechanics testing and have shown good agreement with experimental results. Several test methods for measuring the critical fracture toughness ($K_{IC}$) of brazed joints have been discussed although there is currently no standard available like ASTM E399 for bulk materials. If a test method can be established, the FFS assessment of brazed joints may provide the best approach for...
predicting brazed joint failure because it can account for the stress level at the joint and the presence of defects. Such methodology will likely include FEA modelling (similar to Level 2 and Level 3 assessments in API 579) to calculate the maximum stress and critical stress intensity factor in the brazed joints.
CHAPTER 4

4. RESEARCH OBJECTIVES

Based on the review of the technical literature provided in Chapters 2 and 3 it is clear that predicting brazed joint mechanical properties is challenging and depends on a number of factors including the process parameters, joint design, presence of defects, and BM/FM combinations. There is a need to incorporate these factors in a multi-scale computational model for prediction of brazed joint mechanical properties to improve the design and ensure safety requirements of brazed joints in gas turbine components. Prior work to establish design guidelines for brazed joints has included the development of failure assessment diagrams and fitness-for-service assessments. However, test methods for these types of assessments need to be further developed for brazed joints and the influence of process parameters should be considered in the analysis.

Therefore, the following multi-scale modeling approach, shown schematically in Figure 4.1, is proposed for prediction of brazed joint properties. This approach is similar to the multi-scale computational model introduced by Tong et al. for fusion joining [125]. First, experimental brazing will be performed to characterize the micro-constituent phases that form within the joint and to examine the influence of primary process parameters (joint gap, hold time, brazing temperature and FM/BM composition) on the formation of these phases. Thermodynamic and diffusion simulations using Thermo-Calc
software will then be used to model the observed metallurgical interactions and predict joint microstructure. Multi-scale mechanical testing of brazed joints will be performed to develop microstructure-property relationships, including both strength and fracture toughness to account for the presence of defects. Finally, FEA models will be calibrated based on the experimental results and used to predict the overall performance or fitness-for-service of the brazed component.

Ultimately, we envision a fully coupled model in which the Thermo-Calc simulations and microstructure predictions can be directly used to adjust the material property inputs for FEA models for a range of brazed joints and processing parameters. Such a tool would significantly reduce the time and cost associated with development of brazed joints and improve analysis of existing joints. The overall objective for this initial investigation was to generate data and develop methodology to support such a model for conventional nickel-based brazing FMs used in gas turbine applications. This includes development of experimental techniques and procedures for metallurgical characterization, thermodynamic simulations, mechanical testing and FEA modeling of brazed joints. Results will be used to provide a framework for future multi-scale models that can be applied to a wide range of brazed joints and process parameters.
Figure 4.1: Schematic outline of proposed multi-scale computational model for brazed joints.

**Research Tasks**

The following research tasks were proposed to develop data and methodology for the components of the multi-scale model and validate the overall modeling approach:

1. Perform microstructural characterizations of brazed joints with selected Ni-base superalloys and FMs to determine the properties, dispersion parameters, and composition of joint micro-constituents.

2. Conduct brazing experiments to evaluate the influence of process parameters (hold time, joint gap, temperature, and BM/FM composition) on the joint microstructure.
3. Generate thermodynamic and kinetic simulations with Thermo-Calc and DICTRA software to predict joint microstructure based on process parameters studied in Task 2.

4. Perform multi-scale mechanical testing of Ni-base superalloy brazed joints to develop microstructure-property relationships, determine maximum lap shear and tensile stresses and FEA modeling techniques based on brazed joint mechanical behavior.

5. Evaluate fracture mechanics test methods for measuring the fracture toughness (KIC) of brazed joints for fitness-for-service assessments.
CHAPTER 5

5. METALLURGICAL CHARACTERIZATION

5.1 Introduction

This Chapter contains an introduction to the materials and vacuum furnace brazing procedures used throughout this study as well as the experimental techniques and results from metallurgical characterizations of Ni-base superalloy brazed joints. Brazed joint test coupons were analyzed using conventional metallographic techniques such as light optical microscopy (LOM), scanning electron microscopy (SEM) and micro-hardness mapping along with more unconventional techniques such as low angle micro-sectioning (LAMS), ImageJ analysis, and electron probe micro-analysis (EPMA) to determine the properties, composition and dispersion parameters of joint micro-constituents. This data is important to establish microstructure-property relationships. Based on the literature findings in Chapter 2 additional brazing experiments were designed and carried out to examine the effect of the primary brazing process parameters on joint microstructure.

5.2 Materials

The chemical composition (wt%) of the BMs and FMs used in this study are listed in Table 5.1. Two nickel-based superalloy, Alloy CMSX-4 and Inconel 718, were selected as the BMs for this study based on their use in the gas turbine industry. Alloy
CMSX-4 is a second generation, rhenium containing single crystal (SX) material primarily strengthened by γ’ (Ni₃(Al,Ti)) precipitation and exhibits excellent creep-rupture, fatigue, and oxidation properties [126]. Inconel 718 (IN718) is a widely used high strength, corrosion resistant poly-crystalline superalloy primarily strengthened by γ” (Ni₃Nb) [127]. IN718 is well known for improved weldability and resistance to strain age cracking due to the sluggish γ” precipitation reaction [26]. SX alloys like CMSX-4 are primarily used for high temperature blade applications while IN718 is a workhorse for many components with slightly lower operating temperatures such as vanes and nozzles. Rolled IN718 plates of 0.25 and 0.5 inch thickness were acquired from Manzi Metals Inc and as-cast CMSX-4 material was provided by Rolls-Royce Corporation for this study.

The two FMs, BNi-2 and BNi-9, were selected for investigation based on suggestions from the industry sponsor. BNi-2 is a Ni-Cr-B-Si-Fe based FM that is widely considered to be the work-horse brazing alloy for a variety of Ni-base and stainless steels with a solidus/liquidus temperature of 971°C and 999°C respectively. BNi-9 is a Ni-Cr-B containing FM that is often used for improved corrosion resistance and high strength applications with liquidus/solidus temperatures of 1020°C and 1055°C [128]. Metallurgical characterization was performed on brazed joints with both FMs, however only BNi-2 was selected for mechanical testing to reduce time and cost. As a result, BNi-2 was the main focus in the brazing studies to examine the effect of process parameters.
Table 5.1. Chemical composition of BMs and FMs investigated in this study.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Composition (wt%)</th>
<th>Ni-base Superalloy BM</th>
<th>others</th>
</tr>
</thead>
<tbody>
<tr>
<td>CMSX-4</td>
<td>balance 6.4</td>
<td>Ni 6.4 Cr 2.9 Re 9.6</td>
<td>Ni 0.05 Zr, 0.1 Hf, 0.042 Fe, 0.0037 C</td>
</tr>
<tr>
<td>Inconel 718</td>
<td>balance 19</td>
<td>Ni 19 Cr 3.1</td>
<td>Ni 0.30 Cu, 0.08 C</td>
</tr>
<tr>
<td>Brazing FM</td>
<td></td>
<td>Ni-7Cr-3.1B-4.5Si-3Fe</td>
<td>Liquidus/Solidus: 971°C/999°C</td>
</tr>
<tr>
<td>BNi-2</td>
<td>Ni-15Cr-3.5B</td>
<td></td>
<td>Liquidus/Solidus: 1020°C/1055°C</td>
</tr>
<tr>
<td>BNi-9</td>
<td>Ni-15Cr-3.5B</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

5.3 Experimental Techniques and Procedures

For initial characterization, three brazing coupons were prepared for each joint using two CMSX-4 plates (2.5 cm x 1.5 cm x 0.3 cm) with a Nicro-blast surface condition and laser tack welding for a joint gap of 80-100 μm. BNi-2 and BNi-9 dry FM powder supplied by Rolls-Royce was mixed with Vitta Braz-Binder Gel: Grade ST in a 9:1 powder to binder ratio by weight to create FM paste. The FM paste was then applied to the joint area and fillet regions. Two brazing cycles were performed in a batch-type vacuum furnace at Rolls Royce facilities under high vacuum (<10^-6 torr) with the heating steps listed in Table 5.2 for each FM. A relatively short holding time of 10 minutes at brazing temperatures (Tb) of 1066°C and 1121°C was used for BNi-2 and BNi-9 joints, correspondingly.
Table 5.2: Standard brazing cycles for CMSX-4 brazed joints with BNi-2 and BNi-9 FMs.

<table>
<thead>
<tr>
<th>Standard Brazing Cycles</th>
<th>BNi-2 (AMS-4777)</th>
<th>BNi-9 (Amdry 775)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ramp (F/min)</td>
<td>T&lt;sub&gt;b&lt;/sub&gt; (°F)</td>
<td>T&lt;sub&gt;b&lt;/sub&gt; (°C)</td>
</tr>
<tr>
<td>20</td>
<td>1000</td>
<td>538</td>
</tr>
<tr>
<td>30</td>
<td>1650</td>
<td>899</td>
</tr>
<tr>
<td>30</td>
<td>1950</td>
<td>1066</td>
</tr>
<tr>
<td>Vacuum Cool</td>
<td>1000</td>
<td>538</td>
</tr>
<tr>
<td>Gas fan cool</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Brazed samples were sectioned both normal to the braze joints and using a low angle micro-sectioning (LAMS) technique originally developed at OSU for observing narrow regions at weld interfaces [129]. Figure 5.1 illustrates how LAMS increases the width of visible joint from 0.1 mm to 2.5 mm for better revealing the joint microstructure in a CMSX-4/BNi-2 joint sample. The sectioning angle (θ) was calculated to relate the width of LAMS joint gap (W<sub>LAMS</sub>) to the width of the normal joint gap (W<sub>normal</sub>) as follows:

\[
\theta = \sin^{-1} \frac{W_{normal}}{W_{LAMS}} \tag{5.1}
\]

For LAMS samples, θ typically ranged from 1-5°. After sectioning, samples were polished using standard metallography techniques to 1 µm and metallurgical images were taken using an Olympus GX51 LOM and a FEI/Philips XL-30 Field Emission ESEM at an acceleration voltage of 20 kV. Average volume fraction, particle size and particle spacing of centreline eutectic phases were determined using threshold and particle analysis tools in the image processing software ImageJ.
Compositional analyses of the CMSX-4/BNi-2 and CMSX-4/BNi-9 joint samples were initially performed with energy-dispersive spectroscopy (EDS) scans in the SEM. Detecting boron, a lighter element, in nickel braze joints with SEM/EDS has always been technically very difficult. As a result, two LAMS samples for each joint were further analysed using a JEOL JXA-8200 electron microprobe at an accelerating voltage of 10 kV and 10 µm beam diameter to measure boron content more accurately. EPMA point scans, line scans, and X-ray intensity maps across the braze joints were used to determine elemental distribution and phase composition. Micro-hardness indents were made using an automated LECO LM 100AT micro-hardness tester across the BNi-2 LAMS sample and BNi-9 fillet regions with a 100 gram force. Average Vickers Hardness values were calculated for each phase and IgorPro software was used to create contour plots of the results.
5.4 Characterization of CMSX-4 Braze Joints

5.4.1 Joint Microstructure

Brazing of CMSX-4 with both FMs produced excellent joints with no visible porosity and good fillets. Figure 5.2 and Figure 5.3 show the joint microstructure after brazing for the BNi-2 and BNi-9 FMs respectively with LAMS and normal sectioning applied. Isothermally solidified zone and athermally solidified zones (labelled) are easily identified with LAMS. The ISZ appears as a white, single phase ($\alpha$-Ni phase) approximately 20 $\mu$m in width on either side of the joint. As previously discussed, this region forms during the initial BM dissolution due to short-range boron diffusion which has been shown to occur within one minute or two once the FM has completely melted [41]. Ramirez et al. estimated BM dissolution is complete in less than 10 seconds [75], so isothermal solidification is expected to begin during the 10 minute hold time due to MPD (boron) diffusion into the CMSX-4 BM. However, as previously discussed isothermal solidification is limited with conventional brazing times compared to TLP bonding or diffusion brazing. As a result, on cooling the remaining liquid FM enriched in boron forms the ASZ centreline eutectic during normal solidification.
Figure 5.2: Joint microstructure of CMSX-4 vacuum furnace brazed with BNi-2 FM imaged via LOM at 20X for LAMS (left) and 50X for normal sectioned samples (right).

The BNi-2 FM produced a multi-component eutectic containing two primary phases that appear as a grey and dark black phase, these were identified as Ni-rich and Cr-rich phases by EDS point scans. A two phase eutectic surrounding these bulky phases consisted of a silicon-rich (light brown) phase and a nickel-rich phase (grey). Brazing with the BNi-9 FM produced only one Cr-rich phase within a two phase (Cr-rich and α-Ni phases) eutectic matrix as shown in Figure 5.3. The morphology of this phase in the BNi-9 joint changed from a thin platelet shape in the normal sectioned sample to a bulky, irregular shape in LAMS indicating that these constituents are oriented parallel to the joint interface and occupy a larger volume of the joint than is apparent with normal sectioning.
Figure 5.3: LOM images of CMSX-4/BNi-9 brazed joint microstructures after LAMS and normal sectioning.

### 5.4.2 Compositional Analysis

The distribution of FM elements for the CMSX-4/BNi-2 brazed joint is shown in Figure 5.4 from X-ray intensity mapping across the LAMS sample. In these plots, light regions show high concentrations and darker regions indicate low concentrations of FM elements. For a more quantitative analysis, Figure 5.5 shows the results of the EPMA line scans across the CMSX-4/BNi-2 (blue line) and CMSX-4/BNi-9 (red line) brazed joints for each element detected. Due to symmetry only half of the joints are shown with the micrographs at the top indicating the location of the line scan starting in the unaffected CMSX-4 and moving into the diffusion-affected zone, isothermally solidified zone and centreline eutectic regions. The normalized distance was calculated from the LAMS angle (θ). From these line scans and other individual point scans, the average chemical composition (at%) of each phase in the ASZ for both FMs is listed in Table 5.3. It was found that both FMs produced the same CrB boride phase with almost identical
compositions. As expected, eutectic constituents were enriched in FM elements, Si and Fe for BNi-2 and Ni and Cr for BNi-9.

![Figure 5.4: X-ray intensity maps of CMSX-4/BNi-2 brazed joint (LAMS) indicating relative distribution of BNi-2 FM elements (B, Cr, Ni, Si and Fe).](image)

In general, the EPMA line scans show little diffusion across the joint boundary for all elements except boron which had a smooth compositional gradient in the DAZ also observed in Figure 5.4. Boron peaks in the athermally solidified zone indicate the presence of CrB and Ni$_3$B borides. Only trace quantities of CMSX-4 elements were detected in the eutectic matrix except for Re, Mo and W enrichment in CrB borides. These elements have been known to promote boride formation and likely entered the liquid FM during the BM dissolution stage [56]. Aluminium and titanium, important elements for the $\gamma'$ strengthening precipitates in CMSX-4 were significantly reduced in the joint regions. Chromium showed strong segregation to the CrB type borides which may be of a concern from corrosion resistance stand point, while nickel and iron were more evenly distributed across the joint.
Figure 5.5: EPMA line scans of CMSX-4/BNi-2 and CMSX-4/BNi-9 brazed joints moving from BM on the left to joint centerline on right.
Table 5.3: Average chemical composition of micro-constituent phases in CMSX-4/BNi-2 and CMSX-4/BNi-9 brazed joints measured from EPMA.

<table>
<thead>
<tr>
<th>FM</th>
<th>Phase</th>
<th>Chemical Composition (at %)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Ni</td>
</tr>
<tr>
<td>BNi-2</td>
<td>CrB</td>
<td>0.90</td>
</tr>
<tr>
<td></td>
<td>Ni3B</td>
<td>65.09</td>
</tr>
<tr>
<td></td>
<td>Si-rich Eutectic</td>
<td>60.63</td>
</tr>
<tr>
<td></td>
<td>Ni-rich Eutectic</td>
<td>78.16</td>
</tr>
<tr>
<td></td>
<td>α-Ni (ISZ)</td>
<td>77.65</td>
</tr>
<tr>
<td>BNi-9</td>
<td>CrB</td>
<td>1.95</td>
</tr>
<tr>
<td></td>
<td>α-Ni</td>
<td>74.50</td>
</tr>
<tr>
<td></td>
<td>Eutectic</td>
<td>59.06</td>
</tr>
</tbody>
</table>

Boron was the only element to show significant diffusion during the brazing process. The DAZ was enriched in boron while the ISZ was depleted in both BNi-9 and BNi-2 joints. Boron and silicon concentration profiles are shown in more detail by the plot of composition (at%) vs. distance from the joint interface in Figure 5.6 for the BNi-2 joint. As shown, the average depth of boron diffusion and width of DAZ was calculated to be approximately 20-30 μm. Silicon which can also act as an MPD element in BNi-2 was highly concentrated in the eutectic matrix and did not show significant diffusion into the CMSX-4 BM. The boron concentration profile for BNi-9 was almost identical as BNi-2 indicating that silicon and boron diffuse independently of each other for these brazing conditions.
Figure 5.6: Boron and silicon EPMA concentration profiles in the DAZ of CMSX-4/BNi-2 brazed joints (left) and LOM image of the DAZ after oxalic etching (right).

No peaks in boron composition were observed in the DAZ indicating boride precipitation did not occur in the CMSX-4 single crystal. Boride formation is often reported in the BM during TLP bonding or when brazing polycrystalline superalloys like IN718 [76, 43]. Without the presence of grain boundaries boron diffusion in the CMSX-4 single crystal is much slower eliminating the formation of borides in this region. However, a small region of boride precipitation occurred in the ISZ at the interface for the BNi-2 FM. The width of the DAZ was also measured experimentally for the normal sectioned BNi-2 joint as shown in Figure 5.6 using an electrolytic etch with oxalic acid. The boron enriched DAZ appears as a lightly etched region on either side of the joint approximately 25 μm in width. This corresponds well to the width determined from the EPMA composition profile and allows for easy measurement of the DAZ width when EPMA is not readily available.
To investigate the effect of increased boron composition on the precipitate strengthened CMSX-4 microstructure, SEM images of the joint boundary were taken after etching as shown in Figure 5.7 at low and high magnifications. The size and distribution of γ'/γ in the CMSX-4 BM appears to be unaffected by boron diffusion and the volume fraction of γ’ was approximately 0.7 right up to the joint interface. A clear boundary line was observed at the joint interface along with a transition region of decreasing γ’ precipitate size moving into the BNi-2 isothermally solidified zone labelled in Figure 5.7. This transition region may be due to the steep Ti/Al compositional gradient at the interface resulting in local precipitation of γ’ during the brazing cycle.

Figure 5.7: SEM images of γ/γ’ prime precipitates and γ’ transition at CMSX-4/BNi-2 joint interface. CrB and Ni$_3$B type borides are present in the joint microstructure.

5.4.3 Micro-hardness Mapping

Micro-hardness measurements give a relative indication of the yield strength of underlying microstructural constituents and are displayed in Figure 5.8 for the BNi-2 brazed joint. The contour plot was generated from over 200 individual indents spaced 100 microns from each other across the LAMS sample. Results indicate the isothermally
solidified zone ($\alpha$-Ni phase) is much softer than both the athermally solidified zone and BM regions. Indents across the normal sectioned joint at higher magnification show the large difference in indent sizes between the $\alpha$-Ni phase and intermetallic constituents. The average Vicker’s Hardness (HVN) values for each phase listed in Figure 5.8 indicate that the CrB and Ni$_3$B borides are extremely hard with HVN of 820 and 983, respectively. For comparison most martensitic steels used for cutting tools have hardness around 800-900 HVN. The $\alpha$-Ni phase is much softer with 308 HV. This drastic change in micro-hardness properties between the ISZ and ASZ over such short distances may lead to strain localization or crack initiation under load around the eutectic constituents and may ultimately contribute to the brittle behavior typically observed in brazed joints with Ni-base FMs. In the CMSX-4 BM a gradient in micro-hardness from the diffusion affected zone (500-586 HV) to the unaffected CMSX-4 (425 HV) was observed. This suggests that boron diffusion increases the local hardness of the $\gamma/\gamma'$ in diffusion affected zone regions likely due to solid solution strengthening.
Figure 5.8: Micro-hardness results for BNi-2 brazed joints. LAMS joint (top) and normal sectioned joint (bottom) used to calculate average Vicker’s hardness for each phase.

In Figure 5.9, micro-hardness results across the normal-sectioned fillet regions of the BNi-9 joint were used to determine average Vickers Hardness values for each phase. The sharp transition in micro-hardness between the isothermally solidified zone and athermally solidified zone in the internal joint regions are difficult to see without LAMS. However, similar to the BNi-2 joint the large difference in material properties between intermetallic phases was apparent in the fillet regions as indicated by the indent size at higher magnification. The α-Ni phase, which formed a dendrite structure in the fillet, was very soft with an average hardness of 396 HV compared to the hard eutectic and CrB phases with hardness values exceeding 1000 HV. The eutectic constituent and α-

<table>
<thead>
<tr>
<th>Phase</th>
<th>Average Hardness (HVN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CrB</td>
<td>983</td>
</tr>
<tr>
<td>Ni₃B</td>
<td>820</td>
</tr>
<tr>
<td>Eutectic Matrix</td>
<td>610</td>
</tr>
<tr>
<td>α-Ni</td>
<td>308</td>
</tr>
<tr>
<td>DAZ (CMSX-4)</td>
<td>586</td>
</tr>
<tr>
<td>Unaffected CMSX-4</td>
<td>425</td>
</tr>
</tbody>
</table>
Ni phase in the BNi-9 joint had a higher hardness than in the BNi-2 braze joint, most likely due to the increased Cr and B content.

Figure 5.9: Micro-hardness mapping and average hardness of micro-constituent phases in fillet region of CMSX-4/BNi-9 brazed joint with normal sectioning.

5.4.4 Dispersion Parameters

Although rarely found in literature dispersion parameters such as volume fraction, average particle size and inter-particle spacing are of particular interest for modelling the response of brazed joints to loading. In addition, this data may be useful for determining cracking susceptibility, validating thermodynamic simulations or quantitatively comparing braze alloy systems similar to welding [26]. For brazed joints these parameters may be determined by distinguishing between the ISZ and centreline eutectic. Since the single phase ISZ is essentially the same for any nickel BM only the phases in
the centreline eutectic will be unique for a given brazed joint. To sort through the multi-component eutectic structure, the color threshold of LAMS images were adjusted in ImageJ to reveal only the phase of interest and then particle analysis was performed to determine average volume fraction and particle size distribution. Figure 5.10 shows the particle size distributions for the Ni$_3$B borides in BNi-2 joints and CrB borides in the BNi-9 joints from analysis of over 200 individual measurements.

![Graphs showing particle size distributions for Ni$_3$B and CrB](image)

**Figure 5.10:** Particle size distribution for primary Ni$_3$B and CrB phases in CMSX-4/BNi-2 and CMSX-4/BNi-9 respectively.

For simplicity all particles were assumed to be circular and a particle radius was calculated from the average particle size. A normal Gaussian distribution was observed for both primary and secondary eutectic phases, with a summary of the results presented in Table 5.4. Ni$_3$B and CrB borides were approximately the same sizes in both BNi-2 and BNi-9 joints, but the average size of the eutectic matrix constituents was much finer (112 μm$^2$) in the BNi-9 brazed joint. Inter-particle spacing for the random distribution of particles was also calculated using Equation 2 below provided by Sha and Guo [130]:

$$\text{Equation 2}$$
\[ L = (1.23 \sqrt{\frac{2\pi}{3f}}) - 2 \sqrt{\frac{2}{3}}r \]  

(5.2)

Where \( L \) is the inter-particle spacing, \( r \) is the particle radius and \( f \) is the precipitate volume fraction. It is unclear at this point why the volume fraction of CrB in the BNi-9 joint (0.07) was lower than the BNi-2 joint (0.10). This may be due to a higher solubility of boron or chromium in the BNi-9 eutectic matrix compared to the Si-rich eutectic matrix for BNi-2.

Table 5.4: Dispersion parameters assessed for micro-constituent phases in CMSX-4/BNi-2 and CMSX-4/BNi-9 braze joints.

<table>
<thead>
<tr>
<th>FM</th>
<th>Vol. Fraction of ISZ</th>
<th>Eutectic Phases</th>
<th>Vol. Fraction (( V_f ))</th>
<th>Avg. Particle Size (( \mu m^2 ))</th>
<th>Avg. Particle Radius (( \mu m ))</th>
<th>Interparticle Spacing (( \mu m ))</th>
</tr>
</thead>
<tbody>
<tr>
<td>BNi-2</td>
<td>0.32</td>
<td>Ni\textsubscript{3}B</td>
<td>0.22</td>
<td>3052</td>
<td>31.17</td>
<td>67.39</td>
</tr>
<tr>
<td></td>
<td></td>
<td>CrB</td>
<td>0.10</td>
<td>2675</td>
<td>29.18</td>
<td>116.61</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ni-rich Eutectic</td>
<td>0.24</td>
<td>622</td>
<td>14.07</td>
<td>28.15</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Si-rich Eutectic</td>
<td>0.44</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>BNi-9</td>
<td>0.29</td>
<td>CrB</td>
<td>0.07</td>
<td>3327</td>
<td>32.54</td>
<td>165.80</td>
</tr>
<tr>
<td></td>
<td></td>
<td>( \alpha )-Ni</td>
<td>0.25</td>
<td>112</td>
<td>5.97</td>
<td>11.51</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Eutectic</td>
<td>0.68</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

5.5 Joint Microstructure of IN718/BNi-2 Braze Joints

Brazed joints with the Inconel 718 BM using the BNi-2 FM produced similar joint microstructures to the CMSX-4 brazed joints as shown in Figure 5.11 after vacuum furnace brazing at 1065°C for 10 minutes. The joint microstructure consists of DAZ, ASZ and ISZ regions as expected. As shown, the DAZ is well defined for the
IN718/BNi-2 joints due to the difference between the single crystal and poly-crystalline BMs with both grain boundary and intra-granular boride precipitation in the DAZ. For polycrystalline materials such as IN718, grain boundaries act as sinks for interstitials or vacancies and have significantly higher diffusivity compared to bulk diffusion [131]. The volume fraction of eutectic constituents in the IN718/BNi-2 joints were markedly lower than the CMSX-4/BNi-2 joints as a result. Although EPMA was not performed the centerline eutectic constituents appear to be similar to the Ni$_3$B and CrB phases observed in the LOM images of CMSX-4/BNi-2 braze joints.

![LOM image of typical IN718/BNi-2 brazed joint microstructure.](image)

Figure 5.11: LOM image of typical IN718/BNi-2 brazed joint microstructure.

### 5.6 Effect of Process Parameters

As discussed in Chapter 2, brazing process parameters such as hold time, joint gap and brazing temperature can significantly influence the brazed joint microstructure especially the volume fraction of centerline eutectics which in turn can directly affect mechanical properties. To examine the influence of these process parameters on the
CMSX-4 braze joint microstructure experimental brazing was performed at OSU using a smaller light radiation furnace (LRF).

CMSX-4 coupons (0.5” x 0.25” x 0.12”) were cut from the same BM plate used in the initial characterization work. All samples were cleaned in acetone before BNi-2 FM was applied to the joint area with a machine finish surface condition. Both paste (powder and binder) and amorphous foil forms of the BNi-2 FM were tested using the LRF setup shown in Figure 5.12 for brazing.

Figure 5.12: Light radiation furnace setup for brazing experiments at OSU.

A total of 20 brazing cycles at different hold times, joint gaps and brazing temperatures were tested with the brazing conditions for each listed in Table 5.5. Heating/cooling rates were kept the same as the standard cycles (Table 5.2) and an inert argon atmosphere (99.99% purity) shielding gas was used in lieu of a vacuum. After brazing all samples were prepared for LOM analysis using conventional metallographic techniques.
LOM was used to measure the average width of the eutectic (ASZ) and ISZ microstructural regions with the width of the isothermally solidified zone ($w_{iso}$) measured as the distance from the FM/BM interface to the start of the centerline eutectic.

Measurements of joint gap ($w_0$) and $w_{iso}$ were averaged over 5-10 measurements taken from several regions for each sample. Using these measurements the average width of the eutectic ($w_{eutectic}$) was calculated as:

$$w_{eutectic} = w_0 - 2 \times w_{iso} \quad (5.3)$$

The volume fraction of eutectic constituents ($f_e$) can then be calculated as:

$$f_e = \frac{w_{eutectic}}{w_0} \quad (5.4)$$

Results from this study are summarized in Table 5.5 with experimental measurements for each braze joint examined. Typical standard deviations for the measured $w_{eutectic}$ ranged between 5-15 microns. Despite the relatively high standard deviation the effect of hold time, brazing temperature and joint gap were clear. In general, increasing temperature and hold time or decreasing joint gap while the other parameters were kept constant led to a decrease in $f_e$. 

80
Table 5.5: List of LRF brazing parameters used for CMSX-4/BNi-2 brazing cycles and experimental measurements of joint microstructural regions.

<table>
<thead>
<tr>
<th>#</th>
<th>Brazing Temperature (°C)</th>
<th>Hold Time (min)</th>
<th>Furnace Form of FM</th>
<th>Avg. w₀ (µm)</th>
<th>Avg. w_{ISZ} (µm)</th>
<th>Avg. w_{eutectic} (µm)</th>
<th>Fraction eutectic fₑ</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1000</td>
<td>10</td>
<td>LRF Paste</td>
<td>212</td>
<td>5</td>
<td>202</td>
<td>0.95</td>
</tr>
<tr>
<td>2</td>
<td>1000</td>
<td>10</td>
<td>LRF Paste</td>
<td>294</td>
<td>9</td>
<td>276</td>
<td>0.94</td>
</tr>
<tr>
<td>3</td>
<td>1055</td>
<td>10</td>
<td>LRF Paste</td>
<td>120</td>
<td>26</td>
<td>68</td>
<td>0.57</td>
</tr>
<tr>
<td>4</td>
<td>1055</td>
<td>10</td>
<td>LRF FOIL</td>
<td>54</td>
<td>10</td>
<td>34</td>
<td>0.63</td>
</tr>
<tr>
<td>5</td>
<td>1065</td>
<td>10</td>
<td>Vacuum FOIL/paste</td>
<td>87</td>
<td>15.5</td>
<td>56</td>
<td>0.64</td>
</tr>
<tr>
<td>6</td>
<td>1065</td>
<td>10</td>
<td>Vacuum FOIL</td>
<td>110</td>
<td>18</td>
<td>74</td>
<td>0.68</td>
</tr>
<tr>
<td>7</td>
<td>1065</td>
<td>10</td>
<td>Vacuum Paste</td>
<td>394</td>
<td>21</td>
<td>353</td>
<td>0.90</td>
</tr>
<tr>
<td>8</td>
<td>1100</td>
<td>10</td>
<td>LRF FOIL</td>
<td>97</td>
<td>21</td>
<td>54</td>
<td>0.56</td>
</tr>
<tr>
<td>9</td>
<td>1100</td>
<td>10</td>
<td>LRF Paste</td>
<td>37</td>
<td>13</td>
<td>11</td>
<td>0.30</td>
</tr>
<tr>
<td>10</td>
<td>1100</td>
<td>10</td>
<td>LRF FOIL</td>
<td>175</td>
<td>32</td>
<td>111</td>
<td>0.63</td>
</tr>
<tr>
<td>11</td>
<td>1170</td>
<td>10</td>
<td>LRF Paste</td>
<td>132</td>
<td>32</td>
<td>68</td>
<td>0.51</td>
</tr>
<tr>
<td>12</td>
<td>1200</td>
<td>10</td>
<td>LRF FOIL</td>
<td>119</td>
<td>52</td>
<td>15</td>
<td>0.13</td>
</tr>
<tr>
<td>13</td>
<td>1200</td>
<td>10</td>
<td>LRF Paste</td>
<td>186</td>
<td>62</td>
<td>62</td>
<td>0.33</td>
</tr>
<tr>
<td>14</td>
<td>1200</td>
<td>10</td>
<td>LRF FOIL</td>
<td>296</td>
<td>79</td>
<td>139</td>
<td>0.47</td>
</tr>
<tr>
<td>15</td>
<td>1065</td>
<td>90</td>
<td>LRF Paste</td>
<td>100</td>
<td>26</td>
<td>47</td>
<td>0.47</td>
</tr>
<tr>
<td>16</td>
<td>1100</td>
<td>90</td>
<td>LRF FOIL</td>
<td>129</td>
<td>37</td>
<td>54</td>
<td>0.42</td>
</tr>
<tr>
<td>17</td>
<td>1100</td>
<td>90</td>
<td>LRF FOIL</td>
<td>80</td>
<td>35</td>
<td>11</td>
<td>0.14</td>
</tr>
<tr>
<td>18</td>
<td>1100</td>
<td>90</td>
<td>LRF Paste</td>
<td>133</td>
<td>47</td>
<td>39</td>
<td>0.29</td>
</tr>
<tr>
<td>19</td>
<td>1170</td>
<td>90</td>
<td>LRF Paste</td>
<td>221</td>
<td>86</td>
<td>50</td>
<td>0.23</td>
</tr>
<tr>
<td>20</td>
<td>1190</td>
<td>90</td>
<td>LRF Paste</td>
<td>56</td>
<td>27</td>
<td>2</td>
<td>0.04</td>
</tr>
</tbody>
</table>

Figure 5.13 shows the effect of hold time on CMSX-4/BNi-2 joint microstructure at a joint gap of approximately 87 µm and a brazing temperature of 1100°C for two different hold times of 10 (left) and 90 minutes (right) corresponding to sample #8 and #17. As shown, the joint centerline eutectic is significantly reduced by increasing the hold time with the average fₑ decreasing from 0.56 to 0.14 for the 10 and 90 minute hold respectively. In addition, the brown colored, silicon rich phase is absent from the joint indicating that silicon diffusion may be possible at longer hold times.
Figure 5.13: Effect of hold time on CMSX-4/BNi-2 joint microstructure for 10 minute hold (left) and 90 minute hold (right) at 1100°C and 100 µm joint gap.

Brazing temperature has a similar effect on the joint microstructure as shown in Figure 5.14 for CMSX-4/BNi-2 joints with a 10 minute hold time at three different brazing temperatures of 1000°C, 1100°C and 1200°C from left to right corresponding to samples #1, #10 and #13 respectively. The effect of isothermal solidification can clearly be seen from these images as the width of the ISZ at the joint interface increases with brazing temperature due to the increased boron diffusion.

Figure 5.14: Effect of brazing temperature on CMSX-4/BNi-2 joint microstructure for brazing temperatures of 1000°C, 1100°C and 1200°C from left to right with a 10 minute hold time and 100 µm joint gap.
The plot in Figure 5.15 provides a good summary of the influence of brazing temperature and hold time on the fraction eutectic for all joints evaluated. Since the fraction eutectic is directly related to the extent of diffusion that occurs during brazing, increasing both temperature and time reduces the average volume fraction eutectic. While there was some scatter in the data, the overall trend, without considering the joint gap or form of FM, is a linear decrease in fraction eutectic with increasing brazing temperature for both 10 and 90 minute hold times. Also, for a given brazing temperature, the volume fraction eutectic was lower for the 90 minute hold time compared to the 10 minute hold time for almost all samples.

Figure 5.15: Effect of brazing temperature and hold time on fraction eutectic in CMSX-4/BNi-2 braze joints.
One additional observation that should be noted from this study was that the form of FM also appeared to have an influence on the volume fraction of eutectic constituents present in the joint. Normal and LAMS joint microstructures using paste (left) and amorphous foil (right) are shown in Figure 5.16 for the standard CMSX-4/BNi-2 brazing cycle at a temperature of 1065°C with a 10 minute hold time. As shown, the amorphous foil produces a significantly different microstructure compared to the paste and the ISZ/ASZ regions are hard to distinguish.

Figure 5.16: LOM images of CMSX-4/BNi-2 brazed joint microstructures using paste form of FM (left) and amorphous foil (right).
This phenomenon was only observed at lower brazing temperatures of 1065°C or lower whereas higher brazing temperatures resulted in a move conventional microstructure with the typical ASZ and ISZ regions. Amorphous foil brazing FMs are made using a rapid solidification melt-spinning process that create a uniform atomic structure due to the rapid cooling rates on the order of ~1,000,000 C/s and avoid oxide formation on their surfaces compared to conventional powder FMs. This allows them to melt quickly within a narrow temperature range and since the foil can be pre-placed in narrow joint gaps they have been reported to form less detrimental eutectic phases [132].

Micro-hardness mapping across the LAMS samples of the foil and paste joint microstructures in Figure 5.17 indicates the reduction of eutectic phases from the joint microstructure also significantly reduces the hardness of the joint as well. A similar effect would be expected for allowing complete isothermal solidification to occur. As shown, the hardness in the joint area is lower than the surrounding BM regions associated with the DAZ. As previously mentioned the diffusion of boron into these regions increases the local hardness of the BM via a solid-solution strengthening mechanism.
Figure 5.17: Comparison of micro-hardness in CMSX-4 brazed joints made with powder (top) and amorphous foil (bottom) form of BNi-2 FM.

5.7 Summary

CMSX-4 and Inconel 718 brazed joints produce two primary microstructural regions for both BNi-2 and BNi-9 FMs, a hard, multi-component centerline eutectic and soft, ductile solid solution ISZ at the joint interface. Dispersion parameters including volume fraction, average particle size and inter-particle spacing were measured experimentally for the micro-constituents present in these regions for the CMSX-4 brazed joints. Compositional analysis using EPMA and X-ray intensity mapping identified the
primary eutectic constituents as Cr- and Ni-rich borides. In addition, a boron compositional gradient was observed in the CMSX-4 BM indicating boron was the primary diffusing MPD element for these FMs. The boron diffusion did not seem to affect the γ/γ’ distribution in the CMSX-4 BM, however it did result in boride precipitation in the diffusion affected zone for the poly-crystalline IN718 BM.

Micro-hardness showed large difference in properties between the ISZ and eutectic microstructural regions with the BM hardness falling between these two. The Cr-rich and Ni-rich borides exhibited even higher hardness from the eutectic matrix with peak values ranging from 800 to 1000 HVN.

The process parameters (hold time, temperature, joint gap) had a strong influence on the brazed joint microstructure by affecting the extent of boron diffusion and isothermal solidification. As a result, increasing time, temperature and decreasing joint gap all led to a reduction of centerline eutectic constituents. In addition, the amorphous foil form of FM was found to produce joints with lower volume fraction of eutectic constituents at temperatures below 1065°C. Micro-hardness measurements show the reduction of centerline eutectic constituents can dramatically change the properties of the joint material.
CHAPTER 6

6. THERMODYNAMIC AND KINETIC SIMULATIONS

6.1 Introduction

In the previous chapter, the brazing process parameters such as joint gap, hold time and brazing temperature were shown to significantly influence joint microstructure and volume fraction of centreline eutectic phases. Therefore, an accurate multi-scale model of brazed joints should incorporate these effects as the microstructure can directly influence joint properties. This has been accomplished to some degree by analytical and numerical modelling as described in chapter 2, however these calculations are limited in describing the transient processes such as dissolution and isothermal solidification that occur during the brazing process.

CALPHAD based calculations using Thermo-Calc and multi-component diffusion simulations in DICTRA are better suited for solving these transient problems. However, there have been few publications using these simulations to model actual brazed joint microstructure. As a result, in this chapter simulations will be performed with Thermo-Calc and DICTRA software to predict the microstructural evolution and final joint microstructure observed in the Ni-base superalloy brazed joints.

Diffusion controlled transformations, i.e. dissolution and isothermal solidification, modelled with DICTRA and Thermo-Calc (CALPHAD) equilibrium calculations will be
used to predict the eutectic constituents formed during normal solidification of the remaining FM. Simulations and experimental work will be focused on the CMSX-4 Alloy using both BNI-2 and BNI-9 FMs since extensive characterization was carried out for these brazed joints. A new technique, single sensor differential thermal analysis [133], will be used to measure phase transformation temperatures for comparison with Thermo-Calc results and additional experimental brazing of CMSX-4 with BNI-2 will be used to validate the DICTRA simulations. The overall goal of this chapter is to demonstrate the applicability of such simulations for predicting joint microstructure based on brazing process parameters such as joint gap, brazing temperature and hold time. The limitations, areas for improvement and practical implementation of these simulations for multi-scale modelling of brazed joints will also be discussed.

6.2 Model Setup

6.2.1 DICTRA Model Setup

In DICTRA, multi-component diffusion equations are solved using a temperature and concentration dependent diffusivity matrix [134]. Details of the numerical models described by Ohsasa et al. [74] for the TLP bonding process were briefly discussed in Chapter 2 and involve solving diffusion and mass balance equations using the explicit finite difference method. The DICTRA model setup used in this study, shown schematically in Figure 6.1, was adopted from Schnell et al. [45] who used the model to calculate BM dissolution and isothermal solidification for CMSX-4 with a D-15 braze alloy. Schnell et al. used this setup to optimize the brazing temperature and achieve the shortest isothermal solidification time for the D-15 FM (2.3 wt% boron) at a constant
joint gap of 100 µm and found good agreement with experimental joint microstructures. Instead for this study, the model was applied using the BNi-2 boron composition at a constant brazing temperature for a range of joint gaps.

Figure 6.1: Schematic of DICTRA model setup with boundary conditions for CMSX-4 brazed with BNi-2 FM.

Only the Ni-B binary system was considered in the DICTRA simulations as boron was the only MPD element to show significant diffusion (as discussed in Chapter 5) and the kinetics of the solid/liquid interface in multi-component systems is usually controlled by the fastest diffusing element (boron) [45, 135]. The MACRO file or command script generated for the DICTRA model is listed Appendix C for reference. Due to symmetry, half of the joint was modelled with two regions; a liquid region for the BNi-2 FM with an initial boron composition of 3.1 wt% and a CMSX-4 BM region consisting of the FCC (γ) phase with an initial composition of 0 wt% boron. The width of the liquid region was set as half of the initial joint gap (w₀/2) and varied from 5 to 60 µm (10 to 120 µm joint
gap) with 20 elements uniformly distributed across the width for analysis. 100 elements with a single bias were assigned to the BM region which was kept at a constant width (w) of 20 mm so w>>w₀/2 (see Figure 6.1). Thermodynamic databases are limited for brazing FMs [42], so calculations were made using the solid solution thermodynamic database (SSOL4) and mobility database (MOB2) at a constant temperature of 1170°C. Both the boron composition and the position of the solid/liquid interface were calculated for times ranging from 0 to 1x10⁹ seconds.

6.2.2 Thermo-Calc Model Setup

To predict the eutectic constituents that would result in solidification of both nickel-based FMs, equilibrium phases were calculated from room temperature (25°C) to 1300°C for both BNi-2 and BNi-9 compositions (Table 5.1) using the nickel database (TTNi8). Equilibrium conditions were assumed due to the slower heating and cooling rates associated with vacuum furnace brazing. However, Thermo-Calc is also capable of calculating solute redistribution during non-equilibrium solidification through the Scheil equation [74].

6.2.3 Brazing Experiments

EPMA compositional analysis reported in Chapter 2 was used to compare with Thermo-Calc simulations. To validate the DICTRA models additional brazing of CMSX-4 with BNi-2 was carried out using the light radiation furnace (LRF) setup. Shown schematically in Figure 6.2, these specimens were brazed at an angle using a thin thermocouple wire (0.250 mm thick) as a spacer at one end to create a range of joint gaps. This approach was first described by Lugscheider et al. [50] to determine the
maximum brazing clearance (MBC) required for isothermal solidification by measuring the joint gap at which centreline eutectic phases first appear. For a direct comparison to the DICTRA model a brazing temperature of 1170°C and four hold times of 10 minutes, 90 minutes, 4 hours and 7.5 hours were used with two samples at each hold time. All joint specimens were cross-sectioned normal to the joints and polished to a 1 μm finish using standard metallography techniques. LOM was used for evaluation of braze joint microstructure for these samples.

Figure 6.2: Schematic of test specimen used to determine the maximum brazing clearance (MBC) required for isothermal solidification [50].

6.3 Results and Discussion

6.3.1 DICTRA – Dissolution and Isothermal Solidification

The results of kinetic simulations using the DICTRA model for a joint gap of 80 μm at hold times of 0, 1, 4, 600, 3600, 10800, 21600, and 31700 seconds are shown in Figure 6.3. The initial solid/liquid interface at t = 0 s was set at 40 μm from the joint centerline and the initial boron composition in the BNi-2 FM (C₀) was 3.1 wt%. Within the first four seconds at 1170°C DICTRA predicts dissolution of the BM will occur to achieve equilibrium compositions in the liquid (C_L) and solid (C_S) at the interface. As the
holding time increases, boron continues to diffuse into the FCC region and the interface
moves from right to left due to isothermal solidification. This process continues until the
diffusion time reaches 8.8 hours (31700 seconds) at which point the isothermal
solidification is predicted to be complete and no liquid FM remains in the joint.

Figure 6.3: DICTRA results for BNi-2 FM with initial joint gap of 80 μm and hold times
of 0, 1, 4, 600, 3600, 10800, 21600 and 31700 seconds. The position of the solid/liquid
interface shows BM dissolution and isothermal solidification during brazing.

By plotting the position of the solid/liquid interface as a function of hold time (log
scale) the dissolution and isothermal diffusion solidification time (tiso) can be easily
identified as shown in Figure 6.4. Again the initial position at t=0 s is 40 μm. BM
dissolution and widening of the liquid FM region occurs and reaches a maximum width
of 43 µm after 4 seconds. The position of the interface then moves back toward the joint centerline as isothermal solidification takes place until the interlayer is completely solid at t=31,700 s.

Figure 6.4: Calculated position of solid/liquid interface for different holding times in the CMSX-4/BNi-2 DICTRA model with an initial joint gap of 80 µm.

In Figure 6.5, the brazed joint microstructures at an initial joint gap of approximately 80 µm brazed at 1170°C for 10 min, 90 min and 450 min show good correlation with DICTRA results. As the hold time increases, the width of the eutectic region decreases until isothermal solidification is complete as shown for the 450-minute (7.5-hour) hold time which consists only of the solid-solution ISZ phase.
Figure 6.5: CMSX-4/BNi-2 joint microstructures for hold times of 10, 90 and 450 minutes at 1170°C and initial joint gap of 80 µm. ISZ grows as MPD elements diffuse from joint and solidification occurs at the brazing temperature.

Using the same approach $t_{iso}$ was calculated for joint gaps ranging from 10-120 µm for the BNi-2/CMSX-4 setup and plotted in Figure 6.6 with each point representing a separate DICTRA calculation. The predicted hold time dependency on the initial joint gap is closely described by the square root relationship provided by Ramirez and Liu [67] as:

$$\sqrt{t_{iso}} = \frac{w_0}{\gamma_s \sqrt{4D_s}}$$ (6.1)

where $\gamma_s$ is a dimensionless parameter related to the solidification characteristics of the brazing process, $w_0$ is the initial joint gap and $D_s$ is the boron diffusivity in solid nickel. This plot is a useful tool for brazing engineers to determine the joint clearance required for complete isothermal solidification. As expected $t_{iso}$ is much shorter for small joint gaps since there is less liquid FM i.e. boron and the distance required for boron diffusion is much shorter.
Figure 6.6: Time required for complete isothermal solidification at various joint gaps predicted by the DICTRA model setup compared to experimental results and results reported by Schnell [45] and Nishimoto [136].

The maximum brazing clearance for isothermal solidification (MBC) of the actual joint microstructures showed similar results for $t_{iso}$. As shown in Figure 6.7 for the 10, 90 and 450 minute hold times, the initial joint gap where the eutectic phases first appear is taken as the MBC and was determined to be 9, 34 and 94 μm respectively. These measurements are somewhat subjective as the initial joint gap is difficult to identify after brazing due to BM dissolution, therefore nearby voids or areas free of the brazing FM were used to determine the initial joint gap. The range between MBC measurements for the same hold time was typically ±9 μm. Nishimoto et al. reported similar results when
brazing CMSX-2 with a MBF-80 (Ni-15.5Cr-3.7B) and determined a $t_{iso}$ of $\sim$2.7 hours for an initial joint gap of 40 $\mu$m at $T_b$ of 1180°C [136]. Schnell et al. reported $t_{iso}$ for a 100 $\mu$m $\pm$ 10 $\mu$m joint gap with CMSX-4/D-15 braze joints was 8 hours at $T_b$ of 1200°C [45].

Figure 6.7: CMSX-4/BNi-2 joint microstructure after brazing at 1170°C for 10, 90 and 450 minute hold times shows the maximum brazing clearance for complete isothermal solidification.

Experimental results from this study and those reported by Nishimoto and Schnell are plotted in Figure 6.6 and show good correlation with DICTRA simulations for the initial joint gap required for isothermal solidification. For wider joint gaps, DICTRA predicts slightly higher $t_{iso}$ than the experimentally measured joint gap, indicating slower diffusion rates for the CMSX-4/BNi-2 joints. This could be attributed to the segregation of boron at the joint interface resulting in the formation of small boundary borides, as shown in Figure 6.7. Similar to boride formation in the DAZ of polycrystalline superalloys, the high concentration of boron in these phases would reduce the overall
boron content for diffusion and as a result, lead to shorter isothermal solidification times at larger joint gaps.

DICTRA simulations applied to the dissolution and isothermal solidification in high temperature brazing of nickel based superalloys can be useful in several ways. First, the volume fraction of centerline eutectic remaining in the joint, which influences the overall joint properties, can be predicted based on the diffusion between the FM and BM. Second, the time required for complete isothermal solidification can be estimated for different joint gaps to determine the maximum brazing clearance allowable in TLP bonding or diffusion brazing. Third, BM dissolution which is critical for brazing thin walled structures can be modelled. Finally, with further database development the DICTRA model can be applied for a wide range of process parameters including brazing temperature, joint gap, hold time and FM/BM compositions.

6.3.2 Thermo-Calc Modeling of FM Solidification

The long hold times required for complete isothermal solidification are often not applied in high temperature brazing of superalloys, so some amount of the FM will remain liquid upon cooling from the brazing temperature. In this case, Thermo-Calc can be used to predict the eutectic constituents and phase transformation temperatures that are likely to occur during solidification. The plot in Figure 6.8, shows the volume fraction of equilibrium phases predicted from 25-1500°C and the intermetallic phases identified through metallurgical characterizations for the BNi-2/CMSX-4 braze joint. On cooling from the liquid phase Thermo-Calc predicts four intermetallic phases form, MB_ORTH, FCC_A1, M3B and GAMMA_PRIME.
Figure 6.8: Equilibrium phases predicted with Thermo-Calc for BNi-2 FM and corresponding phases in the BNi-2 brazed joint; 1) Liquid 2) MB_ORTH, CrB boride 3) FCC_A1, α-Ni phase 4) M3B, Ni$_3$B boride 5) M2B_TETR phase 6) GAMMA_PRIMA, Ni$_x$Si$_y$

The composition and volume fraction of these equilibrium phases at 25°C from Thermo-Calc is listed in Table 6.1 along with the experimentally determined compositions from EPMA analysis of the joint micro-constituents. As labelled in Figure 6.8, the CrB, Ni$_3$B, α-Ni and silicon rich eutectic phases identified in the BNi-2 joint microstructure correspond well with Thermo-Calc equilibrium phases as do the volume fractions of these phases determined experimentally using ImageJ analysis [135]. Similar phases were reported for BNi-2 by Vargas et al. [42] using a pure nickel substrate. The MB_ORTH or the CrB boride is expected to solidify first at 1260°C in the centreline eutectic regions, followed by the FCC_A1 phase which is same white colored phase as
the ISZ. M3B (Ni$_3$B) phase is predicted to form at the very end of solidification just before the eutectic temperature is reached around 1041°C which is slightly above the solidus temperature of 971°C listed for BNi-2 in the AWS A5.8 standard [36]. The GAMMA_PRIME phase, of the Ni$_x$Si$_y$ variety, forms in the solid-state below the liquidus temperature and appear as gold colored intermetallic phase in the joint microstructure.

Table 6.1: Composition and volume fraction of equilibrium phases predicted by Thermo-Calc and measured through EPMA for CMSX-4/BNi-2 braze joints.

<table>
<thead>
<tr>
<th>Method</th>
<th>Phase</th>
<th>Vol. Fraction</th>
<th>Chemical Composition (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Ni</td>
</tr>
<tr>
<td><strong>Experimental (EPMA)</strong></td>
<td>CrB</td>
<td>0.13</td>
<td>1.94</td>
</tr>
<tr>
<td></td>
<td>Ni$_3$B</td>
<td>0.22</td>
<td>81.88</td>
</tr>
<tr>
<td></td>
<td>α-Ni</td>
<td>0.40</td>
<td>80.80</td>
</tr>
<tr>
<td></td>
<td>Eutectic</td>
<td>0.25</td>
<td>83.70</td>
</tr>
<tr>
<td><strong>Calculated (Thermo-Calc @ 25°C)</strong></td>
<td>CrB (MB_ORTH)</td>
<td>0.12</td>
<td>--</td>
</tr>
<tr>
<td></td>
<td>Ni$_3$B (M3B)</td>
<td>0.33</td>
<td>94.22</td>
</tr>
<tr>
<td></td>
<td>α-Ni (FCC_A1)</td>
<td>0.25</td>
<td>97.10</td>
</tr>
<tr>
<td></td>
<td>GAMMA_PRIME</td>
<td>0.30</td>
<td>85.39</td>
</tr>
</tbody>
</table>

Similar equilibrium calculations were performed for the BNi-9 FM which produced a slightly different centerline eutectic than BNi-2 due to the difference in composition. Figure 6.9, shows the Thermo-Calc equilibrium phases for the BNi-9 composition at temperatures from 220-1500°C and the BNi-9/CMSX-4 joint microstructure with intermetallic phases labelled as CrB, Ni3B and α-Ni (FCC). These phases were also reported by Ohsasa et al. when brazing with a similar Ni-Cr-B FM on pure nickel substrate [74]. As expected, without any silicon in BNi-9 the silicon rich intermetallic phase (GAMMA_PRIME) was not found in the joint microstructure and wasn’t predicted by Thermo-Calc. BNi-9 solidification begins with the α-Ni phase.
(FCC_A1) at 1065°C with a higher chromium and boron content than the FCC_A1 found in BNi-2. As cooling continues the same MB_ORTH, CrB phase found in BNi-2 is predicted to form before a final eutectic temperature at 1010°C is reached. The calculated BNi-9 solidus (1010°C) and liquidus (1065°C) temperatures predicted by Thermo-Calc are within 10°C of the standard values, 1020°C and 1055°C respectively. Below 1010°C a multi-component eutectic consisting of FCC_A1, MB_ORTH and a nickel boride (M3B) is expected.

![Thermo-Calc Diagram](image)

Figure 6.9: Equilibrium phases predicted with Thermo-Calc for BNi-9 FM and corresponding phases in the BNi-9 brazed joint; 1) Liquid 2) FCC_A1, α-Ni phase 3) MB_ORTH, CrB boride 4) M3B, Ni3B boride.

Table 6.2, shows the chemical composition and volume fraction of these phases at 220°C predicted by Thermo-Calc correlate well with EPMA and ImageJ analysis of the
actual joint microstructure. EPMA results also confirm the CrB type borides in the BNi-9 joints are the same as those found in BNi-2 joints. The two-phase, eutectic matrix surrounding the CrB borides in the BNi-9 joint microstructure was found to contain the α-Ni phase and a nickel/boron rich phase.

Table 6.2: Composition and volume fraction of equilibrium phases predicted by Thermo-Calc and measured with EPMA for CMSX-4/BNi-9 braze joints.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Vol. Fraction</th>
<th>Chemical Composition (wt%)</th>
<th>Others (W, Re, Al, Ti, Co, Mo)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CrB</td>
<td>0.09</td>
<td>3.79 20.48 64.18 11.55</td>
<td></td>
</tr>
<tr>
<td>α-Ni</td>
<td>0.30</td>
<td>73.70 0.56 12.63 13.11</td>
<td></td>
</tr>
<tr>
<td>Eutectic</td>
<td>0.61</td>
<td>70.40 5.10 11.60 12.90</td>
<td></td>
</tr>
<tr>
<td>CrB (MB_ORTH)</td>
<td>0.28</td>
<td>0.00 17.21 82.79 0.00</td>
<td></td>
</tr>
<tr>
<td>α-Ni (FCC_A1)</td>
<td>0.63</td>
<td>99.99 0.00 0.00 0.00</td>
<td></td>
</tr>
<tr>
<td>Ni₃B (M3B)</td>
<td>0.09</td>
<td>94.22 5.78 0.00 0.00</td>
<td></td>
</tr>
</tbody>
</table>

6.4 Single Sensor Differential Thermal Analysis

To validate the Thermo-Calc equilibrium calculations a simple, inexpensive technique was developed to study the phase transformations of the BNi-2 and BNi-9 FMs. Conventional differential thermal analysis (DTA) are often used to measure liquidus/solidus temperatures as well as any solid-state transformations, however these techniques require a relatively large amount of braze alloy material that is isolated from a reference material [137, 138]. This can be expensive if testing a range of alloy compositions especially when dealing with precious metals like Ag- or Au-based FMs. In addition, the metallurgical interactions between the braze alloy and BM during brazing
can result in significant changes in composition and change the effective solidus/liquidus temperature of the braze joint thus changing the effective operating temperature of the joint [51]. As a result, we have developed a simple technique using single sensor differential thermal analysis (SS DTA) to quickly and accurately determine the liquidus/solidus temperatures and identify any potential solid-state transformations for braze alloys.

The SS DTA technique was originally developed by Alexandrov et al. for studying phase transformations during welding and post-weld heat treatments [139]. A MATLAB code is used to generate a reference curve (a best fit curve) from the heating or cooling curve measured from a thermocouple that is in direct contact with a material sample of interest. The reference curve is generated in a region without phase transformations and is extrapolated through the temperature range of the phase transformation of interest. The difference between the actual and reference thermal histories is then plotted to observe thermal variations corresponding to a latent heat of transformation. SS DTA has been shown to be very sensitive and accurate for measuring solid-liquid and solid state transformations such as melting and solidification, liquation and formation of eutectic constituents, austenitization temperatures in steels, precipitation of carbides, nitrides or γ'/γ” in nickel alloys [140]. This technique is well suited for braze alloys because it can be directly applied in a brazing heat cycle for a range of heating methods, it only requires a small volume of material, and has exhibited excellent test-to-test repeatability. In this section, the experimental procedure using this technique for
studying braze alloys and initial SS DTA results with the BNi-2 and BNi-9 FMs will be discussed.

The setup for SS DTA, shown in Figure 6.10, is simple consisting of a single thermocouple (Type K) welded to the substrate material so that the thermocouple will be immersed in the liquid FM on melting. For this initial study approximately 0.25 grams of BNi-2 and BNi-9 powder was spread over the SSDTA thermocouple prior to brazing with CMSX-4 as the substrate. A light radiation furnace (LRF) with inert argon atmosphere was used for heating and a separate control thermocouple was used to regulate the furnace temperature. Standard brazing cycles (Table 5.2) were and temperature measurements from the SS DTA thermocouple were collected during the brazing cycle at 100 Hz using an InstruNet analog/digital input/output system.

Figure 6.10: Experimental setup for SS DTA testing of braze alloys.

Temperature profiles obtained from the SS DTA thermocouples are shown in Figure 6.10 for the BNi-2 and BNi-9 brazing cycles. Both heating and cooling curves
were analyzed with SS DTA after brazing. Since melting occurs in the final heating stage this portion of the curve was used for SS DTA on-heating. A linear best fit curve was selected due to the constant heating and cooling rates applied, however polynomial or exponential fit curves may be more applicable for other heating methods such as torch brazing.

![Graph showing temperature vs. time with two stages of melting and solidification.](image)

**Figure 6.11**: Recorded temperature of SS DTA thermocouples during brazing with BNi-2 (blue) and BNi-9 (black) FMs.

SS DTA results on-heating (left) and on-cooling (right) for the BNi-2 FM are shown in Figure 6.12. As shown, melting occurs in two primary stages with the first stage beginning around 971°C and completing around 1000°C and the second stage starting at approximately 1020°C and completing around 1040°C. The on-cooling curve indicates a similar two stage solidification process with the first stage beginning at 1018°C and the final stage completing at 953°C. This two stage melting/solidification behavior was
previously reported in conventional DTA for the BNi-2 FM by Vargas et al. [42]
indicating good agreement between SS DTA and conventional DTA.

![Figure 6.12: SS DTA results for BNi-2 FM on-heating (left) and on-cooling (right).](image)

Similarly, the SS DTA results for the BNi-9 FM on cooling are shown in Figure 6.13. Interestingly only one peak is observed during the solidification of BNi-9 and the solidification temperature range is much narrower starting at 1043°C and ending at 1010°C. These results agree with both the Thermo-Calc simulations and joint microstructure for the BNi-9 FM which show only a two phase centerline eutectic compared to the multi-component BNi-2 eutectic. No solid-state transformations were observed after solidification was complete for either FM.

Solidus/liquidus temperatures determined by SS DTA for both FMs on-heating and on-cooling are listed in Table 6.3. These are in good agreement with solidus/liquidus values provided in the AWS A5.8 specification of 971°C/999°C and 1055°C/1055°C for BNi-2 and BNi-9 respectively [36], but slightly higher than the Thermo-Calc equilibrium
predictions for these FMs. Repeat testing using the same test setup gave similar results for both FMs and transformation temperatures within +/-3°C. SS DTA has also been very successful in identifying solid-state transformations, however no thermal effects were observed below the solidus temperature for either FM present in this study.

![Graph](image.png)

Figure 6.13: SS DTA results for BNi-9 FM on-heating (left) and on-cooling (right).

Table 6.3: Liquidus/solidus temperatures measured using SSDTA for BNi-2 and BNi-9.

<table>
<thead>
<tr>
<th>Filler Metal</th>
<th>Solidus (°C)</th>
<th>Liquidus (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BNi-2</td>
<td>On-Heating</td>
<td>971</td>
</tr>
<tr>
<td></td>
<td>On-Cooling</td>
<td>953</td>
</tr>
<tr>
<td>BNi-9</td>
<td>On-Heating</td>
<td>1046</td>
</tr>
<tr>
<td></td>
<td>On-Cooling</td>
<td>1010</td>
</tr>
</tbody>
</table>

In general, the SSDTA technique has the following advantages for brazed joints; (1) it requires a small volume of braze alloy, (2) it can be directly applied to a braze joint in a standard brazing heat cycle, and (3) can account for any change in composition that may occur during the brazing process. As a result, this technique provides a simple,
inexpensive method for rapid determination of liquidus/solidus temperatures in braze alloy development. Future work using this technique will involve studying how the diffusion of melting point depressant elements from the joint into the BM during the brazing process affects the liquidus/solidus temperature on-cooling.

6.5 Implementation of Thermodynamic and Kinetic Simulations

The solidification process and equilibrium phases predicted with Thermo-Calc using the nickel superalloy database show surprisingly good correlation with experimental results. However, by assuming equilibrium conditions the diffusion of MPD elements during the process is ignored. As a result, both Thermo-Calc and DICTRA calculations are also necessary to predict the final joint microstructure and volume fraction eutectic in the final joint microstructure. By combining both simulations the total volume fraction of eutectic ($f_e$) remaining in the joint can be calculated as:

$$f_e = 1 - f_{iso} - f_{FCC,A1}$$  \hspace{1cm} (6.2)

where $f_{iso}$ is the volume fraction of the ISZ region predicted with DICTRA and $f_{FCC,A1}$ is the equilibrium volume fraction of the solid solution phase during normal FM solidification predicted in Thermo-Calc. Using rule of mixtures methodology, similar to the approach described by Yu et al., the overall strength of the brazed joint (Eqn. 2.2) can then be roughly estimated from these simulations [59].

It should be noted that there are limited thermodynamic and kinetic databases available for braze alloys. In this study both nickel and solid solution databases were utilized, but a database that includes assessment of MPD elements such as boron, silicon and phosphorous would greatly improve simulation capabilities. As a result, simulations
are currently limited to temperatures and composition ranges that have been assessed for other alloy system and may not be applicable for the wide range of brazing FMs. However, the potential application for these simulations to predict joint microstructure and ultimately mechanical properties may encourage development of such databases. The experimental methods like SS DTA, EPMA and characterization techniques outlined in this study are intended to assist in this development.

6.6 Summary

Microstructural evolutions in CMSX-4 braze joints were simulated using both DICTRA and Thermo-Calc software to predict the final volume fraction of microconstituent phases. The formation of the ISZ due to boron diffusion during brazing was predicted with a DICTRA model based on the Ni-B binary system for various hold times and joint gaps. Thermo-Calc equilibrium calculations were performed using the compositions of the two BFMs, BNi-2 and BNi-9, to predict the solidification behavior and phase transformations on cooling. Results showed that similar CrB and α-Ni equilibrium phases were predicted for the both FMs with an additional Ni$_{x}$Si$_{y}$ phase (GAMMA_PRIME) in BNi-2 joint which contains silicon.

The average maximum brazing clearance for avoidance of centreline eutectics in CMSX-4 specimens brazed with BNi-2 at 1170°C for hold times of 10, 90, 240 and 450 minutes found to be 9, 34, 64 and 94 μm respectively correlated well with the DICTRA model predictions. EPMA composition analyses and volume fraction point counting indicated similar micro-constituents and distributions as predicted by Thermo-Calc
simulations especially for the CrB and Ni$_3$B borides. However, limitations in the thermodynamic and mobility databases for braze alloys were pointed out.

Both kinetic and thermodynamic simulations are necessary to fully predict the microstructural evolution during brazing. Together these can be useful tools in designing braze joints and can significantly reduce time and cost in the development and optimization of brazing processes. As with all modelling efforts, simulations should be validated with experimental characterizations before they are applied in practice.
CHAPTER 7

7. MECHANICAL BEHAVIOR OF BRAZED JOINTS

7.1 Introduction

In this Chapter, the mechanical behavior of the CMSX-4/BNi-2 and IN718/BNi-2 brazed joints will be evaluated using several experimental methods. First, the BM tensile properties will be assessed for both Ni-base superalloys to provide a base line for comparison. Following AWS C3.2 standard IN718/BNi-2 butt joint tensile tests will be performed to measure the UTS of the brazed joint. In addition, a novel approach for brazed joints using a small-scale tensile test system will be used to perform in-situ observations of CMSX-4 and IN718 joint deformation behavior and quantify the influence of joint microstructure on strength and ductility. Finally, the AWS C3.2 lap shear test method will be carried out on both CMSX-4 and IN718 brazed joints to establish the average FM shear stress for a range of overlaps. Due to the non-uniform stress distribution observed in lap shear testing digital image correlation (DIC) will be utilized to observe the strain distribution across the brazed joint. FEA models of the lap shear test specimens will be used to validate the DIC results and develop damage zone models for prediction of the lap shear failure loads.

7.2 Base Metal Properties
The tensile properties of the IN718 and CMSX-4 BMs were evaluated to provide a base line for comparison with the brazed joints and stress/strain properties for FEA models. ASTM E8 test procedures were followed for sample fabrication and testing with sample dimensions listed in Appendix A.

Standard size test samples were used for IN718 and slightly smaller test samples for CMSX-4 due to limited material availability. Samples were tested in the solution and aged condition following industry standard heat treatments. The solution and age heat treatment for IN718 shown in Figure 7.1 is as follows; solution anneal for 1 hour at 1850F followed by a quench and aging cycle at 1325F for 8 hours and 1150 F for 7 hours. Tensile testing was performed at OSU using a servo-hydraulic 810 Material Testing System (MTS) at a strain rate of 0.001 in/in/s.

![Figure 7.1: Post-braze solution and age heat treatment for IN718 mechanical test specimens.](image-url)
The stress/strain curves generated for CMSX-4 and IN718 are provided in Figure 7.2. As shown, the poly-crystalline IN718 has a significantly higher YS and UTS than the single crystal CMSX-4 material in the solution and aged condition. The CMSX-4 was found to be highly anisotropic with both the YS and elastic modulus depending on the orientation of the single crystal. In addition, CMSX-4 exhibited significantly less work hardening for the CMSX-4 BM since plastic deformation occurs along one primary slip system. Due to issues with anisotropy and material costs, the IN718 BM was selected as the primary alloy of interest for evaluating brazed joint mechanical behavior in this study. The average elastic modulus, yield strength, ultimate tensile strength and % elongation to failure determined for IN718 tests are listed in Table 7.1.

Figure 7.2: Stress/strain curves for IN718 and CMSX-4 BMs.
Table 7.1: BM tensile properties for Inconel 718.

<table>
<thead>
<tr>
<th>Test #</th>
<th>YS [ksi]</th>
<th>UTS [ksi]</th>
<th>Modulus (E) [x10^6 psi]</th>
<th>% elongation to failure</th>
</tr>
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<tr>
<td>1</td>
<td>172.2</td>
<td>217.5</td>
<td>29.6</td>
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<td>2</td>
<td>169.5</td>
<td>215.3</td>
<td>30.9</td>
<td>18.5</td>
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7.3 Butt Joint – Tensile Tests

Six IN718/BNi-2 butt joint coupons were fabricated following AWS C3.2 procedures to measure the brazed joint tensile strength. Samples were cut from IN718 plates (0.25” thickness) and prepared for brazing using the same procedures listed in Chapter 5. Two layers of BNi-2 foil (0.001 inch thickness) were pre-placed in the joint area and laser tack welding was applied to the edges to fix the joint gap at 0.002-0.003 inch. Additional BNi-2 paste applied to the edge of the joint to ensure complete braze coverage. Vacuum furnace brazing was performed at Rolls-Royce using the standard brazing cycle for BNi-2 (Table 5.2) with a slightly longer hold time of 30 minutes at 1065°C (1950°F). Following brazing the full IN718 solution and age heat treatment (Figure 7.1) was applied for these samples. Additional machining was performed via wire EDM and surface grinding to reduce the gauge section and remove excess braze FM. The butt joint samples before and after machining are shown in Figure 7.3 with final dimensions listed in Appendix A.
Before testing the joint microstructures of the ‘cutout’ gauge section were analyzed via LOM. No visible porosity was observed within the joints and an average joint gap of 70 µm was measured. As shown in Figure 7.4, the IN718/BNi-2 joints consisted almost entirely of the solid solution ISZ (α-Ni) phase. Some intermetallic phases appear to be Ni$_3$B type borides were also present in some joints, however the volume fraction of centerline eutectic phases was very low (less than 0.1) for all joints tested. This was likely due to the smaller joint gaps and slightly longer hold time applied for these joints compared to the IN718/BNi-2 joint described in Chapter 5.
Similar to the BM samples, tensile testing of the IN718/BNi-2 butt joints was performed on the 810 Material Testing System (MTS) at a target strain rate of 0.001 in/in/s. Engineering stress/strain curves for IN718/BNi-2 butt joints are shown in Figure 7.5 with c/s area, peak load ($P_Q$) and fracture stress ($P_Q$ divided by c/s area) for each sample listed in Table 7.2. The average modulus measured was almost identical to the IN718 BM ranging between $29 \times 10^6$ and $34 \times 10^6$ psi. All joints failed through the brazed joint immediately at the peak load in a brittle-like fracture. No significant plastic deformation was observed as the max tensile stress measured of 161.7 ksi (14,150 kip) for sample Tensile5 never exceeded the BM yield stress. A relatively high average joint strength of 152.8 ksi nearly 90% of the BM yield strength was measured for these joints. However, the ductility of the braze joints is extremely low compared to the IN718 BM with the maximum strain less than 0.5% compared to 18-26% for the BM. As discussed in Chapter 2 this seems to indicate that ductility or toughness rather than strength is more of a concern when dealing with these types of high strength brazed joints.
Figure 7.5: Stress/strain curves for IN718/BNi-2 butt joint tensile tests.

Table 7.2: IN718/BNi-2 butt joint tensile properties at an average joint gap of 70 µm.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>c/s area (in²)</th>
<th>P₀ (lbf)</th>
<th>Fracture Stress (ksi)</th>
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</thead>
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<td>12,256</td>
<td>145.9</td>
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<td>Tensile2</td>
<td>0.0875</td>
<td>12,939</td>
<td>147.9</td>
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<td>Tensile3</td>
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<td>11,895</td>
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<td>0.0840</td>
<td>12,939</td>
<td>154.0</td>
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<td>Tensile5</td>
<td>0.0875</td>
<td>14,150</td>
<td>161.7</td>
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<td>Tensile6</td>
<td>0.0875</td>
<td>14,121</td>
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<td>13,050</td>
<td>152.8</td>
</tr>
<tr>
<td>Std. Dev.</td>
<td></td>
<td>933</td>
<td>7.4</td>
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7.4 Small-scale Tensile Testing of Brazed Joints

To study the local mechanical behaviour of brazed joint material and influence of microstructure on mechanical behaviour small-scale tensile testing of IN718/BNi-2 and CMSX-4/BNi-2 brazed joints was performed. Small-scale tensile testing has been increasingly used in materials science for studying small volumes of materials particularly in thermal barrier coating (TBC) systems or micro-/nano-electro mechanical systems (MEMS/NEMS) [141]. There are several advantages to using a similar technique for brazed joints. First, the gage section consists of a much higher volume fraction of the brazed joint compared to standard size test specimens. As a result, the true stress/strain behaviour of the braze interlayer can be determined providing actual material properties of the FM similar to the AWS D1.1 tensile test for weld metal. Since samples are cut from actual brazed joints the metallurgical interactions that occur during brazing can be captured that would otherwise be neglected in testing of bulk FMs. In addition, by reducing the sample size the tri-axial stress in the joint is lower, so that microstructure-property relationships can be evaluated relatively independently from a change in tri-axial stress. Finally, a LOM camera is used to observe the in-situ local deformation behaviour and crack initiation sites in the joint microstructure during the tests.

This is a relatively unique approach for studying brazed joints as no similar publications using small-scale tensile coupons were found in the literature. In this initial work, experimental procedures and test results were generated for several brazed joints with different levels of centreline eutectic ($f_e$). Developing this technique for brazed joints will improve understanding of brazed joint mechanical behaviour and allow for the
determination of local stress/strain properties that can be used as inputs for multi-scale FEA models.

7.4.1 Experimental Procedures

Seven brazed joints of CMSX-4 and IN718 BMs with BNi-2 FM were selected for small-scale tensile testing. The brazing parameters for each sample are listed in Table 7.3 including the type of atmosphere, average joint gap, brazing temperature and hold time. These parameters were selected to create a range of joint microstructures. Samples were extracted from each brazed joint using fine wire electrical discharge machining (EDM) with the braze joint centered in the gauge section. A schematic of the ‘dogbone’ and target dimensions are shown in Figure 7.6 with a gauge length of 0.6 mm and width of 0.3 mm. This shape was cut out across the length of the brazed joint and then slices approximately 0.250 mm thick were cut from the ‘dogbone’ cut to get the final small-scale test samples.

Table 7.3: Brazing parameters for small-scale tensile test samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th># of samples</th>
<th>BM</th>
<th>FM</th>
<th>Atmosphere (type)</th>
<th>Avg. Joint Gap (µm)</th>
<th>Brazing Temperature (°C)</th>
<th>Hold Time (min)</th>
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</thead>
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<tr>
<td>IN718A</td>
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<td>Inconel 718</td>
<td>BMNi-2</td>
<td>Vacuum</td>
<td>70</td>
<td>1065</td>
<td>30 min</td>
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<td>IN718B</td>
<td>3</td>
<td>Inconel 718</td>
<td>BMNi-2</td>
<td>Vacuum</td>
<td>75</td>
<td>1065</td>
<td>10 min</td>
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<tr>
<td>IN718C</td>
<td>3</td>
<td>CMSX-4</td>
<td>BMNi-2</td>
<td>Argon</td>
<td>150</td>
<td>1065</td>
<td>10 min</td>
</tr>
<tr>
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<td>CMSX-4</td>
<td>BMNi-2</td>
<td>Argon</td>
<td>270</td>
<td>1065</td>
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</tr>
<tr>
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<td>CMSX-4</td>
<td>BMNi-2</td>
<td>Argon</td>
<td>600</td>
<td>1065</td>
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<td>SX3</td>
<td>3</td>
<td>CMSX-4</td>
<td>BMNi-2</td>
<td>Argon</td>
<td>270</td>
<td>1065</td>
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<td>SX4</td>
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<td>CMSX-4</td>
<td>BMNi-2</td>
<td>Argon</td>
<td>600</td>
<td>1065</td>
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</table>
Figure 7.6: Basic dimensions of ‘dogbone’ shape used for small-scale tensile testing.

These steps are shown in Figure 7.7 for the SX1 samples which were extracted from one of the CMSX-4/BNi-2 lap joints used for metallurgical characterization in Chapter 5. SX2 and SX3 samples were cut from an angled joint specimen (Figure 6.2) for approximate joint gaps of 0.150 and 0.250 mm respectively. The SX4 samples consisted of all braze FM and was created by drilling a 0.8 mm diameter hole in the CMSX-4 plate then filling it with BNi-2 paste. IN718 samples were extracted from the ‘cutout’ gage section of the butt joints tested in the previous section with a joint gap of approximately 0.07 mm. A total of 40 samples were tested, the large number of SX1 samples was primarily used to develop the experimental procedures and refine the test setup.
Figure 7.7: Small-scale sample fabrication of SX1 samples from CMSX-4/BNi-2 brazed joint. ‘Dogbone’ cut was made first and then sliced to 0.25 mm thickness to get final small-scale samples.

Each small-scale tensile sample was examined via LOM prior to testing by cold mounting the specimens with a fast cure, clear acrylic powder/liquid mixture and carefully polishing the samples to a 1 µm finish after the acrylic had cured. Figure 7.8, shows LOM images of the typical joint microstructure for SX and IN718 small-scale tensile samples with the brazed joint material at the center of the gauge length. The average volume fraction eutectic, joint gap, sample width and surface roughness was measured with LOM by taking an average value from multiple measurements for each sample following the same methods described in Chapter 5. In addition, any defects observed within the joint over 10 µm were noted. As expected smaller joint gaps (SX1, IN718A, B and C samples) consisted primarily of the α-Ni whereas larger joint gaps
resulted in a high volume fraction of centerline eutectic phases. After imaging, acetone was used to dissolve the acrylic and extract the specimens to measure the thickness of the gauge section. The average final thickness was between 150-200 µm as shown in Figure 7.9.

Figure 7.8: LOM images of gauge length and typical joint microstructures prior to testing. From left to right, IN718 (A, B and C), SX1, SX2, SX3 and SX4 samples.

Figure 7.9: Thickness measurements by LOM of gauge section after polishing.

A small-scale test system originally developed at OSU for rapid measurement of single crystal materials was used for this study [142]. Shown in Figure 7.10, the setup is similar to conventional test frames with grips to hold the sample, a motor-driven screw.

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drive for precise displacement control and a Honeywell-Sensotec Model 31 load cell to measure the applied load. A PixeLINK microscope camera attached to the frame allows for in-situ microstructural observations and non-contact strain measurements across the gage section via 2D-DIC.

DIC is a non-contact optical technique for measurement of strain and displacement on a sample surface originally developed in the 1980’s for studying solid mechanics [143, 144]. In DIC, a random black and white speckle pattern is applied to the surface of a sample and sequential images of this surface are taken during testing. Post-test analysis is then performed using DIC software, which treats each image as a pixel array of grayscale values and divides the images into subsets consisting of a small group of pixels. Iterative algorithms are then used to track the location of each subset between images and calculate the relative displacement and rotation of each subset relative to the initial (reference) image. From this information full-field strain and displacement maps across the samples surface during testing can be generated [145].

The small-scale tensile samples were tested in both the polished condition and with a speckle pattern applied to the polished surface for DIC consisting of white paint and black printer toner. Images were taken at 1 fps during testing and NCorr software an open-source MATLAB based 2D-DIC code developed at Georgia Institute of Technology was used for DIC analysis of the images [146]. Samples were tested at room temperature with a displacement rate of 0.25 μm/s and the peak load was recorded for each sample.
Figure 7.10: Small-scale experimental tensile test setup at OSU with LOM camera, load cell and grips to hold/pull sample.

According to Kashaev et al. for samples of this size it is necessary to account for the surface roughness, therefore a modified correction factor according to the effective load-bearing cross-section, $A_{eff}$, was used to calculate the engineering stress [147]. For this study, since one side in the width direction was polished $A_{eff}$ was calculated as:

$$A_{eff} = (S - 2R_z) \times (W - R_z)$$  \hspace{1cm} (7.1)

where $S$ is the thickness, $W$ is the width and $R_z$ is the average maximum height of the surface roughness profile measured. $R_z$ ranged from 2-8 μm as a result of the EDM cutting process and was measured prior to testing via LOM. To validate the test
procedure small-scale CMSX-4 BM samples were also tested and showed similar stress/strain properties as the standard size samples at the same orientation.

7.4.2 Small-scale Joint Deformation Behavior

A summary of the small-scale brazed joint test results are reported in Table 7.4 including the measured joint gap, \( A_{\text{eff}} \), max force and calculated ultimate tensile stress (UTS). The UTS was calculated as the peak load divided by \( A_{\text{eff}} \). Sequential images of the joint microstructure during testing provide the first real-time observations of brazed joint failure behaviour as shown in Figure 7.11 for selected SX1 and IN718A braze joints corresponding to samples #10 (top), #19 (middle), and #31 (bottom). In these images the applied stress is increased from left to right, with the sample at the right being the last image before failure in the brazed joints. These joints sustained high peak loads and plastic deformation occurred in the BM away from the joint as indicated by the appearance of slip bands in both the single crystal and poly-crystalline superalloy material.
<table>
<thead>
<tr>
<th>BM</th>
<th>Sample</th>
<th>Test #</th>
<th>Joint gap [µm]</th>
<th>$A_{\text{eff}}$ [mm$^2$]</th>
<th>Max Force [lbs]</th>
<th>UTS [ksi]</th>
<th>Defect (&gt;10 µm)</th>
</tr>
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<td>80</td>
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<td>154.46</td>
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</table>
This was an unexpected result as the joint material is typically expected to be the weakest member and as shown in chapter 5 doesn’t contain the same precipitate strengthening elements (Al, Ti or Nb) as the superalloy BMs. In addition, the standard size butt joints showed no plastic deformation in the BM. For these joints, plastic deformation (slip bands) was also observed in the joint material as well and as the applied stress continued to increase crack initiation was observed around the intermetallic phases or defects (sample #19) present in the joint. Cracks continued to propagate and bridge around these phases in the joint until failure occurred shown in the final images on the right in Figure 7.11. This deformation behaviour was only observed in joints with relatively low amounts of centreline eutectic phases. For SX2, SX3 and SX4 braze joint samples with higher $f_c$ no plastic deformation occurred in the BM or FM and a brittle fracture with unstable crack growth occurred immediately at the peak load.
Figure 7.11: Sequential images of SX1 braze joints (top and middle) and IN718A braze joint (IN718/BNi-2) during small-scale tensile testing.

From these qualitative observations the following failure mechanism is proposed for joints containing a low volume fraction of centreline eutectic. First, any remaining
intermetallic phases within the joint that were found to have extremely high hardness will result in strain localization under an applied stress. This leads to plastic deformation in the surrounding softer matrix and crack initiation at the interface between the matrix and these particles. As the stress level continues to increase these cracks propagate through the matrix or bridge with neighbouring cracks that have formed in a similar manner until they reach a critical size at which point failure occurs.

Figure 7.12 shows a closer look of this failure mechanism for one of the IN718B braze joints. Just before failure (right image) plastic deformation in the form of slip bands can be observed surrounding the hard Ni$_3$B or CrB intermetallic phases in the joint as a result of the strain localization between these and the eutectic matrix. This local plastic zone in the solid-solution matrix surrounding these hard phases was also observed by Philips et al. who performed post-test analysis of fracture mechanics testing with a similar FM [116]. However, this phenomenon has never been observed experimentally.
Figure 7.12: Strain localization at intermetallic phases within the braze joint observed at higher magnification of a IN718B test sample during loading.

7.4.3 Microstructure-Property Relationships

Figure 7.13, shows the effect of joint gap on the UTS for all samples tested in this study with IN718 tests plotted in blue and CMSX-4 tests in orange. Samples with defects are marked with a half filled circle and the measured BM yield strength (YS) are also listed for reference. While there was some scatter in the data, the increase in joint gap i.e. volume fraction of centreline eutectic significantly reduced the UTS. For defect-free samples, joint gaps less than 85 µm exceeded the yield strength of the BM with the UTS ranging from 134 ksi to 157 ksi for CMSX-4 joints and 150 ksi to199 ksi for IN718 joints. These results also show the influence of BM on UTS as the higher yield strength and increased diffusion in poly-crystalline IN718 resulted in higher UTS compared to the CMSX-4 joints. As the joint gap is increased for the SX2, SX3 and SX4 samples the UTS
decreases significantly reaching a minimum for the SX4 samples at an average of 41 ksi where the gauge section consists of entirely eutectic FM constituents.

![Graph showing tensile strength vs. joint gap for brazed joint material](image)

**Figure 7.13**: Tensile strength of brazed joint vs. joint gap for all small-scale samples tested.

Stress/strain curves were generated for the brazed joint material using 2D-DIC analysis of speckled samples to measure displacement/strain between selected points on the sample’s surface similar to the approach used by Leinenbach et al. [79]. An example of the DIC setup is shown in Figure 7.14 for one of the speckled IN718A samples (#31), with several extensometers setup corresponding to the joint region and several corresponding to the IN718 BM. Samples were marked to align the LOM images taken
before testing with the speckle pattern to ensure DIC analysis was being performed over the joint region.

Figure 7.14: Example of DIC setup for strain analysis of FM and BM regions in IN718/BNi-2 braze joint used to generate stress/strain curves.

From this setup, average engineering stress/strain curves were generated for the BM and FM regions as shown in Figure 7.15 for the IN718A (#31) sample using the average calculated strain from DIC in the direction of the applied load. Results indicate relatively similar properties (elastic modulus, yield strength) between the brazed joint and BM regions. A slightly higher elastic modulus and lower elongation to failure was observed in the joint region, likely due to the presence of brittle phases within the joint. However, other FMs such as the Au-18Ni FM investigated by Leinenbach et al. or the Ag- and Au-based FMs have been shown to exhibit significantly different stress/strain properties from the BM [98, 79].
Using this method, the elongation to failure (a good measure of ductility) of the BNi-2 joint material was determined for the other speckled braze joints tested and is plotted vs. joint gap in Figure 7.16. As expected, wider joint gaps above 80 microns resulted in very low ductility (less than 0.7%) compared to narrow joint gaps with low volume fractions of eutectic constituents which ranged from 2.5 to 5.0 percent elongation. Measurements of elastic modulus using this technique were difficult to ascertain primarily due to the local changes in microstructure between samples that occur on this small-scale. This was also true for the BM, especially for the poly-crystalline IN718, where the gauge length (~0.3 mm) only consisted of several grains and their relative orientation was not determined prior to testing. As a result, there was some
variation in the measured elastic modulus between samples ranging from 20 to 35 x10^6 psi.

![Graph showing elongation to failure versus joint gap](image)

Figure 7.16: Elongation to failure measured from DIC analysis for all brazed joints tested.

### 7.4.4 Conclusions

Several conclusions were made from this initial study on small-scale tensile testing of Ni-base superalloy brazed joints. First, a failure mechanism in joints with low $f_c$ was identified through in-situ observations of the joint microstructure during loading. Second, stress/strain behaviour of the actual joint material was directly measured using this technique with DIC analysis. By extracting small-scale sample from actual brazed joints the BM/FM metallurgical interactions that occur during brazing are captured and tri-axial stress that develop in standard testing procedures is reduced. Using this
technique the stress/strain behaviour of the joint material can be estimated for FEA modelling. These properties may be especially valuable in modelling thin-walled brazed joints such as honeycomb structures where the BM thickness is approximately the same size as the joint gap and the tri-axial stress is low.

Finally, the influence of centreline eutectic on joint strength and ductility was evaluated by testing brazed joints at various joint gaps. Increasing the volume fraction of centreline eutectic reduces both the ductility and ultimate tensile strength of the joint. Since the tri-axial stress was low in these studies, it would appear the presence of eutectic phases is the more dominating factor for the reduced strength observed with increasing joint gap for Ni-base superalloy brazed joints as suggested by Lugschieder et al. [50]. These results clearly demonstrate the need for incorporating microstructure-property relationships into the multi-scale model.

### 7.5 Lap Shear Testing with Digital Image Correlation

As discussed in Chapter 3, the standard lap shear test method produces FM shear stress values that decrease exponentially with overlap length making it difficult to recommend a safe stress level from a design standpoint. Eqn. 3.1 from AWS C3.2 assumes the stress is uniformly distributed across the joint, but it has been well established that this is not the case due to the peel stresses and stress concentrations that develop during shear-tensile loading [95, 90]. The damage zone models proposed by Flom et al. based on FEA modeling for predicting lap shear failure loads showed good agreement with experimental testing of 347 SS lap joints with a pure Ag brazing FM [97]. However, no attempts have been made to experimentally validate or measure the
damage zone for brazed joints and the use of FEA requires accurate BM/FM material properties that are typically not readily available.

To validate the damage zone model approach for brazed joints and assess the average FM shear stress per AWS C3.2, lap shear testing of CMSX-4/BNi-2 and IN718/BNi-2 brazed joints was performed in this section using DIC to quantify strain across the joints. FEA models of the lap shear test specimens were also generated to calculate maximum principal and shear strains for comparison with the DIC results. Based on the FEA and DIC results, recommendations for applying damage zone models to lap shear brazed joints are provided.

7.5.1 Experimental Procedures

Following AWS C3.2 procedures two sets of lap shear test specimens were fabricated from CMSX-4 and IN718 plates with a BM plate thickness (T) of 0.120 inch for CMSX-4 and 0.200 inch for IN718. For CMSX-4 overlap lengths of 1T, 2T, 3T, 4T and 5T were tested with three samples at each overlap. For IN718, overlap lengths of 1T, 2T, 3T and 5T were tested with 3-4 samples at each overlap. To compare braze joint properties with BM properties additional IN718 lap shear samples consisting of all BM (no braze joint) were fabricated. These were machined to replicate overlaps of 0.5T, 1T and 2T (T=0.200 inch) with a small radius (~0.005 inch) in the fillet region. Drawings with dimensions of the lap shear test specimens are provided in Appendix A.

The same brazing procedures used for butt joint testing were applied for lap shear test specimens with two layers of BNi-2 amorphous foil (0.001 inch thickness) pre-placed in the joint area. Laser tack welding was used to maintain the target overlap length and
joint gaps of 0.002-0.003 inch. Additional BNi-2 paste (binder and powder) was applied to the fillet regions to ensure complete gap filling. Standard BNi-2 brazing cycle (Table 5.2) and post-braze solution and aging heat treatments for CMSX-4 and IN718 were carried out under high vacuum at Rolls-Royce facilities. Figure 7.17 shows an image of the CMSX-4 (top) and IN718 (bottom) test specimens after brazing and heat treatment.

As shown, the IN718/BNi-2 and IN718 BM samples were fabricated with a hole for pin loading. To maintain similar loading conditions for CMSX-4/BNi-2 samples shims (0.120” thickness) were placed at both ends in the grip sections during testing. Excessive FM was used for the IN718/BNi-2 braze joints resulting in large fillets for these samples with an average radius of 0.1 inch compared to the 0.005 inch radius with the CMSX-4/BNi-2 lap joints and IN718 BM. As a result, two IN718/BNi-2 lap joint samples at each overlap went through an additional milling step to remove the fillet. However, the presence of a fillet had very little effect on test results.
Figure 7.17: CMSX-4 (top) and IN718 (bottom) lap shear test specimens after brazing and heat treatment.

Lap shear specimens were tested using the 810 Material Testing System (MTS) at OSU in accordance with AWS C3.2 specifications at a displacement rate of 0.001 in/s. Force vs. crosshead displacement curves were recorded for each sample and sequential images of the overlap were taken for DIC during testing. For the CMSX-4/BNi-2 lap joints a VIC-3D DIC system from Correlated Solutions with two Point Grey GRAS-
20S4M-C cameras was utilized. For IN718/BNi-2 and the IN718 BM samples, a simple 2D-DIC system was developed using high resolution Nikon D7100 camera to acquire images during testing and NCorr software was used for the DIC analysis. Samples were speckled for DIC using flat white and black Rust-oleum™ spray paint by applying a white base layer and then misting the black paint to create a fine speckle pattern. The 3D-DIC and 2D-DIC setup are shown for the CMSX-4/BNi-2 (left) and IN718/BNi-2 lap joints (right) respectively in Figure 7.18.

Figure 7.18: Test setup for lap shear testing with cameras for 3D-DIC (left) and 2D-DIC (right).

Typical joint microstructures for the CMSX-4 and IN718 lap joints are shown in Figure 7.19. Similar to the joint microstructures characterized in chapter 5 with BNi-2 foil, these joints contained relatively low amounts of brittle centerline eutectic phases as
the same brazing cycle was used. However, the fillet regions consisted the eutectic micro-
constituents similar to those shown in Figure 5.9.

Figure 7.19: Typical joint microstructure of CMSX-4/BNi-2 (left) and IN718/BNi-2 (right) lap shear brazed joints.

7.5.2 Lap shear test results

Experimental results from lap shear testing of these joints are listed in Table 7.5 and Table 7.6 for CMSX-4/BNi-2 and IN718/BNi-2 braze joints respectively. The measured peak load, overlap, joint area and BM cross-sectional area were used to calculate the average FM shear stress and BM tensile stress from Eqns 3.1 and 3.2. Force vs. displacement curves for each test are also provided in Appendix B.

Following the AWS C3.2 procedure, FM shear stress and BM tensile stress were plotted versus the joint overlap from these test results in Figure 7.20 and Figure 7.21 for CMSX-4/BNi-2 and IN718/BNi-2 joints respectively.
Table 7.5: Lap shear test results for CMSX-4/BNi-2 brazed joints.

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Table 7.6: Lap shear test results for IN718/BNi-2 brazed joints.

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All braze joint samples failed in a brittle-like fracture through the BNi-2 braze joints except for one of the CMSX-4/BNi-2 test coupons at a 4T overlap (shaded in
which failed in the CMSX-4 BM at a BM tensile stress of 83.24 ksi. It is unclear exactly why this sample sustained such a high peak load relative to the other 4T samples. As expected, increasing the overlap led to high peak loads and average BM tensile stress, however the average FM shear stress decreased significantly. For CMSX-4 the calculated FM shear stress ranged from a max of 45.28 ksi for the 1T overlap to 14.9 ksi at a minimum of 15.65 ksi for the 5T overlap. For the IN718/BNi-2 braze joints the FM shear stress decreased from 74.51 ksi at 1T to 20.47 ksi. Similar to the small-scale testing the BM appears to have a strong influence on shear strength with peak loads for the CMSX-4/BNi-2 braze joints at about 65-70% of the peak load for IN718/BNi-2 joints at the same overlap.

Figure 7.20) which failed in the CMSX-4 BM at a BM tensile stress of 83.24 ksi. It is unclear exactly why this sample sustained such a high peak load relative to the other 4T samples. As expected, increasing the overlap led to high peak loads and average BM tensile stress, however the average FM shear stress decreased significantly. For CMSX-4 the calculated FM shear stress ranged from a max of 45.28 ksi for the 1T overlap to 14.9 ksi at a minimum of 15.65 ksi for the 5T overlap. For the IN718/BNi-2 braze joints the FM shear stress decreased from 74.51 ksi at 1T to 20.47 ksi. Similar to the small-scale testing the BM appears to have a strong influence on shear strength with peak loads for the CMSX-4/BNi-2 braze joints at about 65-70% of the peak load for IN718/BNi-2 joints at the same overlap.

Figure 7.20: AWS C3.2 plot for CMSX-4/BNi-2 lap shear tests.
The lap shear test results for the IN718 BM samples are listed in Table 7.7 and plotted in Figure 7.22. Only the 0.5T and 1T BM samples failed in shear through the overlap region while the 2T overlap failed in tension away from the overlap at a BM tensile stress of 147.8 ksi. Similar to the braze joints the calculate shear stress decreases with overlap due to the inherent lap shear geometry. However, the BM samples exhibited significantly higher strength and ductility than the braze joints. For comparison, the 0.5T BM sample failed at about the same load as the 1T braze joint sample and the 1T BM sample failed around the same load as the 5T braze joint sample. Additional analysis using FEA and DIC will be used to compare the BM and braze joint lap shear results.
Table 7.7: Lap shear test results for IN718 BM samples.

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<th>Peak Load (lbf)</th>
<th>Joint area (in²)</th>
<th>BM c/s area (in²)</th>
<th>FM Shear Stress (ksi)</th>
<th>BM Tensile Stress (ksi)</th>
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Figure 7.22: AWS C3.2 plot for IN718 BM lap shear samples.

One outcome from the lap shear testing that is not been frequently reported in the literature is that the tensile stress required for BM failures is lower than the yield strength or UTS of the BM. For CMSX-4 the 4T overlap sample failed in the BM at a BM tensile stress of 83.3 ksi and from IN718 BM samples the 2T overlap failed in the BM region at
147.8 ksi even though the UTS of these materials (Table 7.1) were approximately 160 ksi and 215 ksi for CMSX-4 and IN718 correspondingly. This effect is also a result of stress concentrations near the edge of the overlap that develop in the BM region.

The influence of fillets at the edge of the joint on lap shear brazed joint properties is not well understood. From a design standpoint, fillets reduce the stress concentration at the edge of the overlap however they consist almost entirely of the brittle, eutectic FM microstructure. In this study, fillets were found to have very little effect on the failure load (Table 7.6) for the IN718/BNi-2 samples. However, they did result in pre-mature cracking in the fillet region. These cracks are shown in Figure 7.23 for the 2TA IN718/BNi-2 brazed joint and appear as ‘pop-ins’ on the force vs. displacement curves in Appendix B at about 85% of the breaking load. Cracking was not observed in the internal joint area however until the peak load was reached. While the failure load was not influenced by these cracks, once the cracks have formed they could easily propagate into the brazed joint with additional fatigue loading or thermal cycling.
Figure 7.23: Images of cracking in the fillet regions of IN718/BNi-2 brazed joints that occurred at approximately 85% of the failure load during lap shear testing.

7.5.3 Digital Image Correlation – Strain Analysis

Figure 7.24 shows two selected 3D-DIC results for the shear strain ($\gamma_{xy}$) measured across CMSX-4/BNi-2 lap joints at overlaps of 1T and 4T just before failure corresponding to a BM tensile stress of 49 ksi (339 MPa) and 71.1 (490 MPa), respectively. 2D-DIC analysis of IN718/BNi-2 brazed joints produced similar plots provided in Appendix B for $\gamma_{xy}$ and normal strains ($E_{xx}$, $E_{yy}$). From these full-field strain maps the stress concentrations and effective damage zones that form at the edge of the overlap sections during loading are clearly visible.
Figure 7.24: 3D-DIC results for CMSX-4/BNi-2 lap joints with shear strain ($\gamma_{xy}$) mapping across overlap region for 1T, 3T and 4T samples at failure load.

At short overlaps, such as the 1T overlap, strains were relatively uniform across the joint. However, as expected for larger overlaps (above 1T) the strain distribution becomes concentrated near the edge of the overlap with very little deformation in the middle of the overlap. This indicates that the applied load is carried mostly by the edge regions and not the full overlap as calculated with Eqn. 3.1. In addition, the deformation is not restricted to the FM with relatively high shear and normal strains also observed in the BM. The brazed joint behavior is quite different from that reported by Stuparu et al. for adhesive bonding lap joints in which all of the strain occurs within the adhesive layer and the adherents remain rigid [148]. Due to the similar mechanical properties between
the BM and FM the brazed joints behaved more like a bulk material rather than an adhesive/adherent system. However, this may depend on the BM/FM combination used though as soft, ductile FMs may exhibit more plastic deformation similar to adhesive bonds under shear loading. Strain concentrations in the BM region adjacent to the joint also explains why the necessary BM stress required for BM failure is well below the yield strength of the BM in the lap joint configuration.

Strain along the brazed joint were further analyzed to quantify the critical damage zone size using a line mapping tool. An example of this is shown by the white line for a CMSX-4/BNi-2 brazed joint at an overlap of 3T in Figure 7.25. For each sample, similar lines were setup across the brazed joint and strain values were extracted along these lines. This is the experimental equivalent of the line path Flom et al. used to extract von Mises stress along the bond line of FEA models [97]. The average strain values across 2-3 lines in the DIC analysis were used to generate the plots listed in Appendix B for the IN718/BNi-2 samples the calculated strains ($E_{xx}$, $E_{yy}$, $\gamma_{xy}$) across the overlap distance at the corresponding failure load listed in Table 7.6. An example of the calculated $E_{xx}$, $E_{yy}$, and $\gamma_{xy}$ strains calculated for the 3TC IN718/BNi-2 brazed joint sample is shown in Figure 7.26 at a failure load of 5.976 kip. The plots in Appendix B show relatively consistent strain values between samples for each overlap at the failure load. As expected, the highest strain was in the direction of the applied load ($E_{yy}$) however $E_{xx}$ and $\gamma_{xy}$ can also be relatively high toward the edge of the joint. As a result, a failure criteria that accounts for the combined effect of these strain values should be applied to determine the critical size of the damage zone.
Figure 7.25: DIC calculated shear strain ($\gamma_{xy}$) for CMSX-4/BNi-2 sample 3Tb at applied load of 6.260 kip showing line mapping tool used to extract strains along brazed joint.

3T overlap
66.9 ksi (461 MPa)
Figure 7.26: 2D-DIC calculations for normal ($E_{xx}$, $E_{yy}$) and shear ($\gamma_{xy}$) strains across the IN718/BNi-2 brazed joint for the 3TC sample at an applied load of 9.453 kip.

For the soft, ductile Ag-based FMs investigated by Flom et al. a yield criteria like the von Mises equivalent stress was appropriate to use to define the stress level for the damage zone since these FMs can show significant plastic deformation. However, this criteria is less applicable for the BNi-2 FM and other nickel-based FMs that produce brittle joints with limited strain to failure. Therefore a maximum principal strain damage zone criteria was utilized instead with the maximum principal strain ($\varepsilon_{\text{max}}$) calculated directly from the average $\tau_{xy}$, $\varepsilon_{xx}$, and $\varepsilon_{yy}$ measurements as [149]:

$$
\varepsilon_{\text{max}} = \frac{\varepsilon_x + \varepsilon_y}{2} + \sqrt{\left(\frac{\varepsilon_x - \varepsilon_y}{2}\right)^2 + \left(\frac{\tau_{xy}}{2}\right)^2} \quad (7.2)
$$
Using Eqn. 7.2 the $\varepsilon_{\text{max}}$ was calculated across the bond line for all samples tested with each plot provided in Appendix B. Similar to the approach used by Flom et al. [89], the average $\varepsilon_{\text{max}}$ at each overlap is plotted across the normalized overlap distance or the distance along the bond line divided by the overlap length for the CMSX-4/BNi-2 and IN718/BNi-2 braze joints in Figure 7.27 and Figure 7.28 respectively.

The CMSX-4/BNi-2 brazed joints showed significantly lower strain values at failure compared to the IN718/BNi-2 joints due to the lower peak loads during testing. This indicates that the failure criteria for defining the damage zone should be assessed on a joint to joint basis. There was also significantly more scatter in the CMSX-4/BNi-2 test results. This could be attributed to the tack welds, excess braze FM or misalignment at the edge of the overlap. For the IN718/BNi-2 post-braze machining was applied to create a smooth edge surface and resulted in much better DIC results. In both joints, $\varepsilon_{\text{max}}$ is relatively uniform across the brazed joint for the 1T overlap (blue line) with a slight decrease at the center of the overlap. However, for larger overlaps $\varepsilon_{\text{max}}$ values at the edge of the overlap were very high over 4% for some of the IN718/BNi-2 joints, whereas the center of the overlap had values less than 0.2%. Based on small-scale testing strains above 0.6-1% strain indicate the onset of plastic deformation.

These normalized overlap plots are similar to the FEA modeling results reported by Flom et al. for 347 SS brazed joints with the pure Ag FM. However, there doesn’t appear to be a clearly defined damage zone size where the strain values converge at a certain distance from the edge of the overlap especially for the IN718/BNi-2 joints and the damage zone seems to decrease with increasing overlap.
Figure 7.27: Average maximum principal strain along the normalized overlap distance for CMSX-4/BNi-2 brazed joints calculated using DIC analysis for 1T-5T overlaps.

Figure 7.28: Average maximum principal strain along the normalized overlap distance for IN718/BNi-2 brazed joints calculated using DIC analysis for 1T, 2T, 3T and 5T overlaps.
Rather than looking at the normalized distance, plots were also generated using the actual distance from one edge of the overlap (up to 0.2 inch) in Figure 7.29 and Figure 7.30 for the CMSX-4 and IN718 joints correspondingly. In these plots the calculated $\varepsilon_{\text{max}}$ appears to show a better convergence at a $\varepsilon_{\text{max}}$ of approximately 0.5% for the CMSX-4/BNi-2 joints and 1.2% for the IN718/BNi-2 joints. Therefore, these $\varepsilon_{\text{max}}$ values were used as the failure criteria to define the damage zone for each joint in the DIC analysis and are indicated by the horizontal dashed line on the plots in Figure 7.27 - Figure 7.30. The distance at which the calculated $\varepsilon_{\text{max}}$ is equal to this value defines the size of the damage zone, for example the measured damage zone for the 2T overlap (X) is indicated by the vertical dashed lines for one side of the overlap in each plot. Similarly, the average damage zone size was measured for the other overlaps using these plots and listed in Table 7.8 for the CMSX-4/BNi-2 and IN718/BNi-2 braze joints. The average experimental damage zone size for the CMSX-4/BNi-2 lap joints (at 0.5% $\varepsilon_{\text{max}}$) was found to be approximately 0.036 inch or a normalized distance of 0.111. For the IN718/BNi-2 joints the average damage zone size (at 1.2% $\varepsilon_{\text{max}}$) was found to be 0.054 inch or a normalized distance of 0.135.
Figure 7.29: Average maximum principal strain across the actual overlap distance for CMSX-4/BNi-2 brazed joints calculated using 3D-DIC analysis for 1T-5T overlaps.

Figure 7.30: Average maximum principal strain across the actual overlap distance for IN718/BNi-2 brazed joints calculated using 2D-DIC analysis for 1T-5T overlaps.
Table 7.8: Damage zone assessment of CMSX-4/BNi-2 and IN718/BNi-2 brazed joints from DIC analysis of lap shear brazed joints.

<table>
<thead>
<tr>
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<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>CMSX4/BNi-2 (ε_{max} = 0.5%)</td>
<td>0.125</td>
<td>4.628</td>
<td>0.187</td>
<td>0.023</td>
<td>0.0042</td>
</tr>
<tr>
<td></td>
<td>0.250</td>
<td>6.222</td>
<td>0.127</td>
<td>0.032</td>
<td>0.0083</td>
</tr>
<tr>
<td></td>
<td>0.375</td>
<td>6.236</td>
<td>0.073</td>
<td>0.027</td>
<td>0.0076</td>
</tr>
<tr>
<td></td>
<td>0.500</td>
<td>6.646</td>
<td>0.067</td>
<td>0.034</td>
<td>0.0087</td>
</tr>
<tr>
<td></td>
<td>0.625</td>
<td>7.238</td>
<td>0.100</td>
<td>0.063</td>
<td>0.0154</td>
</tr>
<tr>
<td>Average:</td>
<td></td>
<td></td>
<td>0.111</td>
<td>0.036</td>
<td>0.0088</td>
</tr>
<tr>
<td>IN718/BNi-2 (ε_{max} = 1.2%)</td>
<td>0.20</td>
<td>7.034</td>
<td>0.250</td>
<td>0.050</td>
<td>0.0039</td>
</tr>
<tr>
<td></td>
<td>0.40</td>
<td>8.950</td>
<td>0.166</td>
<td>0.066</td>
<td>0.0056</td>
</tr>
<tr>
<td></td>
<td>0.60</td>
<td>9.232</td>
<td>0.068</td>
<td>0.041</td>
<td>0.0102</td>
</tr>
<tr>
<td></td>
<td>1.00</td>
<td>11.397</td>
<td>0.055</td>
<td>0.055</td>
<td>0.0112</td>
</tr>
<tr>
<td>Average:</td>
<td></td>
<td></td>
<td>0.135</td>
<td>0.053</td>
<td>0.0077</td>
</tr>
</tbody>
</table>

From these results, the damage zone size appears to be independent of the overlap width as the normalized distance decreased with increasing overlap in both joints except for the 5T overlap for the CMSX-4/BNi-2 joints. The larger damage zone size measured for the 5T sample may have been due to cracks forming at the edge of the overlap prior to the failure load resulting in deceptively high strains in this region. These results suggest the actual damage zone length gives a more consistent failure criteria than the normalized damage zone length as described by Flom et al. for these joints.

DIC analysis was also performed across the BM IN718 lap shear test samples. The top image in Figure 7.31, shows the calculated E_{xy} and γ_{xy} strain maps at the failure load for the 1T overlap IN718 BM sample (bottom) and the IN718/BNi-2 braze joint (top) for comparison. As expected, the BM sample withstood a much higher load and strains to failure relative to the brazed joints. However, the geometry still resulted in
similar strain distribution across the joint with strain concentrations at the edge of the overlap and in the BM region. At the failure load of 11,650 kip for the BM 1T overlap sample the calculated $\varepsilon_{\text{max}}$ ranged from 6-9% across the overlap and failure occurred in shear through this region.

![Strain maps](image)

**Figure 7.31:** Normal ($E_{yy}$) and shear ($\gamma_{xy}$) strain maps from DIC of lap shear IN718/BNi-2 brazed joints (top) and IN718 BM (bottom) for 1T overlap just before failure.
For a more direct comparison of the BM and braze joint, the strain distribution at the same applied load was analyzed using the DIC line mapping tool for the 1T and 2T overlap samples. Based on the force/displacement curves for the BM samples, the image corresponding to the approximate joint failure load of 70.3 kip for the 1T overlap and 89.5 kip for the 2T overlap was analyzed to calculate $\varepsilon_{\text{max}}$ across the overlap for the BM samples. These results are plotted across the normalized overlap distance in Figure 7.32.

As shown, there is very little difference between the strain behavior in the BM (blue lines) and braze joint (black lines) samples for the same load and overlap. In addition, the apparent damage zone size appears to be almost identical between the BM and braze joint. This indicates the brazed joint exhibits BM deformation behavior up until failure at which point fracture occurs through the brazed joint.

Figure 7.32: Comparison of DIC strain ($\varepsilon_{\text{max}}$) distribution across overlap between IN718 BM lap shear samples (black) and IN718/BNi-2 braze joint (blue) lap shear samples at 1T and 2T overlaps.
7.5.4 Finite Element Analysis of Lap Joints

To validate the DIC analysis FEA models of the IN718/BNi-2 lap shear test specimens were generated for stress-strain analysis. Experimental failure loads from lap shear testing were applied to the FEA models to calculate the strains across the brazed joint for a direct comparison with the experimental DIC strains. The FEA model setup, results and discussion are provided in this section.

2D elastic/plastic FEA models of the IN718/BNi-2 lap shear test specimens were created in ABAQUS CAE using a standard/explicit model at each overlap (1T-5T) with dimensions matching the IN718/BNi-2 test specimens. For these models a perfect joint with homogeneous properties and a joint gap of 0.004” was assumed. Two sections were created for the joint and BM regions. Small fillets with a radius of 0.004” were also included at both edges of the braze region. Since the FM properties showed relatively similar stress/strain behavior as the BM for joints with low $f_c$ from small-scale tensile tests and lap shear DIC results indicated similar strain distributions between the BM and brazed joint samples for the same load, the BM properties for IN718 assessed in Section 7.1 were assigned to both the joint and BM regions. An average elastic modulus of 30.8x10⁶, yield strength of 171 ksi, Poisson’s ratio of 0.29 and plastic strain curve from the tensile tests were used as material property inputs in the elastic/plastic analysis.

Boundary conditions were selected to match the experimental testing conditions with one end pinned and the experimental load applied in the y-direction at the other end keeping the x-displacement of this end fixed at zero. A refined mesh adopted from Broughton et al. [95] was setup by partitioning in the joint and fillet regions as shown in
Figure 7.33 for the 3T overlap model to account for the stress concentrations observed in these areas. A mesh size of 0.001” was used in the joint region. The average experimental BM tensile stress at failure for the IN718/BNi-2 brazed joints at each overlap (listed Table 7.9) was applied to the corresponding FEA model.

![Mesh used for lap shear FEA models in ABAQUS CAE.](image)

Table 7.9: Average experimental failure loads applied to lap shear FEA models.

<table>
<thead>
<tr>
<th>Overlap (T)</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Applied Stress for FEA (ksi)</td>
<td>70.35</td>
<td>89.51</td>
<td>92.32</td>
<td>113.96</td>
</tr>
</tbody>
</table>

Results for the FEA calculated max principal strains for the 1T, 3T and 5T models are shown in Figure 7.34. The contour plots the maximum $\varepsilon_{\text{max}}$ was set to 1.2% as this was the failure criteria established to define the damage zone for the IN718/BNi-2 joints. As shown, these plots show the non-uniform stress distribution and damage zone for the
lap shear geometry and similar behavior to the normal strain \((E_{yy})\) DIC results for the same overlaps in Appendix B. To compare the FEA calculated \(\varepsilon_{\text{max}}\) along the brazed joint with the DIC calculated strains a path was setup for each model through the brazed joint region shown by the red line for the 3T overlap in Figure 7.34.

Figure 7.34: FEA models of 1T, 3T and 5T brazed joints at the applied failure load showing the effective damage zone at the edge of the overlap and location of path for extracting \(\varepsilon_{\text{max}}\).

The plots generated for the FEA \(\varepsilon_{\text{max}}\) calculated across the normalized overlap distance for the 1T, 2T, 3T and 5T overlap models are shown in Figure 7.35 at the experimental failure load. These plots indicate similar results to the DIC analysis in that the normalized damage zone distance appears to decrease with increasing joint gap. The \(\varepsilon_{\text{max}}\) strain distribution for the actual overlap distance from 0 to 0.2 inch shown in Figure 7.35 appears to be less dependent on joint gap. Based on the 1.2% failure criteria the FEA calculated damage zone size was smaller than the experimentally observed damage zone with an average for all overlaps of 0.20 inch compared to the 0.52 inch from DIC. One
A possible explanation for the larger damage zone size in the DIC analysis is micro-cracking that may have occurred in the fillet region near the edge of the overlap before the peak load was reached. This cracking was audibly heard during the testing of most joints and would lead to an increase in the $\varepsilon_{\text{max}}$ measured with DIC. Another discrepancy between the FEA models and the DIC results is the $\varepsilon_{\text{max}}$ calculated for the 1T overlap which was found to be much less uniform with FEA than the DIC results.

Figure 7.35: FEA calculated $\varepsilon_{\text{max}}$ vs. normalized overlap distance at the average failure loads for IN718/BNi-2 brazed joints with overlaps of 1T, 2T, 3T and 5T.
Figure 7.36: FEA calculated $\varepsilon_{\text{max}}$ vs. actual overlap distance at the average failure loads for IN718/BNi-2 brazed joints with overlaps of 1T, 2T, 3T and 5T.

Despite these discrepancies FEA can be utilized to predict the failure load for a range of overlaps based on experimental results from just one overlap using the DZM approach. For example, if only the 2T overlap joint samples were tested the average failure load of 8.95 kip would have resulted in an average FM shear stress of 44.75 ksi and average BM tensile stress of 89.5 ksi (Table 7.6). From the FEA model this would produce a damage zone size of 0.021 inch assuming a 1.2% $\varepsilon_{\text{max}}$ failure criteria as shown in Figure 7.36. Based on this calculated damage zone size, the applied load in FEA models for the other overlaps of interest would be adjusted to get a damage zone size equal to 0.021 inch as shown in Figure 7.37 for the 1T, 3T and 5T overlap models. By adjusting the applied load (listed in the legend) the size of the damage zone size at 1.2% strain is equal to 0.021 inch for all models.
The DZM approach provides a much more accurate method for predicting the failure loads at different overlaps compared to assuming some average FM shear stress. Figure 7.38 gives a comparison of these two methods with the actual experimental data for the IN718/BNi-2 braze joints tested in this study using the 2T overlap test results to predict the failure load at other overlaps. As shown, the predicted and actual failure loads are identical for the 2T overlap for both methods since this overlap was used as the basis point. However, using the average FM shear stress (Eqn 3.1) drastically over predicts the failure load at larger overlaps and under predicts the load at shorter overlaps whereas the FEA results using the DZM approach is in good agreement with the experimental results.
Figure 7.38: Comparison between experimental and predicted failure loads from FEA models (DZM approach) and average FM shear stress based on 2T overlap results.

### 7.5.5 Damage Zone Model for Brazed Joints

Results from this study and FEA modeling work indicates a damage zone model may be a useful method for predicting failure in lap shear brazed joints. As a result, the following procedure adopted from adhesive bonding [100] is proposed to predict the critical load for any brazed lap joint using the damage zone method:

I. Test one or more brazed joint(s) using DIC analysis and record the load at which failure occurs and failure mode.

II. Analyze the joint(s) at the experimental failure load using an appropriate analysis tool such as FEA.

III. Using an appropriate failure criterion and selected material allowable(s) calculate the damage zone size in brazed joint region and validate with DIC results.
IV. Use the critical damage zone size calculated in the previous step to predict the critical load of brazed joints with the same BM/FM combination and load path.

Using this type of methodology would eliminate the issues with inconsistent FM shear stress values associated with the lap joint test method and provide a consistent failure criteria that can be used in the design of brazed lap joints. As shown in this initial study, the DIC technique allows for experimental validation of FEA models and direct measurement of the critical damage zone size. However, additional studies following the suggested improvements in this area are recommended to validate the DZM approach for lap shear brazed joints and to investigate the influence of process parameters on the damage zone size.

7.6 Summary

In this Chapter the mechanical behavior of Ni-based superalloys was studied on both the micro- and macro-scale. In general, the brazed joint strength was lower than the BM strength. Standard butt joints with low volume fraction eutectic failed at approximately 90% of the BM yield strength and showed very low strain-to-failure or ductility. Small-scale tensile tests indicated volume fraction of centerline eutectic phases dramatically reduced the local ductility and ultimate tensile strength of the joint. Based on the in-situ observations during testing a micro-scale failure mechanism was proposed based on strain concentrations around hard, brittle intermetallic phases resulting in plastic deformation and crack initiation in the matrix phase of the joint. In addition, local stress/strain properties were quantitatively measured using DIC analysis and showed the
Ni-base brazed joint material has similar elastic/plastic properties with the BM for eutectic free joints.

Lap shear testing was performed for both CMSX-4/BNi-2 and IN718/BNi-2 brazed joints utilizing DIC to quantify the damage zone at the edge of the overlap. FEA models were used to validate the DIC results and showed relatively good agreement with the experimental strain measurements. Based on both DIC and FEA results, damage zone models appear to provide a much better prediction of the brazed joint failure loads. From these results, the actual damage zone rather than the normalized damage zone was found to be more consistent between overlaps. Similar to adhesive lap joints, a methodology for developing damage zone models for lap shear brazed joints was proposed.
CHAPTER 8

8. FRACTURE MECHANICS TESTING

8.1 Introduction

As shown experimentally in Chapter 7 from lap shear and tensile testing, brazed joints typically exhibit low ductility and toughness compared to the BM due to tri-axial stress and formation of eutectic phases. In addition, defects such as lack of bonding, shrinkage porosity or cracks present in the joint significantly reduce the load carrying capacity of the brazed component. Therefore, an accurate model of brazed joint strength should account for the fracture toughness and presence of defects in the final brazed component. As discussed in Chapter 3, for welded structures FFS methods are used to assess the acceptability of flaws by using FADs based on the $S_r$ and $K_r$ ratios to establish “acceptable” or “unacceptable” criteria. In order to apply a similar approach for brazed joints, knowledge of the critical stress intensity factor ($K_{IC}$) of the joint material is necessary in order to calculate the fracture ratio. However, currently there is no standard methodology for evaluating the $K_{IC}$ of brazed joints.

As a result, the objective of this chapter was to evaluate fracture mechanics test methods identified in Chapter 3 for measuring Mode I fracture toughness ($K_{IC}$) of brazed joints. The compact tension (CT), double cantilever beam (DCB), and V-Notch single edge tension (SENT) test specimens were selected for evaluation since these have been
previously studied and $K_{IC}$ calculations have been provided in the literature. Testing was carried out using these geometries for IN718/BNi-2 brazed joints in the same brazing cycle to compare the manufacturing/brazing procedures, $K_{IC}$ values and repeatability of these methods. Recommendations for future fracture mechanics testing of brazed joints based on the results were made to provide a basis for fitness-for-service assessment of brazed joints.

8.2 Experimental Procedure

Due to the cost and anisotropy with the CMSX-4 single crystal material, IN718/BNi-2 brazed joints were selected for the fracture mechanics testing. Initial geometries for the DCB, CT, and V-Notch test samples were water jet cut from IN718 plates to the dimensions shown by the schematic drawings listed in Appendix A. Six samples were fabricated for each specimen type following the same brazing procedures used for lap shear and butt joint test specimens. Brazing surfaces were grit blasted with NicroBlast Grit and cleaned in an acetone bath prior to brazing. V-Notch test specimens were laser tack welded with two layers of pre-placed BNi-2 foil (0.001 inch thickness) in the joint region for an average joint gap of 0.002-0.004 inch. Additional paste was applied to the edge of these joints. For CT and DCB specimens custom graphite fixtures were designed for brazing shown in Figure 8.1, with two layers of BNi-2 foil placed in the joint region. One side was kept open to allow for thermal expansion on heating and the weight of the top piece imparted a slight pressure on the joint to achieve a similar joint gap of 0.002-0.004 inch.
Figure 8.1: Brazing setup with graphite fixtures for CT and DCB specimens.

The standard BNi-2 brazing cycle (Table 5.2) was used for brazing followed by the IN718 post-braze solution and age heat treatment (Figure 7.1). Additional surface grinding was applied after heat treatment to remove any misalignment or excess FM for flat, smooth surface finish. CT and DCB specimens were tested following ASTM E399 testing procedures as closely as possible using the 810 MTS tensile tester. For pin loading, custom clevis grips (drawing in Appendix A) were fabricated from 17-4 martensitic precipitation-hardening stainless steel following the ASTM 399 recommendations. CT and DCB specimens were tested at a constant displacement rate of 0.0007 in/s and a clip-on displacement gauge was used to measure crack mouth opening displacement CMOD at a gauge length of 0.25 inch using knife edges adhesively bonded to the outer edge of the samples. Figure 8.2 shows the CT fracture mechanics test setup.
Two rounds of testing were performed using the same test specimens and brazing/manufacturing procedures. All samples in Round 1 exhibited brittle failure through the braze joint without significant plastic deformation. For Round 2 testing, both sides of the crack surface was machined (milled) off and the joints were re-brazed for additional testing. Re-use of the same samples is not recommended for fracture mechanics testing, but it reduced the cost and time associated with making new samples for this study.

An important step in fracture mechanics testing is pre-cracking to achieve a sharp crack prior to testing. Even the smallest practical machined notch will leave a slight radius at the notch tip which can increase the size of the plastic zone at the crack tip and lead to inaccurate $K_{IC}$ calculations [150]. To obtain a natural pre-crack fatigue pre-cracking techniques, such as those listed in ASTM E399, by cyclic loading at a low stress
level is often required. However, preliminary attempts indicated the brazed joints were found to be too brittle for fatigue pre-cracking and fracture occurred at the onset of fatigue crack initiation. Therefore, three alternative pre-cracking methods were examined for the brazed joints tested in this study; (1) fine wire EDM cutting to produce a narrow notch after brazing, (2) a feeler gauge coated with braze Stop-Off placed in the crack area prior to brazing and (3) Stop-Off paint applied directly in the crack area prior to brazing similar to the technique used by Ghovanlou et al. [108]. In Round 1 testing, the EDM approach was used for all samples. In Round 2, the feeler gauge with Stop-Off and Stop-Off only approaches were used.

Micrographs comparing the crack tip for these three pre-cracking techniques are shown in Figure 8.3 with arrows indicating the location of the brazed joints. From a practical standpoint the EDM method was difficult to align in the center of the joint especially for the DCB test specimens and the feeler gauge method made it difficult to maintain tight joint gaps at the crack tip due to the slight waviness of the gauge. As shown, the Stop-Off method produced the sharpest crack and ensured the crack was centered at the brazed joint. However, initial results indicated the pre-cracking methods had very little effect on the measured fracture toughness. As a result, the Stop-Off method appears to be the best option for pre-cracking brazed joints.
8.3 Results and Discussion

8.3.1 Compact Tension (CT) Results

Figure 8.4, shows an example CT brazed specimen after final machining prior to testing with higher magnification LOM images of the crack tip and brazed joint. Similar images were taken for each sample tested to measure the initial crack length and ensure the notch or pre-crack was located at the center of the brazed joint. In addition, LOM was used to measure any changes in joint gap or joint microstructure. All samples tested were found to contain approximately 30-50% centerline eutectic phases as shown in the right LOM image in Figure 8.4.
Figure 8.4: IN718/BNi-2 brazed joint CT test specimen at low and high magnification.

The equation for calculation of $K_Q$ from ASTM E399 was used to calculate the fracture toughness ($K_{IC}$) of the braze joints in inch-pound units (ksi√in) as:

$$K_Q = \frac{P_Q}{B\sqrt{W}} * \left(\frac{a}{W}\right)^2$$

(8.1)

Where $P_Q$ is the failure load, $B$ is the specimen thickness, $W$ is the specimen width, $a$ is the crack size and $f(a/W)$ is a dimensionless parameter to account for the specimen geometry. For CT specimens:

$$f\left(\frac{a}{W}\right) = \left(2 + \frac{a}{W}\right) \left[0.886 + 4.64\frac{a}{W} - 13.32\left(\frac{a}{W}\right)^2 + 14.72\left(\frac{a}{W}\right)^3 - 5.6\left(\frac{a}{W}\right)^4\right]$$

$$\frac{1}{\left(1 - \frac{a}{W}\right)^{3/2}}$$

(8.2)

For the brazed CT samples $a/W$ ranged from 0.42-0.45. Force vs. crosshead displacement curves for Round 1 and Round 2 tests are shown in Figure 8.5. All samples failed through the BNi-2 joint region at the onset of crack initiation with unstable crack growth once the peak load was reached. A few pop-ins were also observed at lower loads and could be heard audibly during testing. Weiss et al. observed similar pop-ins via acoustic emission measurements during three point bend tests of a similar BNi-5 FM with an Inconel 718
BM [119]. Weiss et al. concluded cracking of brittle phases occurs in the early stages of loading well below the load reaches its maximum value.

Figure 8.5: Force vs. CMOD curves for IN718/BNi-2 CT test specimens.

The peak load and calculated $K_{IC}$ values are listed in Table 8.1 for all samples tested. As shown, the maximum applied load ranged from 859 to 1240 lbf for Round 1 and 810 to 2,200 lbf in Round 2 resulting in an average $K_{IC}$ value of 16.43 ksi√in. Examination of the brazed joint microstructure prior to testing for the two samples from Round 2 brazed that sustained the highest loads (2,683 and 2,383 lbf) indicated a smaller joint gap and low volume fraction of eutectic phases at the crack tip. This was likely due to the re-machining of the crack surfaces before brazing Round 2, but indicates that the joint microstructure and thus the brazing process parameters can play a strong role on the measured $K_{IC}$ for brazed joints.
Table 8.1: CT test results for Round 1 and Round 2 testing.

<table>
<thead>
<tr>
<th>Test Round</th>
<th>Sample ID</th>
<th>Pre-Cracking Method</th>
<th>a/w</th>
<th>(P_0) (lbf)</th>
<th>(K_0) (ksi√in)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>CT1</td>
<td>EDM</td>
<td>0.45</td>
<td>1,191</td>
<td>13.89</td>
</tr>
<tr>
<td>1</td>
<td>CT2</td>
<td>EDM</td>
<td>0.46</td>
<td>859</td>
<td>10.30</td>
</tr>
<tr>
<td>1</td>
<td>CT3</td>
<td>EDM</td>
<td>0.42</td>
<td>1,074</td>
<td>11.53</td>
</tr>
<tr>
<td>1</td>
<td>CT4</td>
<td>EDM</td>
<td>0.42</td>
<td>1,240</td>
<td>13.31</td>
</tr>
<tr>
<td>1</td>
<td>CT5</td>
<td>EDM</td>
<td>0.46</td>
<td>1,240</td>
<td>14.88</td>
</tr>
<tr>
<td>2</td>
<td>CT1 - Rd2</td>
<td>Shim</td>
<td>0.45</td>
<td>1,221</td>
<td>14.23</td>
</tr>
<tr>
<td>2</td>
<td>CT4 - Rd2</td>
<td>Shim</td>
<td>0.45</td>
<td>911</td>
<td>10.62</td>
</tr>
<tr>
<td>2</td>
<td>CT3 - Rd2</td>
<td>Stop-Off</td>
<td>0.45</td>
<td>2,686</td>
<td>31.31</td>
</tr>
<tr>
<td>2</td>
<td>CT5 - Rd2</td>
<td>Stop-Off</td>
<td>0.45</td>
<td>2,383</td>
<td>27.78</td>
</tr>
<tr>
<td></td>
<td>Average:</td>
<td>Rd. 1</td>
<td></td>
<td>1,121</td>
<td>12.78</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Rd. 2</td>
<td></td>
<td>1,800</td>
<td>20.99</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Total</td>
<td></td>
<td>1,423</td>
<td>16.43</td>
</tr>
<tr>
<td></td>
<td>Std. Dev:</td>
<td>Rd. 1</td>
<td></td>
<td>161</td>
<td>1.84</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Rd. 2</td>
<td></td>
<td>866</td>
<td>10.10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Total</td>
<td></td>
<td>650</td>
<td>7.66</td>
</tr>
</tbody>
</table>

8.3.2 Double Cantilever Beam (DCB) Results

Figure 8.6, shows an example vacuum brazed DCB sample after heat treatment and machining. Compared the CT specimens which contained a cutout notch section, the DCB specimens were much easier to align and assemble for brazing. Similar to the CT specimens the joint microstructure of the DCB specimens contained a continuous centerline eutectic region \(f_e \sim 0.3-0.5\). Force vs. CMOD curves for all DCB samples tested are provided in Figure 8.7 and all joints fractured in a brittle-like manner through the BNi-2 joint. As shown, some of the Rd. 2 tests (blue lines) showed crack propagation rather than immediate failure at the peak load. This was likely a result of a poor re-brazed joint as small defects were observed in the internal joint area for these joints. If these defects were in the vicinity of the crack tip they would provide a path for easy crack propagation.
One sample in Rd. 1 testing (not shown in Figure 8.7) exhibited almost twice the failure load as the other samples. Closer examination of this sample in Figure 8.8 (left) showed the location of the EDM crack tip was approximately 300 μm offset from the
joint centerline whereas the other samples had good alignment with the joint and EDM pre-crack (right). This misalignment effectively resulted in a lower stress intensity factor at the braze joint compared to the other samples and thus a higher load to failure. The Stop-Off and feeler gauge pre-cracking techniques avoided this issue in Rd. 2 testing.

![Image of crack tip in IN178/BNi-2 fracture mechanics test specimens pre-cracked using EDM technique with misalignment (left) and good alignment (right) to brazed joint.]

Figure 8.8: Images of crack tip in IN178/BNi-2 fracture mechanics test specimens pre-cracked using EDM technique with misalignment (left) and good alignment (right) to brazed joint.

Table 8.2 lists the DCB test results for all samples tested including the peak load and calculated $K_{IC}$ values. The equation for the critical stress intensity factor for the DCB specimen was derived by Leinenbach et al. [79] based on the peak load ($F$) as:

$$K_I = \frac{F}{B h^{0.5}} \left(8 + 13.25 \frac{a}{h} + 12 \left(\frac{a}{h}\right)^2\right)^{0.5}$$

(8.3)

where, $B$ is the sample thickness, $h$ is half the height of the sample and $a$ is the initial crack length. As shown, Round 1 testing gave much more consistent results compared to Round 2 with the large scatter in Round 2 likely associated with inconsistent machining and re-brazing of the samples. The average $K_{IC}$ value excluding the misaligned sample
(DCB1) was calculated as 14.43 ksi√in for the BNi-2 FM. This value is within 2 ksi√in of the average $K_{IC}$ calculated for CT specimens.

Table 8.2: DCB test results for Round 1 and Round 2 testing.

<table>
<thead>
<tr>
<th>Test Round</th>
<th>Sample ID</th>
<th>Pre-Cracking Method</th>
<th>a/h</th>
<th>F (lbf)</th>
<th>$K_{IC}$ (ksi√in)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DCB1</td>
<td>EDM</td>
<td>0.67</td>
<td>3,390</td>
<td>35.05</td>
</tr>
<tr>
<td>1</td>
<td>DCB2</td>
<td>EDM</td>
<td>0.67</td>
<td>1,406</td>
<td>14.54</td>
</tr>
<tr>
<td>1</td>
<td>DCB3</td>
<td>EDM</td>
<td>0.67</td>
<td>1,553</td>
<td>16.05</td>
</tr>
<tr>
<td>1</td>
<td>DCB4</td>
<td>EDM</td>
<td>0.67</td>
<td>1,328</td>
<td>13.73</td>
</tr>
<tr>
<td>1</td>
<td>DCB5</td>
<td>EDM</td>
<td>0.67</td>
<td>1,367</td>
<td>14.14</td>
</tr>
<tr>
<td>1</td>
<td>DCB6</td>
<td>EDM</td>
<td>0.67</td>
<td>1,543</td>
<td>15.95</td>
</tr>
<tr>
<td>2</td>
<td>DCB1 - Rd2</td>
<td>Stop-Off</td>
<td>0.72</td>
<td>1,016</td>
<td>11.40</td>
</tr>
<tr>
<td>2</td>
<td>DCB2 - Rd2</td>
<td>Stop-Off</td>
<td>0.72</td>
<td>2,051</td>
<td>23.01</td>
</tr>
<tr>
<td>2</td>
<td>DCB4 - Rd2</td>
<td>Shim</td>
<td>0.72</td>
<td>889</td>
<td>9.97</td>
</tr>
<tr>
<td>2</td>
<td>DCB6 - Rd2</td>
<td>Shim</td>
<td>0.72</td>
<td>986</td>
<td>11.07</td>
</tr>
</tbody>
</table>

Average: Rd. 1 1,439 14.88  
Rd. 2 1,235 13.86  
Total 1,349 14.43  

Std. Dev. 
Rd. 1 103 1.06  
Rd. 2 546 6.13  
Total 359 3.87

The average $K_{IC}$ value for the IN718/BNi-2 brazed joints was lower than the value reported by Leinenbach et al. of 44 ksi√in using the same DCB specimens for martensitic stainless steel brazed joints with the Au-18Ni FM. The Au-18Ni FM was found to produce eutectic free brazed joints consisting of a primarily Au solid solution phase compared to the continuous eutectic centerline observed in the IN718/BNi-2 joints. Again, this indicates the FM/BM combination and joint microstructure will play a strong role on the calculated $K_{IC}$ of brazed joints.

8.3.3 V-Notch Single Edge Notch Tension (SENT)
The V-Notch (SENT) specimen was adopted from Gan et al. who used this geometry to measure the fracture toughness of vanadium/stainless steel brazed joints [118]. Figure 8.9 shows one of the IN718/BNi-2 V-Notch samples after vacuum furnace brazing along with a higher magnification LOM image of the pre-crack at the joint. Again, the joint microstructure in Rd.1 was found to consist of a moderate amount of centerline eutectic phases across the joint. Machining and re-brazing in Rd. 2 resulted in larger joint gaps on the order of 0.004-0.008 inch compared to the 0.002-0.004 in Rd. 1 and as a result consisted of a higher volume fraction of centerline eutectic phases.

Figure 8.9: IN718/BNi-2 brazed joint V-Notch test specimens prior to testing at high and low magnification.
Since these samples were pulled in tension, crosshead displacement was measured rather than CMOD. Applied force vs. crosshead displacement curves for the V-Notch brazed joint samples are shown in Figure 8.10 and all samples exhibited brittle fracture through the brazed joints. As shown, there was quite a range in the measured peak load in Rd. 1 (with small joint gaps) ranging from 2,578 to 5,371 lbf for the brazed joints. The large joint gaps observed in the Rd. 2 samples resulted in a 1,000 lbf (6 ksi) decrease of the average failure load.

![Figure 8.10: Force vs. displacement curves for IN718/BNi-2 brazed joint V-Notch test specimens.](image)

For comparison, BM V-Notch samples were also made consisting entirely of IN718 (in the solution and aged condition) without a braze joint. These BM samples were slightly thinner with a thickness of 0.2 inch rather than the 0.25 inch used for the brazed
joint. The force vs. displacement curves from these BM V-Notch samples in Figure 8.11 show a much high peak load (~9735 lbf) even for the smaller cross-section and more ductility compared to the brazed joint samples.

![Force vs. displacement curves](image)

Figure 8.11: Force vs. displacement curves for IN718 BM V-Notch single edge notch tension specimens.

A summary of the V-Notch test results are listed in Table 8.3. The equation Gan et al. used to calculate the $K_{IC}$ for the V-Notch specimen was originally described by Irwin et al. [110] as:

$$K_{IC} = \sigma_c \sqrt{\pi a F \left( \frac{a}{W} \right)}$$

(8.4)

where $\sigma_c$ is the fracture stress far away from the crack, $a$ is the initial crack length, $W$ is the width of the sample and $F(a/W)$ is the geometrical configuration parameter which in the case of a SENT type specimen is:
\[
F\left(\frac{a}{W}\right) = 1.12 - 0.23 \frac{a}{W} + 10.6 \left(\frac{a}{W}\right)^2 - 21.7 \left(\frac{a}{W}\right)^3 + 30.4 \left(\frac{a}{W}\right)^4
\]

(8.5)

For these tests a/W was approximately \(\sim 0.46\) with an initial crack length of 0.35 inch.

Fracture stress \((\sigma_c)\) was calculated as the peak load \((P_Q)\) divided by the c/s area which was \(\sim 0.1855\) in\(^2\) for the brazed joints and 0.15 in\(^2\) for the BM samples.

Table 8.3: V-Notch single edge notch tensile test results for IN718/BNi-2 joints.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>a/w</th>
<th>F(a/w)</th>
<th>(P_Q) (lbf)</th>
<th>(\sigma_c) (ksi)</th>
<th>(K_Q) (ksi√in)</th>
</tr>
</thead>
<tbody>
<tr>
<td>VNotch1</td>
<td>0.46</td>
<td>2.51</td>
<td>3,462</td>
<td>18.67</td>
<td>48.13</td>
</tr>
<tr>
<td>VNotch2</td>
<td>0.46</td>
<td>2.51</td>
<td>5,371</td>
<td>28.95</td>
<td>74.66</td>
</tr>
<tr>
<td>VNotch3</td>
<td>0.46</td>
<td>2.51</td>
<td>3,750</td>
<td>20.22</td>
<td>52.13</td>
</tr>
<tr>
<td>VNotch4</td>
<td>0.46</td>
<td>2.51</td>
<td>2,578</td>
<td>13.90</td>
<td>35.84</td>
</tr>
<tr>
<td>VNotch5</td>
<td>0.46</td>
<td>2.51</td>
<td>2,656</td>
<td>14.32</td>
<td>36.92</td>
</tr>
<tr>
<td>VNotch6</td>
<td>0.46</td>
<td>2.51</td>
<td>3,994</td>
<td>21.53</td>
<td>55.52</td>
</tr>
<tr>
<td>VNotch1-Rd2</td>
<td>0.45</td>
<td>2.43</td>
<td>1,543</td>
<td>8.32</td>
<td>20.82</td>
</tr>
<tr>
<td>VNotch3-Rd2</td>
<td>0.45</td>
<td>2.43</td>
<td>2,920</td>
<td>15.74</td>
<td>39.39</td>
</tr>
<tr>
<td>VNotch4-Rd2</td>
<td>0.45</td>
<td>2.43</td>
<td>2,031</td>
<td>10.95</td>
<td>27.40</td>
</tr>
<tr>
<td>VNotch6-Rd2</td>
<td>0.45</td>
<td>2.43</td>
<td>3,184</td>
<td>17.16</td>
<td>42.95</td>
</tr>
<tr>
<td>VNotch-BM1</td>
<td>0.46</td>
<td>2.51</td>
<td>9,706</td>
<td>64.71</td>
<td>166.86</td>
</tr>
<tr>
<td>VNotch-BM1</td>
<td>0.46</td>
<td>2.51</td>
<td>9,735</td>
<td>64.90</td>
<td>167.35</td>
</tr>
<tr>
<td>Average:</td>
<td>Rd. 1</td>
<td>3,635</td>
<td>19.60</td>
<td>50.53</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Rd. 2</td>
<td>2,419</td>
<td>13.04</td>
<td>32.64</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Total</td>
<td>3,149</td>
<td>16.98</td>
<td>43.38</td>
<td></td>
</tr>
<tr>
<td>Std. Dev:</td>
<td>Rd. 1</td>
<td>1,026</td>
<td>5.53</td>
<td>14.26</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Rd. 2</td>
<td>764</td>
<td>4.12</td>
<td>10.31</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Total</td>
<td>1,083</td>
<td>5.84</td>
<td>15.29</td>
<td></td>
</tr>
</tbody>
</table>

The average \(K_Q\) value for the IN718/BNi-2 joints from V-Notch testing was found to be 43.38 ksi√in with a relatively high standard deviation of 15.29 ksi√in. For comparison \(K_Q\) values reported by Gan et al. were slightly lower ranging from 1.5 to 18 ksi√in depending on the FM tested. The \(K_{IC}\) value from V-Notch testing is also
significantly higher than the $K_{IC}$ measured using the DCB and CT test specimens. Similar results were reported by Weiss et al. and were attributed to the restraint of the BM on the joint when loaded in tension [119].

The BM V-Notch samples exhibited significantly higher $K_{IC}$ values for IN718 compared to the brazed joints reaching a maximum of $167.3 \text{ ksi} \sqrt{\text{in}}$ at an applied load of $9,735 \text{ lbf}$. However, as a general rule in fracture mechanics testing, in order for a plane strain fracture toughness value to be considered valid the maximum thickness ($t$) required for a material with a given yield strength ($\sigma_{YS}$) is [114]:

$$t \geq 2.5 \left( \frac{K_{IC}}{\sigma_{YS}} \right)^2$$

(8.6)

Therefore, the thickness of the BM samples was not sufficient enough for a valid measure of $K_{IC}$ for IN718. Despite not having a direct comparison of the $K_{IC}$ between the joint and BM the large difference in the V-Notch fracture stress clearly demonstrates a large reduction in the joint toughness relative to the BM.

These results also indicate that large defects in brazed joints cannot be simply treated as a reduction in joint area. The average fracture stress of the joint for the notched sample, calculated by the average applied load from Rd. 1 testing ($3,635 \text{ lbf}$) divided by the reduced area at the joint ($0.1 \text{ in}^2$) due to the notch, is $36.35 \text{ ksi}$. This is only 23% of the average fracture stress measured for the un-notched IN718/BNi-2 butt joints ($152.8 \text{ ksi}$) from Section 7.2. This “notch-effect” was less significant for the BM as the average tensile strength for the reduced area ($0.08 \text{ in}^2$) for the BM samples was $121.6 \text{ ksi}$, approximately 56% of the BM UTS.
8.4 FEA Modeling

To validate the $K_{IC}$ values calculated for the IN718/BNi-2 brazed joints and the equations provided in the literature, 2D linear-elastic FEA models of the test specimens were generated in ABAQUS. The initial crack length was defined using the contour integral crack tool based on linear elastic fracture mechanics. The average experimental failure load was then used to calculate the Mode I critical stress intensity factor ($K_{IC}$). Due to the brittle nature of the joint linear elastic fracture mechanics was assumed for these models using the elastic properties determined in Chapter 7 with an elastic modulus of $29.8 \times 10^6$ psi and Poisson’s ratio of 0.29. Partitioning was used to decrease the mesh size and increase the number of nodes at the crack tip. A mesh sensitivity analysis showed very little effect of the mesh size on the calculated $K_{IC}$.

The FEA model for the CT test specimen is shown Figure 8.12. As shown, a half-crack model was used due to symmetry. Boundary conditions (top image) were assigned to simulate the test configuration with the brazed joint region fixed in the y-direction and a load applied at the center of the hole with the hole constrained as a rigid body. A crack length of 0.25 inch was applied to the model for an approximate $a/W$ of 0.46. The FEA calculated maximum principal strain at the average experimental peak load of 1,423 lbf was found to be very low (<0.2%) even near the crack tip as shown in the lower image in Figure 8.12. For this applied load $K_{IC}$ was calculated to be 16.20 ksi $\sqrt{\text{in}}$ for the CT geometry which is in good agreement with the $K_{IC}$ values calculated using Eqn. 8.1.
Figure 8.12: Mesh and boundary conditions (top) and calculated $\varepsilon_{\text{max}}$ (bottom) from FEA model of the CT IN718/BNi-2 braze joints fracture mechanics test specimen.

Similar to the CT test specimen the FEA model for the DCB specimens was setup as a half-crack model with the boundary conditions shown in Figure 8.13. For this geometry a crack length of 1.38 inches from the edge of the sample was assigned and a load of 1349 lbf was applied to the rigid body hole to simulate the average experimental peak load. The strain distribution at the crack tip was very similar to the CT model and a $K_{\text{IC}}$ of 12.86 ksi $\sqrt{\text{in}}$ was calculated. This was slightly lower than the values calculated
using Eqn. 8.3 which may be due to small deviations in the crack length between the experimental setup and the FEA model.

Figure 8.13: Mesh and boundary conditions (top) and calculated $\varepsilon_{\text{max}}$ (bottom) from FEA model of the DCB IN718/BNi-2 braze joints fracture mechanics test specimen.

The FEA model of the V-notch test specimen is shown in Figure 8.14. As shown, the average tensile stress (50.53 ksi) of the Rd.1 testing was applied to the half-crack model with the brazed joint region fixed in the y-direction. Crack size was set to 0.125 inch for an $a/W$ of 0.46 to match the test geometry. The calculated $K_{IC}$ from the FEA
model was 48.23 ksi\(^1\!/\text{in}\) giving good agreement with the experimental \(K_{\text{IC}}\) of 50.53 ksi\(^1\!/\text{in}\).

![Applied Stress 19.6 ksi](image)

![Braze Joint \(\mu_r = 0\), Crack Length \(a = 1.38\)"

Figure 8.14: FEA model of V-Notch test specimen showing mesh and boundary conditions (right) and calculated maximum strain (right) for IN718/BNi-2 joints.

### 8.5 Fractography

Two fracture paths were observed within the brazed joints and can be seen by the LOM images of DCB specimens after testing in Figure 8.15. The first path (left image) is at the joint interface between the ISZ and the BM and the second (right) is through the centreline eutectic (ASZ) region. While the second type of cracking was most prevalent, most test specimens exhibited both types with cracks randomly migrating between the interface and centreline across the length of the crack.
Figure 8.15: Fracture paths observed in IN718/BNi-2 fracture mechanics testing occurred both at the joint interface (left) and through the centerline eutectic (right).

The fracture surface from one of the V-Notch IN718/BNi-2 joint test specimens was further analysed via SEM. At low magnification (30X) in Figure 8.16, light and dark ‘islands’ appear on the fracture surface where the crack occurred at the joint interface rather than through the joint centreline. In this case, the light regions are raised and represent the interface between the BM piece the broke off during testing while the dark regions are recessed indicating the BM/joint interface for the BM piece below the fracture surface. The opposite was observed on the fracture surface of the other piece. However, as shown the majority of the fracture surface area was through the joint centreline.
In Figure 8.16 the fracture surface for the centreline eutectic was observed at higher magnifications of 6000X (left) and 4000X (right). In these images the fracture surface features cleavage facets surrounded by micro-ductility dimples indicating a mixed brittle-ductile fracture mode. This can be related to the failure mechanism described in Section 7.4 as eutectic free regions exhibit more ductile behaviour while intermetallic phases such as CrB and Ni$_3$B result in a brittle fracture leaving the flat, smooth cleavage features behind.
Figure 8.17: High magnification SEM images at 6000X (left) and 4000X (right) of fracture surface features for Rd.1 V-Notch test sample.

8.6 Summary

An evaluation of fracture mechanics test methods for measuring the critical stress intensity factor ($K_{IC}$) in Ni-base superalloy brazed joints was provided in this chapter. As introduced in Chapter 3, accurate measurements of $K_{IC}$ values are necessary to perform fitness-for-service assessments and to calculate the fracture ratio ($K_r$) in failure assessment diagrams for brazed joints containing defects. Therefore, IN718/BNi-2 brazed joints were fabricated and tested using the CT, DCB, V-Notch and SENB test specimens with manufactured defects to measure the average $K_{IC}$ of the braze joint. DCB and CT test specimens resulted in a similar average $K_{IC}$ values of 13.49 ksi$\sqrt{\text{in}}$ and 14.23 ksi$\sqrt{\text{in}}$ respectively. The V-Notch or single edge notch tensile specimen gave a higher $K_{IC}$ of 43.38 ksi$\sqrt{\text{in}}$. This was attributed to the restraint introduced through the BM in pure tensile loading. FEA modeling was used to validate the calculated $K_{IC}$ values and
equations provided in literature showed good agreement with the modeling results. Fractographic analysis indicated two fracture modes occurred in cracks through the brazed joints one along the centerline eutectic and another at the joint interface.

Results from two rounds of testing with these test specimens indicate the joint microstructure and joint gap have a large effect on the $K_{IC}$. When an increase in the volume fraction of centerline eutectic phases was observed in Rd. 2 testing such as for the V-Notch and DCB specimens the $K_{IC}$ was 60-70% of the average $K_{IC}$ calculated in Rd. 1. The opposite was true for samples with small joint gaps and low centerline eutectic phases at the crack tip such as the CT3 and CT5 specimens in Rd. 2 which resulted in a 20-30% increase in the average Rd. 1 $K_{IC}$.

Based on these findings the CT or DCB type specimens provides a more conservative $K_{IC}$ values and therefore are recommended for measuring $K_{IC}$ for brazed joints. The Stop-Off pre-cracking technique is best suited for making starter cracks for as it creates a sharp crack centered at the brazed joint. Details on specimen geometry and brazing procedures have also been established for future fracture mechanics testing of brazed joints.
CHAPTER 9

9. CONCLUSIONS

In this research, data and methodology was generated to support the proposed multi-scale model for prediction of mechanical properties in Ni-base superalloy brazed joints for gas turbine applications. The overall modeling approach is based on microstructure-property relationships that have been observed when brazing these materials with conventional Ni-base FMs. These relationships were further investigated in this work through metallurgical characterizations and mechanical testing of Inconel 718 and CMSX-4 brazed joints. It was shown that an increase in the volume fraction of centerline eutectic (\(f_e\)) significantly reduces the joint material strength and ductility and that the volume fraction of centerline eutectic phases can be estimated by combining thermodynamic and kinetic simulations of the metallurgical interactions that occur during brazing. Since these simulations can account for a wide range of process parameters they can ultimately be used to estimate the overall joint properties based on the selected materials, joint gap, brazing temperature and hold time.

In addition to providing the basic framework for a multi-scale model, several novel experimental techniques were developed to address some of the inherent challenges in assessing brazed joint strength. Small-scale tensile testing of brazed joints was used to measure the local stress/strain properties by extracting miniature dog-bone samples from
the brazed joint. Digital image correlation was also applied to experimentally validate the damage zone model approach for predicting lap shear failure loads and showed good agreement with FEA models of the test specimens at various overlaps. Finally, several fracture mechanics test methods were evaluated to establish a test geometry best suited for measuring the average fracture toughness of brazed joints a key component in the fitness-for-service assessment of a joint containing flaws or defects. Using these techniques improves understanding of brazed joint mechanical behavior and provides data that can be directly applied in the overall multi-scale model.

In general, this investigation provides an initial step towards a multi-scale computational model of Ni-base superalloy brazed joints. The overall approach to account for the properties of micro-constituents and properties across the joint when modeling and designing brazed joints will ultimately improve the mechanical reliability and applied factors of safety for brazed joints in gas turbine applications. Developing a predictive model based on the physical metallurgy will also reduce time and cost associated with the selection of BM/FM materials, development of new FMs, and/or optimization of the brazing process parameters for a given application. Furthermore, the multi-scale modeling approach may also be applied for dissimilar joints in general that have extreme material property gradients over a narrow distance. For example in dissimilar metal welds used in oil and gas, petrochemical, nuclear and power generation industries for a range of material combinations, welding processes and service conditions [129, 151].

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A summary of the specific research contributions from each section of this investigation is given as follows:

**Metallurgical Characterization**

1. Ni-base superalloy brazed joints with Ni-base FMs consist of three primary microstructural regions: a diffusion affected zone (DAZ) in the Ni-base superalloy BM that is enriched in MPD elements, a solid solution α-Ni phase that forms at the joint interface due to isothermal solidification (isothermally solidified zone, ISZ), and a multi-component centerline eutectic that forms during athermal solidification (athermally solidified zone, ASZ).

2. For CMSX-4/BNi-2 and CMSX-4/BNi-9 braze joints the intermetallic phases in the ASZ were identified via electron probe micro-analysis (EPMA) as CrB and Ni₃B type borides within a eutectic matrix enriched in FM elements. EPMA also indicated boron was the only element to show significant diffusion across the BM/FM interface.

3. Micro-hardness mapping across the joints indicated the eutectic phases have extremely high hardness (800-1000 HVN) relative to the soft, ISZ phase (300-400 HVN) and BM (425-450 HVN). Since the underlying material properties can be related to the joint strength these measurements also indicate the joint strength will be affected by the volume fraction of these phases.

4. The primary brazing process parameters including joint gap, brazing temperature, and hold time were found to directly influence the volume fraction of centerline eutectic. Increasing temperature and hold time and/or decreasing joint gap led to a
decrease in centerline eutectic phases due to the increased diffusion of MPD elements (primarily boron) from the joint region into the BM.

5. Amorphous foil form of FM produced a significantly lower volume fraction of eutectic constituents and more uniform hardness properties across the joint compared to the paste form of FM at brazing temperatures below 1100°C.

**Thermodynamic and Kinetic Simulations**

6. A DICTRA diffusion model based on the Ni-B binary system was used to estimate the time for BM dissolution and isothermal solidification of the FM based on the extent of boron diffusion across the solid/liquid interface during the brazing process. The model inputs include the primary process parameters such as BM/FM composition, joint gap, brazing heat cycle, and hold time. As a result, the volume fraction of eutectic constituents and corresponding mechanical properties can be estimated for a wide range of processing conditions.

7. Due to limitations with the thermodynamic/kinetic databases, additional brazing experiments were carried out at 1190°C to validate the DICTRA simulations by measuring the maximum brazing clearance required for isothermal solidification. The DICTRA model showed reasonable agreement with experimental and historical results.

8. Thermo-Calc equilibrium simulations using the composition of the two FMs were utilized to predict the phases that form during solidification of the BNi-2 and BNi-9 FMs. The composition and volume fraction for MB_ORTH, M3B and
FCC_A1 phases predicted in Thermo-Calc corresponded well to the CrB, Ni3B, eutectic and α-Ni phases identified via EPMA and metallurgical characterizations.

9. A new technique using single sensor differential thermal analysis (SSDTA) was developed for studying the phase transformations and solidification of the brazing FMs. Compared to conventional DTA techniques, SS DTA requires only a small volume of liquid, does not require a reference material, and can be applied during the brazing cycle to account for the effect of BM/FM interaction on the solidification process. Consistent solidus/liquidus temperature were reported and showed good agreement with the values reported in AWS A5.8 and Thermo-Calc simulations for the brazing FMs tested.

10. SSDTA results indicated the BNi-9 solidifies in one stage whereas the BNi-2 FM solidification had two primary stages possibly indicating the solidification of the bulky Ni₃B phase found in the BNi-2 joint microstructure before the eutectic temperature was reached. No solid-state transformations were observed in either FM.

Mechanical Behavior of Brazed Joints

11. The average ultimate tensile strength of IN718/BNi-2 of standard size butt joints containing low volume fractions of centerline eutectic phases (<0.05) was found to be relatively high at 152.8 ksi and only slightly below the IN718 BM yield strength of 172 ksi (215 ksi UTS). However, the brazed joints exhibited very low ductility and strain to failure ranging from 0.05 to 0.07 % elongation compared to
18 to 26 % elongation measured for the BM. This is due to the BM restraint and tri-axial stress that develops in the joint material during testing.

12. A novel small-scale tensile test technique was utilized to conduct in-situ observations of the local deformation behavior in IN718/BNi-2 and CMX-4/BNi-2 brazed joints and examine the influence of microstructure on mechanical properties.

13. In some cases, small-scale test samples of defect-free joints with low volume fractions of centerline eutectic constituents exceeded the BM yield strength and plastic deformation (slip bands) was observed in the BM prior to fracture in the brazed joint.

14. A failure mechanism for brazed joints was proposed based on qualitative observations. The hard, brittle intermetallic phases like CrB or Ni₃B borides lead to strain localization in the surrounding matrix and crack initiation at the particle/matrix interface. As the stress continues to increase these cracks propagate through the matrix and link up with other cracks until the crack size reaches a critical size and joint fracture occurs.

15. 2D digital image correlation (DIC) was applied during small-scale tensile testing to extract the stress/strain behavior of the joint material. Both the ultimate tensile strength and strain-to-failure was shown to decrease significantly with increasing joint gap and the volume fraction of eutectic constituents. A minimum tensile stress of approximately 40 ksi was measured for samples consisting entirely of the BNi-2 eutectic constituents.
16. Lap shear testing was carried out for IN718/BNi-2 and CMSX-4/BNi-2 brazed joints at various overlaps. The average FM shear stress decreased from 70.3 ksi to 22.8 ksi for IN718/BNi-2 joints and from 39.4 ksi to 15.7 ksi for CMSX-4/BNi-2 joints as the overlap was increased from 1T to 5T.

17. Digital image correlation (DIC) of the lap shear test specimens was used to quantify the shear, normal, and max principal strains along the brazed joint. At larger overlaps, the strain distribution was found to be highly non-uniform with an effective damage zone at the edge of the overlap. FEA models of the lap shear test specimens showed good correlation with the DIC analysis.

18. Results from the DIC and FEA analysis were used to validate the damage zone model (DZM) approach for predicting failure loads in lap shear brazed joints as proposed by Flom et al. A basic methodology for the use of DZMs for lap shear brazed joints was proposed in this study to ultimately reduce the number of test specimens required to generate the standard AWS C3.2 stress vs. overlap curves. The damage zones experimentally measured in this investigation were also found to remain relatively independent of the overlap distance. As a result, the actual length of the damage zone rather than the normalized length (actual length/overlap distance) may provide a more accurate failure criteria for brazed joints similar to the approach used in adhesive bonding.

**Fracture Mechanics of Brazed Joints**

19. To account for the presence of flaws and defects in brazed joints, fitness-for-service assessments based on calculated stress ($S_r$) and fracture toughness ($K_r$)
ratios have been proposed similar to the API 579 or BS 7910 standards used for welded structures. To support this approach for defect-containing brazed joints, several fracture mechanics test methods (CT, DCB and V-Notch) were evaluated in Chapter 8 for measuring the average critical stress intensity factor ($K_{IC}$) of IN718/BNi-2 brazed joints. $K_{IC}$ is necessary to calculate the $K_r$ in FFS assessments and rarely reported in the technical literature for brazed joints.

20. Test geometries, brazing procedures, testing and pre-cracking methods were developed for each geometry to assist in developing a standard fracture mechanics test method for brazed joints. For pre-cracking the use of brazing Stop-Off was determined to be the most appropriate method for creating a sharp crack with the crack tip centered at the brazed joint.

21. CT and DCB test specimens gave the most conservative $K_{IC}$ of 16.43 and 14.43 respectively. FEA models were used to validate the calculated $K_{IC}$ values and showed good agreement with calculations provided in the literature.

22. The joint microstructure appeared to have an effect on the calculated $K_{IC}$ with an increase in the volume fraction of centerline eutectic constituents or joint gap resulting in a lower $K_{IC}$. Fractography indicated two crack paths in the IN718/BNi-2 brazed joints with the primary path along the centerline eutectic and a secondary path along the BM/FM interface.
CHAPTER 10

10. SUGGESTIONS FOR FUTURE WORK

Additional work is recommended to further the development of multi-scale computational models for brazed joints. The following outlines several suggestions for future work in the main areas investigated in this study:

**Multi-scale Computational Modeling**

While it was shown that the overall strength and toughness of the brazed joints were significantly influenced by the volume fraction of eutectic constituents and therefore a function of the brazing process parameters the role on the individual intermetallic phases within the eutectic was not extensively studied. The dispersion parameters of specific phases within the eutectic constituent may also play a role on joint properties, for example elimination or reduction of the bulky CrB or Ni₃B phases may improve the overall ductility of the joint. The contribution of these individual phases on joint properties could be further analyzed using the small-scale tensile testing technique by applying DIC at a higher resolution for example using hafnium oxide nano-particles as a speckle pattern. By doing this in combination with material property prediction software such as JMatPro™ the stress/strain properties of the individual phases could be determined. Using the material properties assessed for individual phases as inputs, FEA models of the small-scale tensile samples could also be generated based on the relative
size and distribution of the phases observed in the brazed joint prior to testing or based on Thermo-Calc simulations. Applying experimental loads from small-scale testing to the corresponding small-scale FEA models would then be used to calculate the maximum stresses and strains within the joint microstructure that result in crack initiation and fracture of the joint. Ideally, results from these small-scale FEA models could be linked to define material properties or maximum stress/strain for brazed joints in a fully coupled macro-scale FEA model of the overall component.

**Microstructure Characterization**

A more thorough investigation into the role of the form of FM on joint microstructure is also recommended based on this investigation. As shown in Chapter 5 for CMSX-4/BNi-2 joints, the foil form of BNi-2 produced less eutectic constituents and a more uniform micro-hardness across the joint compared to the BNi-2 paste. This was attributed to the amorphous nature of the metal foil that forms due to the rapid quenching of the melt spinning technique and could be quite beneficial in terms of mechanical properties. However, there is a general lack of understanding as to why these joints solidify with this unique structure as both forms of FM have the same composition and are liquid at the brazing temperature. More extensive characterization perhaps via transmission electron microscopy or SEM of the initial atomic structure in these foils and additional brazing studies would improve understanding of these differences.

**Thermodynamic/kinetic Simulations**

The limitations of DICTRA and Thermo-Calc simulations for brazed joints were briefly pointed out in Chapter 6. There is a growing need to develop a braze alloy
database with mobility and thermodynamic data for materials containing high compositions of MPD elements such as B, Si and P with compositions ranging from 0-5 wt%. Diffusivities of these elements in nickel at various temperatures would improve DICTRA simulations. Thermodynamic data should also be adjusted to account for the lower eutectic temperatures observed in brazing FMs. This will likely require software companies such as Thermo-Calc to develop a new database to include these parameters, but experimental validation in this area would greatly assist in this development. EPMA or more advanced compositional analysis should be used for accurate detection of boron and the SSDTA technique is recommended for assessment of the solidus, liquidus and other phase transformation temperatures for brazing FMs.

**Mechanical Testing of Brazed Joints**

Small-scale tensile testing was shown to be an effective way of measuring stress/strain behavior of the actual joint material. Future work in this area comparing bulk FM properties to the joint FM properties for a braze system could also be used to assess the FM/BM interaction strength listed in Eqn. 2.2.

For lap shear testing, it is recommended that rather than testing a range of overlaps for the same joint as described in AWS C3.2 test only one overlap in the 2T-3T overlap range and use the DZM approach to estimate the failure loads for other overlaps. Therefore, rather than testing the same brazed joint at a range of overlaps, more research should be done to examine the influence of process parameters at a single overlap. The effect of thickness (T) on damage zone size and lap shear results should also be investigated.
In addition, although this initial investigation focused on developing data and methodology to support a multi-scale computational of primarily the static strength of brazed joints at room temperature future modeling efforts should be extended to include creep, fatigue and high temperature properties.

**Fracture Mechanics of Brazed Joints**

The influence of joint microstructure on $K_{IC}$ should be assessed in more detail. As shown, in this preliminary investigation the volume fraction of eutectic constituent appeared to have a similar effect on $K_{IC}$ as it did on the UTS, but more testing using a range of brazing parameters would validate these observations. To reduce the time and cost a more simple geometry such as the single edge notch bend (SENB) test specimen listed in ASTM E1820 is recommended as it requires less material and can provide direct comparison with the $K_{IC}$ of the BM. Finally, to validate FFS assessments for brazed joints mechanical testing of more complex geometries with bi-axial loading and pre-manufactured defects should be performed using the material properties evaluated in this study to calculate $S_r$ and $K_r$. 
REFERENCES


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[82] Y. Flom, Interview with Y. Flom, Interview, Brazed Joint Review at The Ohio State University. [Interview]. 19 August Welding Engineering, 2014.


APPENDIX A – SAMPLE GEOMETRIES

Base metal tensile test

Butt joint tensile test

Small-scale tensile test cut out
Lap shear BM sample – 0.5T

Lap shear BM sample – 1T
Lap shear BM sample – 2T

Lap shear brazed joint coupons
Clevis grips

Compact tension (CT)
Double cantilever beam (DCB)

V-Notch Single Edge Notch Tension (SENT)
Single edge notch bend (SENB)
APPENDIX B – MECHANICAL TEST RESULTS

CMSX-4/BNi-2 lap shear 1T overlap

![Graph showing load vs. crosshead displacement for CMSX-4/BNi-2 lap shear 1T overlap with a peak load of 4998 lbf.]  

CMSX-4/BNi-2 lap shear 2T overlap

![Graph showing load vs. crosshead displacement for CMSX-4/BNi-2 lap shear 2T overlap with a peak load of 6420 lbf.]  

CMSX-4/BNi-2 lap shear 3T overlap

![Graph showing load vs. crosshead displacement for CMSX-4/BNi-2 lap shear 3T overlap.]
CMSX-4/BNi-2 lap shear 4T overlap

CMSX-4/BNi-2 lap shear 5T overlap
IN718/BNi-2 lap shear 1T overlap

IN718/BNi-2 lap shear 2T overlap
IN718/BNi-2 lap shear 3T overlap

IN718/BNi-2 lap shear 5T overlap
IN718 base metal lap shear

IN718/BNi-2 lap shear DIC results
<table>
<thead>
<tr>
<th></th>
<th>1T overlap 69 ksi</th>
<th>2T overlap 88 ksi</th>
<th>3T overlap 94 ksi</th>
<th>5T overlap 119 ksi</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\gamma_{xy}$</td>
<td><img src="image1" alt="Image" /></td>
<td><img src="image2" alt="Image" /></td>
<td><img src="image3" alt="Image" /></td>
<td><img src="image4" alt="Image" /></td>
</tr>
<tr>
<td>$E_{yy}$</td>
<td><img src="image5" alt="Image" /></td>
<td><img src="image6" alt="Image" /></td>
<td><img src="image7" alt="Image" /></td>
<td><img src="image8" alt="Image" /></td>
</tr>
<tr>
<td>$E_{xx}$</td>
<td><img src="image9" alt="Image" /></td>
<td><img src="image10" alt="Image" /></td>
<td><img src="image11" alt="Image" /></td>
<td><img src="image12" alt="Image" /></td>
</tr>
</tbody>
</table>
IN718/BNi-2 lap shear 1T max principal strain

IN718/BNi-2 lap shear 1T normal strain ($E_{yy}$)
IN718/BNi-2 lap shear 1T normal strain ($E_{xx}$)

IN718/BNi-2 lap shear 1T shear strain ($\gamma_{xy}$)
IN718/BNi-2 lap shear 2T max principal strain

![Graph showing maximum principal strain vs. actual distance (in)].

IN718/BNi-2 lap shear 2T normal strain ($E_{yy}$)

![Graph showing normal strain ($E_{yy}$) vs. actual distance (in)].
IN718/BNi-2 lap shear 2T normal strain ($E_{xx}$)

Actual distance (in)

IN718/BNi-2 lap shear 2T shear strain ($\gamma_{xy}$)

Actual distance (in)
IN718/BNi-2 lap shear 3T max principal strain

IN718/BNi-2 lap shear 3T normal strain ($E_{yy}$)
IN718/BNi-2 lap shear 3T normal strain ($E_{xx}$)

![Graph showing normal strain ($E_{xx}$) for different distances.]

IN718/BNi-2 lap shear 3T shear strain ($\gamma_{xy}$)

![Graph showing shear strain ($\gamma_{xy}$) for different distances.]

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IN718/BNi-2 lap shear 5T max principal strain

IN718/BNi-2 lap shear 5T normal strain ($E_{yy}$)
IN718/BNi-2 lap shear 5T normal strain ($E_{xx}$)

IN718/BNi-2 lap shear 5T shear strain ($\gamma_{xy}$)
APPENDIX C – DICTRA MODEL

@@
@@ ISOTHERMAL SOLIDIFICATION MODEL FOR NI-B SYSTEM
@@

@@
@@ SELECT DATABASE
@@

go da
sw ssol4

@@
@@ DEFINE SYSTEM
@@

def-species b ni

@@
@@ REJECT ALL PHASES EXCEPT FCC AND LIQUID
@@

rej ph *
rest ph
fcc_a1 liquid

get

@@
@@ APPEND MOBILITY DATA FOR FCC AND LIQUID
@@

app
mob2
def-sys
ni b
rej ph *
rest ph fcc_a1 liquid
get

@@
@@ GO TO DICTRA MODULE
@@

go d-m

@@
@@ SET TEMPERATURE (BRAZING TEMP)
@@

set-condition global t 0 1443; °N
ENTER REGION FOR LIQUID FM AND FCC BM

ENTER REGION

LIQUID

ENTER REGION fcc

LIQUID

YES

ENTER GRID FOR EACH REGION, WIDTH OF LIQUID REGION EQUALS JOINT GAP

ENTER GRID

LIQUID

50e-6

LINEAR

20

ENTER GRID

FCC

20e-3

geo

100

1.2

ENTER PHASES FOR EACH REGION

ENTER PHASE

ACTIVE

LIQUID

MATRIX

liquid

ENTER PHASE

ACTIVE

FCC

MATRIX

fcc_a1

ENTER COMPOSITION OF Ni AND B FOR EACH REGION, USE FM COMPOSITION FOR LIQUID

ENTER COMPOSITION

LIQUID

LIQUID

ni

w-p

B

LINEAR

3.1

3.1

ENTER COMPOSITION

FCC
FCC_A1
ni
w-p
B
LINEAR
0
0

@@ SET SIMULATION TIME AND INCREMENTATION
@@

set-simulation-time
1e9
YES

1E-07
1E-07

@@ SET SIMULATION CONDITIONS TO TRACK POSITION OF SOLID/LIQUID INTERFACE
@@

s-s-c
0
1
2
Y ACTIVITIES
YES
YES
1.0
2
NO
YES

@@ SAVE MODEL
@@

save
set-inter

@@ RUN SIMULATION
@@

sim

@@ SWITCH TO POST-PROCESSOR TO VIEW RESULTS (COPY/PASTE SEPARATELY)
@@

post

@@ SET DIAGRAM AXIS - TIME VS. POSITION OF INTERFACE (LOG SCALE)
@@
s-d-a x time
s-d-a y position-of-interface fcc lower
s-a-type x log

@@
@@ PLOT RESULTS
@@

plot

@@
@@ USE TOOLS TO ZOOM IN ON PLOT TO FIND TIME WHERE POSITION OF INTERFACE = 0
@@ THIS IS THE TIME FOR COMPLETE ISOTHERMAL SOLIDIFICATION
@@ REPEAT SIMULATION USING DIFFERENT JOINT GAP, BRAZING TEMP, OR FM/BM COMPOSITION
@@