**Development and Characterization of the Resulting Microstructure of a Technique
for Wide Gap Brazing Superalloy IN-738**

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of the requirements for the degree of
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Abstract

Wide gap brazing is a common process to use for superalloy repair and joining when the base alloy composition makes it difficult to weld. The current limitation on the technology is the poor mechanical properties that result from a highly heterogeneous microstructure. A procedure was established where by using the tape casting process, braze inserts were produced to fill a fixed volume in a manner representative of an actual repair application and producing a sample that would be suitable for future mechanical testing. After brazing the samples, they were sectioned and analyzed to determine their suitability for future mechanical testing and the resulting microstructure characterized. It was found that the process for producing the joints was not reliable enough to be used to produce large amount of mechanical samples.

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List of Acronyms

ADB	Activated Diffusion Bonding
ADH	Activated Diffusion Healing
AWS	American Welding Society
BSE	Back Scattered Electrons
EDS	Energy (Electron) Dispersive Spectroscopy
FCC	Face Centered Cubic
GB	Grain Boundary
HAZ	Heat Affected Zone
LPS	Liquid Phase Sintering
MPD	Melting Point Depressant
ODS	Oxide Dispersion Strengthening
PFZ	Precipitate Free Zone
PH	Precipitation Hardening
SEM	Scanning Electron Microscope
SSH	Solid Solution Hardening
TLP	Transient liquid phase
TMF	Thermomechanical Fatigue
WDS	Wide Angle Energy Dispersive Spectroscopy
WGB	Wide Gap Brazing

List of Symbols

B	Braze
C	Composition
$C_{1,2,3}$	Intermediate Composition
C_F	Filler Composition
C_L	Liquidus Composition
C_S	Solidus Composition
E	Eutectic Reaction
F	Filler
I	Insert
L	Liquid
m	Mass
m_B	Braze alloy mass
m_F	Filler alloy mass
M	metal
r_B	Braze ratio
S	Solid
T	Temperature
T_B	Brazing Temperature
T_L	Liquidus Temperature

T_s	Solidus Temperature
x	Radial position
β	Beta
γ	Gamma
γ'	Gamma prime

Chapter 1. Introduction

The goal of this investigation was to reproduce the commercially available wide gap brazing (WGB) superalloy process and characterize the resulting microstructure.

Superalloys are structural alloys engineered to achieve superior properties at high temperatures for use in applications such as gas turbine engines. The aggressive superalloy operating environment and high manufacturing cost have led to a well developed component repair industry. While the most widely used technique for joining and repair is welding, alloy composition can be a limiting factor. For alloys that are not easily welded, alternative techniques such as brazing are alternatives when the defects to be repaired are small or in non-critical locations. Repair of larger defects in critical locations is the current focus of advancements in repair technology. Wide gap brazing (WGB) is a technique that permits repairing large defects but is currently limited by the poor mechanical properties of the added material. This investigation seeks to reproduce the processes of commercial repair services as a stepping stone to developing a more advanced repair technique.

As will be explained in the following sections, the current limitations on WGB lie in the joint microstructure, which is a result of the composition required to execute the repair.

The long term objective of this research program is to develop a WGB repair process that can produce a joint with environmental resistance and mechanical properties equaling or exceeding that of the base metal.

Chapter 2. Theory

When adding material to a component, for joining or repair purposes, the ideal process will result in added material that is indistinguishable from the original component material. However, this is hard to achieve and there often is a discontinuity between the original material and the added material. Since this investigation will be considering a repair process to repair a specific alloy (IN-738), it is important to start considering the properties of the base alloy. The first part of the theory section will introduce the characteristic composition and microstructure of IN-738 and why its composition makes it hard to weld.

Following sections will show the evolution of repair processes (brazing and transient liquid phase bonding) that led to wide gap brazing (WGB). The WGB solidification mechanics and microstructure will then be discussed.

2.1 Superalloys

The alloy chosen for this investigation, IN-738, belongs to a group of metal alloys called superalloys, which are “...alloys based on Group VIII-A (Fe, Ni, Co) elements developed for elevated-temperature service which demonstrate combined mechanical strength and surface stability [Sims, 1987].” When compared to other alloying systems (Figure 1) it is

clear that superalloys have a superior combination of surface stability (oxidation resistance) and load carrying capacity (stress rupture capacity). This leads to the use of superalloys in applications where components will withstand significant mechanical loads at high temperatures, the most common example being the combustion and hot gas path section of gas turbine engines.

Figure 1. Relative capabilities of alloy systems [Sims, 1987]

The characteristic components of superalloy microstructure are a face centered cubic (FCC) γ matrix phase, rich in the alloy's main alloying element (Fe-Ni, Ni or Co) and a distribution of one or more secondary phases. The FCC arrangement of the matrix phase is advantageous for high temperature properties, since the close-packed arrangement hinders diffusion through the crystal structure, improving long-term alloy stability.

Superalloys can be classified by their main alloying element as Fe-Ni, Co and Ni-base superalloys. These can be further subdivided according to the main strengthening mechanisms present in the alloy, which can be precipitation hardening (PH), solid solution hardening (SSH), carbide strengthening or oxide dispersion strengthening (ODS). A thorough review of each of these strengthening mechanisms is provided by [Sims, 1987]. IN-738 is a PH Ni-base superalloy, which as seen in Figure 2 is an alloy group that stands out due to its higher mechanical properties at high temperature.

Figure 2. Comparative Strengths of Different Superalloy Families [Donachie, 2002]

In addition to the matrix phase element, superalloys usually have a high content of other elements that enable them to achieve the desired properties. For Ni-base alloys, a summary of the alloying elements and their role is given in Table 1.

Table 1. Role of elements in Ni-base superalloys [Vander and James, 1978]

Effect	Element
Solid Solution Hardening	Co, Cr, Fe, Mo, W, Ta
Carbide formation: -MC -M ₇ C ₃ -M ₂₃ C ₆ -M ₆ C	W, Ta, Ti, Mo, Nb Cr Cr, Mo, W Mo, W
Precipitation Hardening (γ')	Al, Ti
Raise γ' Solvus Temperature	Co
Oxidation Resistance	Al, Cr
Sulfidation Resistance	Cr
GB strengthening	B
GB Segregation (undesirable)	B, C, Zr
TCP phase (σ , μ or laves) formation (undesirable)	Cr, Co, Re, Mo, W

The main strengthening mechanism of PH Ni-base superalloys is the precipitation of γ' -Ni₃(Al, Ti), or other precipitate, in the Ni-rich γ matrix [Ross and Sims, 1987]. In addition to γ' there usually are other significant secondary phases present in the alloy (Table 2), although some phases, such as the topologically close packed (TCP), are undesirable and detrimental to the mechanical properties. In this investigation, the phases that will be focused on are the γ matrix and the carbo-boride phases M_x(C_yB_z)

Table 2. Major Secondary Phases in PH Ni-base Superalloys (Adapted from [Vander and James, 1978], [Donachie, 2002])

Phase	Formula	Comments
γ'	$\text{Ni}_3(\text{Al, Ti})$	<ul style="list-style-type: none"> Provides precipitation hardening for the matrix phase Shape varies from spherical to cubic; size varies with exposure time and temperature (Most modern alloys have a cuboidal γ' distribution) Variations in Mo content and in the Al/Ti ratio can change the morphology of γ'. With increasing γ/γ' mismatch, the shape changes in the following order: spherical, globular, blocky, cuboidal.
η	Ni_3Ti (no solubility for other elements)	<ul style="list-style-type: none"> Found in alloys with high Ti/Al ratios after extended exposure May form intergranularly in a cellular form or intragranularly as acicular platelets in a Widmanstätten pattern.
MC	TiC NbC HfC	<ul style="list-style-type: none"> Variable composition, TiC has some solubility for Ni, Zr and Mo Appears as globular, irregularly shaped particles that are grey to lavender; "M" elements can be Ti, Ta, Nb, Hf, Th, or Zr.
M_{23}C_6	Cr_{23}C_6 $(\text{Cr, Fe, W, Mo})_{23}\text{C}_6$	<ul style="list-style-type: none"> Precipitation morphology is important; can precipitate as films, globules, platelets, lamellae, and cells Usually forms at grain boundaries "M" element is usually Cr, but Ni-Co, Fe, Mo and W can substitute.
M_6C	$\text{Fe}_3\text{Mo}_3\text{C}$ $\text{Fe}_3\text{W}_3\text{C}$ - $\text{Fe}_4\text{W}_2\text{C}$ $\text{Fe}_3\text{Nb}_3\text{C}$ $\text{Nb}_3\text{Co}_3\text{C}$ $\text{Ta}_3\text{Co}_3\text{C}$	<ul style="list-style-type: none"> Randomly distributed carbide "M" elements are generally Mo or W; there is some solubility for Cr, Ni, Nb, Ta and Co.
M_7C_3	Cr_7C_3	<ul style="list-style-type: none"> Generally observed as a blocky intergranular shape
M_3B_2	Ta_3B_2 V_3B_2 $\text{Nb}_3\text{B}_2(\text{Mo, Ti, Cr, Ni, Fe}_3\text{B}_2)$ Mo_2FeB_2	<ul style="list-style-type: none"> Observed in alloys with 0.03 wt% B or greater Appearance similar to carbides, but are not attacked by preferential carbide etchants; "M" elements can be Mo, Ta, Nb, Ni, Fe or V.
μ	Co_2W_6 $(\text{Fe, Co})_7(\text{Mo, W})_6$	<ul style="list-style-type: none"> Generally observed in alloys with high levels of Mo or W Appears as coarse, irregular Widmanstätten platelets; forms at high temperatures.
σ	FeCr FeCrMo CrFeMoNi CrCo CrNiMo $(\text{Ni, W})(\text{Cr, Re})$	<ul style="list-style-type: none"> TCP phase Appears as irregularly shaped globules, often elongated; forms after extended exposure between 540 and 980°C

2.1.1 IN-738

IN-738 is the corrosion resistant evolution of alloy IN-713. The microstructure of IN-738 is typical of PH Ni-base superalloys: a γ -Ni matrix containing a γ' dispersion whose size, shape and distribution are controlled through heat treatment (a typical heat treatment is solutionizing for 2h at 1120°C followed by ageing for 24h at 620°C [Kattus, 1984]). In addition to PH, the alloy's other significant strengthening mechanisms are SSH of the γ -Ni phase by Cr, Mo, W and Co and grain boundary (GB) strengthening by boride and carbide precipitates [Kattus, 1984]. Its high strength makes it hard to forge and it is mostly cast as polycrystalline alloy for the fabrication of gas turbine components such as buckets, nozzles and turbine wheels with service temperatures up to 980°C.

The chemical compositions of the alloys IN-713, IN-738 and IN-738LC are given in Table 3. Notably, IN-738 has a higher Cr content than IN-713. The role of Cr in IN-738 is critical for this investigation since it contributes both to the environmental and mechanical properties of the alloy. Environmentally, chromium forms an adherent surface scale that is hot corrosion and, to a lesser extent, oxidation resistant [Pettit and Giggins, 1987]. Mechanically, the element is present in solution in the γ -Ni phase, where it provides SSH and forms strengthening grain boundary (GB) precipitates. High levels of Cr, in combination with high levels of refractories (W, Mo, Ta, Nb), can lead to the formation of TCP phases, which is why the higher Cr level of IN-738 (when compared to IN-713) is compensated with lower Mo content. High refractory content is also undesirable since it can lead to microsegregation during solidification through rejection

of solute atoms into the liquid at the advancing solid/liquid interface, ultimately resulting in a compositional gradient and increasing the propensity for localized TCP phase formation when the component is in service.

Table 3. Composition of IN-713C, IN-738 and IN-738LC (wt%) [Kattus, 1984], [Donachie 2002]

<i>Element</i>	IN-713C	IN-738		IN-738LC	
		<i>Min.</i>	<i>Max.</i>	<i>Min.</i>	<i>Max.</i>
C	0.12	0.15	0.20	0.09	0.13
Co		8.00	9.00		
Cr	12.5	15.70	16.30		
Mo	4.2	1.50	2.00		
W		2.40	2.80		
Ta	1.75	1.50	2.00		
Nb	0.9	0.60	1.10		
Al	6	3.20	3.70		
Ti	0.8	3.20	3.70		
Al+Ti		6.50	7.20		
B	0.012	0.005	0.015	0.007	0.012
Zr	0.1	0.05	0.15	0.03	0.08
Fe		-	0.05		
Mn		-	0.02		
Si		-	0.03		
S		-	0.015		
Ni	Bal.	Bal.			

Cobalt behaves similarly to Ni and generally forms part of the γ -Ni matrix, providing SSH and improved corrosion resistance. The refractories in IN-738 (Mo, W and Ta) are present mainly to provide SSH of the matrix phase but can also form part of strengthening GB precipitates.

The three trace elements in IN-738 (B, C, Zr) are present in carefully controlled levels to form strengthening precipitates at the GBs. Low carbon versions of IN-738 (C and LC) are available to facilitate welding, since the presence of C can lead to Cr depletion in the GBs through sensitization.

2.1.2 Welding IN-738

When selecting a process to repair or join a component there is a wide range of techniques available [Spinat and Honnorat, 1986], however the most commonly used technique is welding. A photograph of a weld-repaired superalloy gas turbine component is shown below.

Figure 3. Weld repairs on large industrial gas turbine first stage nozzle (FSX-414)

However, there are drawbacks to welding that limits its use on PH Ni-base superalloys:

- Process produces a weld seam with a re-cast microstructure, a material discontinuity with respect to the base alloy. This re-cast region generally has

higher microsegregation which can lead to TCP phase precipitation and reduced oxidation and corrosion resistance due to element depletion [Simms, SAII].

- The heat affected zone (HAZ) may exhibit excessive grain growth and solutioning of secondary phases (e.g. γ' and carbides) that may re-precipitate in detrimental morphologies.
- Localized heat input in the weld and HAZ results in localized γ' dissolution and reprecipitation. The volumetric changes associated with these localized reactions lead to strains and residual stresses, which in turn cause hot cracking during welding or strain-age cracking during postweld heat treatment [Kattus, 1984], [Sidhu et al., 2005].

This last point is of special consideration for the alloy composition, since the residual stresses and strains are proportional to the γ' volume fraction. Therefore, the combined content of γ' formers, Al and Ti, can be used as a guideline to determine weldability (Figure 4) since these are the main γ' formers. Welding is generally not recommended or feasible for alloys with a Al + Ti content greater than 6% [Sidhu, 2005]. IN-738 has an Al + Ti content of 7% and is generally considered unweldable, requiring the use of other techniques.

Figure 4. Weldability of Ni-based superalloys based on Al and Ti content [Henderson et al, 2002]

2.2 Conventional Brazing

Often used as an alternative to welding, brazing is defined by the American Welding Society (AWS) as “...a group of welding processes which produce coalescence of materials by heating them to a suitable temperature and by using a braze alloy having a liquidus above 450°C and below the solidus of the base materials. The braze alloy is distributed between the closely fitted surfaces of the joint by capillary attraction.” [AWS, 1976]. It differs from soldering only in that the process is carried out above 450°C.

Figure 5 shows the brazing process schematically in three steps:

1. A reservoir of braze alloy is placed adjacent to the volume to be filled and heat is applied until the braze alloy melts.
2. Temperature is maintained and held at the brazing temperature (T_B) as the molten alloy infiltrates the volume through capillary attraction until fill is complete or the reservoir is exhausted. A metallurgical bond is produced by erosion of the base alloy surface through element diffusion at the solid/liquid interface.
3. The joint is cooled and solidification is achieved by cooling of the liquid braze alloy.

Figure 5. Schematic of the conventional brazing process

Brazing temperature is a key consideration in the process. Generally, it is low enough so that incipient melting does not occur in the base alloy during brazing, but high enough to eliminate the risk of melting the added material when the component is in service (this occurs when the solidus of the braze alloy is below the service temperature).

Because of limited element diffusion, the composition (hence the melting point) of the added material is dependent almost exclusively on the composition of the braze alloy. As will be shown in the following section, the composition of the braze alloy is the limiting factor in using conventional brazing for superalloy repair.

2.2.1 Braze Alloys

When selecting (or designing) a braze alloy composition, there are several key considerations:

- Melting point - The braze alloy must have a liquidus temperature (T_L) lower than the solidus temperature (T_S) of the base alloy, ensuring complete braze alloy melting while avoiding melting of the base alloy. The braze alloy's solidus must be high enough to eliminate the risk that the added material could melt when the finished component is in-service.
- Flow - At the brazing temperature, the liquid alloy must have high fluidity to infiltrate all areas of the volume to be filled while retaining sufficient surface tension for capillary attraction to drive the flow. When selecting the braze alloy, the balance between fluidity and surface tension is reflected in the difference between solidus and liquidus temperatures (solidification range). Alloys with a large solidification range flow sluggishly and bridge large gaps. A narrow

solidification range results in flow that penetrates narrow gaps and crevices more easily, with eutectic alloys having maximum fluidity. An additional condition for capillary flow is that the liquid braze alloy must wet the base alloy, requiring compatibility between both alloys. This is often ensured by using a braze alloy where the main alloying element is the same as that of the base alloy.

- Final properties – Because the braze alloy composition is the key driver of the final properties of the added material, the braze alloy must have some of the same alloying elements that are present in the base alloy for it to achieve the desired properties (Table 1).

The solidus and liquidus behavior of the braze alloy is modified by the use of an alloying element called melting point depressant (MPD). The simplest braze alloys are binary alloys of a base metal and an MPD. For Ni-base braze alloys, the MPD is generally B, Si and/or P. The compositions of some simple Ni-base superalloy brazing alloys are given in (Table 4). The highlighted eutectic alloy, BNi-9, is the one that was used for this investigation.

Table 4. Properties and Composition of some AWS Brazing Alloys [AWS, 1976]

Alloy	T _S (°C)	T _L (°C)	T _B (°C)	Alloying element (Wt%)					
				Ni	Cr	B	Si	Fe	P
BNi-1	977	1038	1066-1204	Bal.	13-15	2.75-3.5	4.0-5.0	4.0-5.0	
BNi-2	971	999	1010-1177	Bal.	6-8	2.75-3.5	4.0-5.0	2.5-3.5	
BNi-3	982	1038	1010-1177	Bal.		2.75-3.5	4.0-5.0		
BNi-4	982	1066	1010-1177	Bal.		1.5-2.2	3.0-4.0	1.5	

Alloy	T_s (°C)	T_L (°C)	T_B (°C)	Alloying element (Wt%)					
				Ni	Cr	B	Si	Fe	P
BNi-5	1079	1135	1149-1204	Bal.	18.5-19.5		9.75-10.50		
BNi-6	877	877	927-1093	Bal.					10.0-12.0
BNi-7	888	888	927-1093	Bal.	13-15				9.7-10.5
BNi-9	1055	1055	1065-1205	Bal.	13	3.5			

Notably, Cr is added even in simple alloys to maintain continuity with respect to the base alloy properties as described in section 2.1. In fact, in order to attain the desired final properties, braze alloys often have alloying additions similar to superalloys. Alloying additions tend to increase the solidification range and make the braze alloy more suitable for wide gaps. For Ni-base braze alloys the limit between narrow and wide-gaps is considered to be around 100 μ m (0.004”).

The compositional factor that limits brazing of superalloys is the MPD content of the braze alloy. MPD is necessary to attain the solidification behavior required for the brazing process, but its presence is detrimental to the final properties due to the following reasons:

- MPD will promote the formation of eutectic structures, which do not have desirable properties in Ni-base superalloys (low ductility, low incipient melting temperature, poor resistance to crack propagation).
- MPD diffusion into the base alloy can depresses the base alloy’s solidus and lead to incipient melting when the component is in-service.

- The amount of MPD required to sufficiently depress the solidus of the braze alloy is usually much higher than the MPD solubility in the γ -Ni matrix phase and results in precipitate formation before and during cooling from the braze temperature.

The last point is the main concern when considering the properties of brazed components. The MPD content in excess of the matrix phase solubility results in the formation of brittle intermetallic compounds, either within the matrix or between grains. These precipitates are undesirable because:

- Precipitates that form preferentially at the grain boundaries can act as paths for crack propagation. This is of particular concern when the precipitates form long networks.
- Precipitates can act as crack initiation points.
- Precipitates with high Cr content can cause localized Cr depletion leading to decreased corrosion resistance

Intermetallic precipitation can be mitigated by prolonging time at the brazing temperature or performing post-brazing heat treatments to break up extensive intermetallic networks and round blocky precipitates. However, heat treatments represent an additional manufacturing step that may cause excessive grain growth in the base metal [Stankowski, 2002] or simply not be economical.

Mitigating the effects of the necessary MPD content is the main challenge in brazing.

Since this investigation will focus on the use of B as the MPD, the following section will discuss the compounds formed by B in superalloys.

2.2.2 Boron and Borides

The reason for using B as the MPD over Si and P is that its smaller atomic radius results in higher diffusivity through the γ -Ni phase, decreasing the times and temperatures required to mitigate intermetallic precipitation [Duvall et al, 1974]. This will be especially useful for the diffusion-type solidification of the WGB process.

The disadvantage of B (and this is also true for Si and P) is low solubility of B in γ -Ni [Teppo and Taskinen, 1993]. This thorough review of the Ni-B phase diagram shows negligible solubility of B in γ -Ni. Even at the low alloying level of B in IN-738 (0.005-.015 wt%), B will manifest itself as GB precipitates.

Since the B concentration in braze alloys ranges from 1.5 - 3.5 wt% (Table 4), boron containing intermetallic precipitates are almost unavoidable. Techniques such as Rapid Solidification Processing have been shown to increase the solubility of B in γ -Ni to 0.8 wt% [Shrivastava et al., 1989] but the cooling rates required (10^5 - 10^7 °C/s) are not feasible during brazing.

For the Ni-Cr-B system, borides can form in a fine distribution within a γ -Ni matrix (γ -Ni / Ni_3B eutectic phases) and discrete brittle hard phases with Cr_xB_y composition [Stankowski, 2002].

Considering the equilibrium phase diagram (Figure 6) and invariant reactions (Table 5) for the Ni-Cr-B system, the brazing alloy composition used in this investigation is indicated by point E_2 and corresponding invariant reactions E_2 and E_2' . From these invariant reactions, it can be expected that the braze alloy will form either Cr_5B_3 and/or CrB precipitates upon solidification in addition to the γ -Ni / Ni_3B eutectic phases.

Figure 6. Theoretical Ni-Cr-B liquidus projection with isothermal contours in the Ni-Cr-B system
[Campbell and Kattner, 2002]

Table 5. Ni-Cr-B System Invariant Reactions [Campbell and Kattner, 2002]

Phase Transformation		Liquid Composition (Wt%)			Temp. (°C)
		<i>Ni</i>	<i>Cr</i>	<i>B</i>	
$L \rightarrow Cr_3B_4 + Ni_2B + Ni_3B$	E_1	Bal.	1.4	7.9	1105
$L \rightarrow CrB + Ni_2B + Ni_3B$	E_1'	Bal.	1.8	7.9	1103
$L \rightarrow Cr_5B_3 + Ni_3B + \gamma-Ni$	E_2	Bal.	14.1	4.3	1100
$L \rightarrow CrB + Ni_3B + \gamma-Ni$	E_2'	Bal.	23.9	4.2	1080
$L \rightarrow Cr_2B + bcc-Cr + \gamma-Ni$	E_3	Bal.	59.0	2.0	1222
$L + Cr_5B_3 \rightarrow CrB + \gamma-Ni$	U_1'	Bal.	0.1	5.6	465
$L + Cr_5B_3 \rightarrow CrB + Ni_3B$	U_2	Bal.	5.4	6.4	1127
$L + Cr_3B_4 \rightarrow CrB + Ni_3B$	U_3	Bal.	4.3	6.9	1124
$L + Cr_2B \rightarrow Cr_5B_3 + \gamma-Ni$	E_1'	Bal.	33.8	3.3	1180

While a good first approximation, the equilibrium phase diagram of the Ni-Cr-B system cannot be used to predict the interaction of B with elements other than Ni and Cr present in the base alloy and the effects of non-equilibrium solidification. For this, it is necessary to study the microstructures of the joints.

Boride compositions that have been observed in Ni-base alloys are given in Table 6. In general, boron tends to form compounds with Cr, Mo, W and Ni. It has been suggested that addition of Ti to the braze alloy reduces the Cr depletion associated with Cr_xB_y precipitation by promoting the formation of TiB_2 [Shrivastava et al., 1989]

Table 6. Boride Types Commonly Found in Ni-base Alloys [Shrivastava et al., 1989], [Teppo and Taskinen, 1993]

Boride	Crystal Structure	Type
$M_{23}B_6$	FCC	
Cr_5B_3	Tetragonal	
MoB_4	Hexagonal	
NiB	Orthorhombic	CrB
TiB ₂	Hexagonal	AlB ₂
Ni ₂ B	Tetragonal	CuAl ₂
Ni ₃ B	Orthorhombic	Fe ₃ C
Ni ₄ B ₃ (m)	Monoclinic	Ni ₄ B ₃ (m)
Ni ₄ B ₃ (o)	Orthorhombic	Ni ₄ B ₃ (o)
CrB	Tetragonal	
Cr ₂ B ₃	Orthorhombic	

During brazing, borides will form as eutectic (γ -Ni / Ni₃B) and brittle hard phases such as chromium carboborides (e.g. Cr(B,C)) and refractory borides with Cr, W and Mo [Stankowski, 2002]. Once boride phases formed they are morphologically stable and have little variation in chemistry [Iacocca, 1996], [Lal et al., 2000]. This is evidenced by the high solvus temperature (1210°C) of phases such as Cr₂B.

More complex borides can also form, with $M_{23}B_6$ -type borides having been identified in alloys with 3.5 wt% B [Barbassat et al, 1986]. These borides have a high Ta content compared to the matrix γ -Ni phase. A smaller fraction of the borides formed were identified as Cr₅B₃, poor in Ta and rich in Mo. It is worth noting that at the B levels studied, the boride volume fraction ranged from 58 to 70%.

Needle-like W-Cr-Ni boride precipitates have been observed at the interfaces of PH Ni-base superalloy TLP joints due to diffusion of B into the base alloy [Miglietti, 1993]. In

the braze region itself, two distinct borides are observed, Cr-rich and Ni-Cr-Co-Al-W rich.

It should be noted that identifying the stoichiometry of B-containing phases is difficult since boron can only be detected qualitatively using energy dispersive spectroscopy (EDS) analysis. Often, precipitates are identified as borides indirectly through the presence of other elements [Idowu, Richards and Chaturvedi, 2005]. An additional complication is that boron can substitute for carbon in carbides and vice-versa. Since both borides and carbides have similar composition, morphology and effects on alloy properties, this investigation will not differentiate between the two.

2.2.3 Limitations of Conventional Brazing

Due to the poor mechanical properties that result from the intermetallic precipitation, brazing is currently limited to repair of short cracks in non-critical locations [Heikinheimo et al, 2005]. Consequently, developments in the field of superalloy braze repair have been focused on improving the mechanical properties of the repaired region [Stankowski, 2002]. One way of achieving this is by limiting intermetallic precipitation through a process called transient liquid phase (TLP) bonding, which is a precursor to WGB. Therefore, a short review of TLP bonding is pertinent.

2.3 *Transient Liquid Phase Bonding*

The TLP process relies on solid-state diffusion to prevent the formation of undesirable precipitates. The process can be used to form joints where the joint is indistinguishable from the base alloy. An excellent overview of the process is given by [Gale and Butts, 2004].

Similarly to conventional brazing, in the TLP process a braze alloy is used to bond base alloy components. Unlike conventional brazing, solidification occurs isothermally at the brazing temperature (i.e. solidification does not occur upon cooling). High temperature is important for the diffusion processes that minimize intermetallic formation. Referring to Figure 7, the TLP process occurs as follows:

- a) Initial conditions - A thin layer of braze alloy (interlayer) is placed between the surfaces of the components to be joined.
- b) Initial solid-state diffusion - At the brazing temperature the joint consists of an MPD-rich liquid braze alloy layer between two solid base alloy surfaces (either poor or devoid of MPD). The compositional gradient drives a diffusion process where MPD diffuses into the base alloy until solidus composition is reached at the interface.
- c) Solid phase dissolution – Continued MPD diffusion dissolves the solid's surface, increasing the joint liquid volume. The liquid volume will increase until the liquid achieves the liquidus composition at the brazing temperature.

- d) Isothermal solidification - The MPD continues to diffuse from the solid/liquid interface into the bulk of the base alloy. The concentration of MPD in the liquid phase cannot be less than the liquidus composition. Therefore, liquid undergoes an isothermal phase transformation from liquid into solid, the liquid maintaining the liquidus composition and the solid forming with solidus composition.
- e) End of isothermal solidification – Solidification continues until the volume of the liquid phase is reduced to zero and solidification of the joint is complete. This can only be achieved if there is sufficient base alloy for the MPD to diffuse into and leave a final MPD concentration less than the solidus composition. It is important to note that if cooling occurs before solidification is complete, the remaining liquid will not undergo the desired phase transformation and will form one or more intermediate phases in addition to a final eutectic structure.
- f) Solid-state homogenization - After solidification is complete, the joint is maintained at a high temperature (which may be lower than the brazing temperature) so that solid-state diffusion decreases MPD concentration at the joint centreline (where MPD concentration is highest) to an acceptable level. This step is intended to avoid formation of precipitates at the joint centerline.

Figure 7. TLP Process schematic [Tseng *et al.*, 1999] from [Tuah-Poku *et al.*, 1988]

In theory, the process results in a precipitate free joint. In practice, interactions between the MPD and other alloying elements in the base alloy often result in precipitate formation in the base alloy (Figure 8).

Figure 8. SEM image of a superalloy TLP joint [Arafin et al, 2007]

Since the isothermal solidification process relies on solid-state diffusion, it is strongly dependent on time and temperature to achieve the desired joint features of eutectic and precipitate free. Increasing either time or temperature is not always possible since the upper limit for temperature is incipient melting of the base alloy and long exposure to high temperature is undesirable since it can lead to excessive grain growth in the base alloy as well as increase processing costs.

An alternative for ensuring sufficient diffusion to achieve the desired microstructure is to minimize the amount of diffusion required, either by minimizing MPD content or diffusion distance. This is achieved by using thin braze inserts, where the upper limit for interlayer thickness in TLP joints is 50 μm [Gale and Butts, 2004]. This limitation on interlayer size is the main disadvantage of the TLP process. As the table below shows,

long times at the brazing temperature are required to achieve complete isothermal solidification and a precipitate free joint.

Table 7. TLP Parameters using Ni-15Cr-4B

Base Alloy	Gap (μm)	T _B (°C)	Time (h)	Comments	Source
IN-738LC	100	1175	1	Isothermal solidification not achieved	1
Mar-M247	100	1150	1/3	Continuous centerline precipitates	2
Mar-M247	100	1150	1	Grain nucleation at interface	2
Mar-M247	100	1150	6	Homogenous microstructure	2
Mar-M247	100	1200	1	Continuous centerline precipitates	2
Mar-M247	100	1200	3	Discontinuous centerline precipitates	2
Pure Ni	30	1100	1	Isothermal solidification not achieved	3
Pure Ni	30	1200	1	Almost isothermal solidification (0.5 μm eutectic layer)	3
1 - [Idowu, Richards and Chaturvedi, 2005]					
2 - [Miglietti, 1993]					
3 - [Ohsasa, Shinmura and Narita, 1999]					

For the alloying system used in this investigation, IN-738 with a Ni-15Cr-4B braze alloy, it is important to highlight the results of [Idowu, Richards and Chaturvedi, 2005], where a 100 μm interlayer did not achieve isothermal solidification when brazed for 1h at 1175°C. Figure 9 shows the precipitates and eutectic structures that form when the joint is cooled before isothermal solidification is complete.

Figure 9. SEM micrograph of 100 μm joint (IN-738LC/Ni-150) bonded for 1h at a) 1130°C, b) 1145°C, c) 1160°C, d) 1175°C [Idowu, Richards and Chaturvedi, 2005]

2.4 Overview of Wide Gap Brazing

In WGB, the same solidification mechanics and principles of TLP (namely MPD diffusion) are used to bridge larger gaps, applicable to both component joining and repair. The process was developed during the early 1970's and has been reported with several different names, such as Activated Diffusion Bonding (ADB) by General Electric [Hoppin and Berry, 1970], Liquid Phase Diffusion Sintering [Jahnke and Demny, 1983],

Transient Liquid Phase Sintering (TLPS) [Tandon and German, 1994], Activated Diffusion Healing (ADH) by General Electric [Pallos 2001] and Liburdi Powder Metallurgy (LPM) [Ellison et al, 1994].

Like in TLP, a layer of material is pre-placed between the surfaces to be joined. However, this layer of material (brazing insert) is composed of a mixture of a brazing alloy and a filler alloy (both usually in powder form). In some applications, the insert is completely devoid of brazing alloy and the brazing alloy supplied through an external reservoir, as shown schematically in Figure 10. The simplified process shown starts with the application of heat until the brazing alloy melts and infiltrates the brazing insert. The solidification mechanics of WGB are identical to those of TLP brazing, with MPD diffusion from the liquid into the solid phase results in isothermal solidification. The major difference is that solute diffuses into the filler alloy in the brazing insert as well as the base alloy. The filler acts as a sink for the MPD, reducing the diffusion distance since there is material in the joint that can accommodate additional MPD content. By reducing diffusion distance, solidification and homogenization can be achieved in a shorter time. Having the filler in powder form greatly increases the liquid/solid interface area, which reduces the time required for isothermal solidification by more than two orders of magnitude when compared to TLP bonding [Zhuang and Eagar, 1997]. When the brazing alloy is not contained in the brazing insert, the filler alloy in the insert also provides a physical structure containing capillary paths for the brazing alloy to flow into the joint.

Figure 10. Schematic of WGB Joint

2.4.1 Properties of WGB Joints

When performed correctly, the WGB process can produce joints with high tensile strength, equal to or above that of the base metal with acceptable ductility [Lugscheider and Schittny, 1988], [Fritsche, Satir-Korloz, Baumann, 2000], [Ellison et al, 1994]. A property of the WGB microstructure is that the distribution of the original filler powder particles gives the material properties, namely the stress-rupture behavior [Ellison et al, 1994], similar to the base-metal with a fine grain structure. Fine grain structure is also known to improve superalloy resistance to thermomechanical fatigue by delaying crack initiation [Stevens and Flewitt, 1980], [Vander and James, 1978].

Strength in WGB is generally not a concern, but ductility and cracking resistance are. Porosity, precipitates and eutectic structures have been documented to act as initiation

points for failure [Lugscheider and Schittny, 1988]. In terms of crack propagation, eutectic structures and intermetallic precipitate chains are known to be preferential paths for crack propagation in WGB joints [Yu and Lai, 1995], [Su, Chou, Chang, Liu, 1999]. Studies of superalloys in general have shown that for thermomechanical fatigue resistance, carbide type and distribution critically influences crack propagation behavior [Woodford and Mowbray, 1974], [Vander and James, 1978]. Long networks of grain boundary $M_{23}C_6$ carbides are undesirable because they provide a path for propagation. Blocky MC carbides are more effective in impeding propagation. If this is true for carbides, it is likely also true for the borides discussed in this investigation.

2.5 WGB Solidification Mechanics

Reviews of the process can be found in [Jahnke and Demny, 1983], [Corbin, 2002] and others, but will also be presented here. The WGB model that will be presented has the following simplifying assumptions:

- Filler and braze alloys are both binary alloys of the same base and MPD combination. Composition is defined in terms of MPD concentration as C_x .
- The model considers a single filler powder particle surrounded by a liquid braze alloy reservoir of finite size at the brazing temperature.
- The filler powder particle has no internal grain boundaries.

- MPD diffusion in the liquid phase is infinitely fast and there is no compositional gradient in the liquid.
- The relative amounts of braze and filler alloys will allow isothermal solidification at the brazing temperature. The validity of this assumption will be revisited later (Section 2.5.1).

The initial conditions are shown in Figure 11. The solid filler powder particle is surrounded by liquid as shown in the top-right corner. The solid has composition C_F , which corresponds to a continuous γ -phase microstructure at the brazing temperature (T_B) as shown in the phase diagram to the left. The braze alloy, with composition C_B , is completely liquid at the braze temperature. The chart on the right shows the compositional gradient from the centre of the powder particle to the finite end of the liquid reservoir (x_L). The solid/liquid interface position is designated x_i and at the start of the process is designated x_0 . It is important to note that the amount of MPD present in the system is finite.

Figure 11. WGB Process - Initial Conditions

The compositional gradient at the solid/liquid interface drives MPD diffusion from the liquid into the solid phase (Figure 12). Since there is a finite amount of MPD and the solid phase is becoming enriched in MPD, the composition of the liquid changes from the initial C_B to C_1 . A compositional gradient starts developing in the solid with the composition at the interface increasing from C_F to C_S , the solidus composition.

Figure 12. WGB Process - Initial solid-state diffusion

When the MPD concentration in the solid at the interface reaches C_S , further MPD diffusion results in dissolution of the solid's surface (Figure 13). The solid/liquid interface will continue to recede until the liquid reaches C_L , the liquidus composition. This is the maximum liquid volume that will develop in the process.

Figure 13. WGB Process - Solid-phase dissolution

The compositional gradient at the interface continues to drive MPD diffusion into the solid; however, the liquid cannot reject more MPD and still remain liquid at the brazing temperature. This is the start of isothermal solidification (Figure 14), with solid forming with composition C_S at the interface and the liquid remaining at composition C_L .

Simultaneous to solidification at the interface, MPD solid-state diffusion redistributes the MPD throughout the solid phase and the minimum MPD concentration in the solid phase increases to C_2 .

Figure 14. WGB Process - Isothermal Solidification

Isothermal solidification continues until the last remaining liquid transforms into solid γ phase with solidus composition (Figure 15). As a result, the minimum MPD concentration in the solid reaches C_3 .

Figure 15. WGB Process - End of isothermal solidification

While it appears that the WGB process is complete, it is important to point out that the temperature is still very high. When cooling reduces the temperature to T_4 (Figure 16), the fraction of the γ phase with composition C_4 and greater will undergo a phase transformation $\gamma \rightarrow \gamma + \beta$, forming precipitates in a fraction of the final microstructure.

Figure 16. WGB Process - Precipitate formation upon cooling

While the model presented previously is fundamental for understanding the WGB process and consequently its microstructure, it is important to note some features that are not included in the model:

- The model assumes that initially there is no interaction between the filler and braze alloys as they are heated up to the brazing temperature. In practice, as the braze insert is heated, solid-state diffusion can occur between the filler and braze, which can result in isothermal solidification beginning prior to complete melting of the braze alloy [Tandon and German, 1994], [Choi et al, 2004]. This is mitigated by using high heating rates (e.g. 10-20°C/s) to minimize solid-state diffusion between braze and filler alloys or by using a braze reservoir in lieu of adding braze alloy in the insert.
- If the filler powder particles have internal grain boundaries, there are additional complexities, especially surrounding consolidation. This is described in detail by [Tandon and German, 1994].
- Diffusion of elements is not limited to the MPD diffusing into the filler and base alloy. It has been shown that when the braze alloy is low in heavy elements, Mo, W and Ta can diffuse from the base and filler alloys into the liquid phase [Tandon and German, 1994]. It was further reported that Ti (but not Al) will also diffuse from the filler into the braze [Su, Chou, Chang, Liu, 1999].
- MPD solubility in the γ -phase is so low that precipitate formation will occur in the solid phase as early as in the initial solid-state diffusion.

- The assumption of a single filler particle in a liquid reservoir does not account for the porosity of braze inserts and the mechanisms of void formation. A thorough review of these phenomena is given by [Lorenz et al, 2004] and [Lim et al., 1995].

2.5.1 Braze Insert

Because of its importance to the process, the braze insert needs to be discussed, both in terms of composition and structure. The WGB model presented in the previous section assumed that the relative amounts of braze and filler would allow isothermal solidification at the brazing temperature. For this to occur, insert composition must fall within a certain range. Insert composition, in combination with braze temperature, are the critical factors in determining the final microstructure [Lugscheider et al, 1989].

Considering the braze insert alone (i.e. neglecting interaction with the base alloy), the insert MPD concentration (C_I) will be the same as that of the added material and will depend on the mass of filler (m_F) and braze alloy (m_B) present in the insert and the MPD concentrations of the filler (C_F) and braze alloys (C_B), as shown in equation 1.

$$C_I = (m_B \times C_B + m_F \times C_F) / (m_B + m_F) \quad \text{Eqn. 1}$$

A more complex formula, accounting for insert porosity is given by [Lorenz et al, 2004].

This formula is difficult to apply practically, since it requires knowledge of the insert void volume (porosity) and the volume fractions of braze and filler alloys.

Considering the simple binary phase diagram (Figure 17) used in the solidification model, insert composition will fall in one of three areas:

1. If composition C_I is between C_F and C_S , complete isothermal solidification is achievable given sufficient time at the braze temperature. All solidification will occur in a $L \rightarrow \gamma$ reaction.
2. If composition C_I is between C_S and C_L , isothermal solidification will begin, but not be complete, independent of time at the brazing temperature. The liquid that does not solidify at T_B will solidify in a $L \rightarrow \gamma + \beta$ transformation as the sample is cooled.
3. If composition C_I is between C_L and C_B , no solidification will occur at the braze temperature. Complete dissolution of the filler alloy will occur and solidification will occur non-isothermally during cooling.

Figure 17. Ideal binary alloy phase diagram

The insert should be manufacture with a composition ideally below the solidus composition but in no case greater than the liquidus, setting an upper limit in insert composition.

In the WGB process considered in this investigation, the MPD concentration in the filler alloy is negligible ($m_F = 0$). Therefore, the previous formula can be simplified to:

$$C_I = m_B / (m_B + m_F) \times C_B \quad \text{Eqn.2}$$

For this investigation, the previous equation can be further simplified since a single braze alloy composition was used. Insert composition (and hence MPD concentration) was characterized by the braze ratio defined as:

$$r_B = m_B / (m_B + m_F) \quad \text{Eqn. 3}$$

While the braze ratio captures the amount of MPD, real superalloy WGB systems cannot be described in terms of MPD concentration alone. As previously mentioned, the braze alloy in this investigation is a ternary Ni-Cr-B alloy. However, a ternary alloy does not match the base superalloy composition. In order to maintain compositional similarity between base and insert, the filler alloy must have similar composition to the base alloy. In this investigation, the filler alloy used had the same composition as the base alloy.

The previous considerations showed there is an upper limit on braze ratio based on the solidification behavior, however, there is also a lower limit due to the insert's structure. As mentioned previously, inserts are usually made using metallic powders, resulting in inserts that are not fully dense and have some porosity. In practice, the lowest insert porosities attainable are between 30 and 40% volume, independent of the method used to produce the insert (slip casting, tape casting, compaction, sintering) [Chasteen and Metzger, 1979], [Lugscheider and Schittny, 1988], [Zhuang and Eagar, 1997]. Sintering the insert has been found to be able to reduce porosity an additional 10%.

The insert porosity means that a certain amount of liquid volume is necessary to fully infiltrate the insert and eliminate porosity in the final component, representing the lower limit of the braze ratio. While the braze alloy does not have to be present in the insert (it can be placed as a reservoir and infiltrate the insert at the braze ratio), the amount of infiltrated liquid needs to be accounted for in the braze ratio.

This makes it difficult to determine the insert braze ratio when using a braze reservoir, since the insert porosity is hard to determine. For this investigation, the braze ratio will only be calculated when using inserts with no braze reservoir.

In closing, at high insert braze ratios (60-100%), minimizing the braze ratio will minimize the amount of B, leading to improved mechanical properties [Yu and Lai, 1995]. However, lowering the braze ratio excessively leads to porosity in the final microstructure, since there is not enough liquid alloy to fill all of the voids in the braze insert. Braze ratios from 30 to 60% have been reported for superalloy WGB [Heikinheimo et al, 2005]. For the IN-738LC/BNi-9 combination used in this work, a braze ratio of 30% has been reported as the lower limit without excessive porosity [Lim et al., 1995], [Jahnke and Demny, 1983].

Table 8. Reported Solidus and Liquidus Temperatures for IN-738LC and Ni-Cr-B Powder Blends
[Jahnke and Demny, 1983]

Blend	r_B (%)	Solidus (°C)	Liquidus (°C)
100% BNi-9	100	1040	1055
60% IN-738LC / 40% BNi-9	40	1090	1225
70% IN-738LC / 30% BNi-9	30	1130	1295
70% IN-738LC / 30% BNi-9 (Sintered at 1160°C for 2h)	30	1125	1310
100% IN-738LC	0	1200	1330

2.5.2 Brazing Cycle

While the previous sections have focused on the effects of composition, the brazing cycle will also influence the microstructure. A typical brazing cycle consists of the following four steps:

1. The component will be heated from room temperature to the binder evaporation temperature. Most braze inserts use a polymer to consolidate the metal powders and it is undesirable for this polymer to form part of the final composition. Also, this material will vaporize at the brazing temperature and may create porosity in the finished component. Therefore, the component is held at a low temperature until all the binder evaporates (this can be monitored through the chamber pressure).
2. After binder evaporation is complete, the temperature will be increased to a second hold step. The purpose of this second hold step is to allow sintering between the powder particles that make up the insert, ensuring sufficient rigidity in the insert structure to provide capillary paths at the brazing temperature. This step requires balancing the need for insert rigidity and preventing excessive solid-state diffusion between braze and filler alloys that will make their behavior at the brazing temperature unpredictable.

3. After sintering is complete, the component will be quickly heated to the brazing temperature and held there for a short period of time. A high heating rate ($20^{\circ}\text{C}/\text{min}$) is recommended to minimize element diffusion between braze and filler powders prior to reaching the brazing temperature. Since the powder particles that make up the insert have a large surface area, the time required for diffusional processes to be complete is greatly reduced when compared to TLP bonding. This step tends to be between 10 and 20 minutes long (compared to over an hour for TLP bonding).
4. After isothermal solidification is complete the component can be cooled to room temperature. A homogenization-hold prior to cooling can be used to mitigate precipitate formation in the same way as it is done in TLP bonding.

The times and temperatures required for each step depend on the alloying system being considered. Similar to the conventional brazing and TLP processes, increasing time and temperature during WGB is known to improve the mechanical properties of the final microstructure [Fritsche, Satir-Korloz, Baumann, 2000], [Miglietti, 1993].

This investigation used a single brazing cycle (detailed in Section 4.2.6) and no attempts were made to modify microstructure through heat treatment.

In terms of the equipment, the preferred heating method for WGB is a furnace with a 10^{-4} to 10^{-6} torr vacuum to avoid oxide formation at the brazing temperature [Gale and Butts, 2004].

2.6 WGB Microstructure

The ideal WGB microstructure consists of a homogeneous distribution of the original filler powder particles surrounded by an isothermally solidified matrix. In practice, precipitate formation is unavoidable and precipitates will form in the remaining original solid phase of the filler powder particles and in the base alloy, both intragranularly and intergranularly (grain boundary precipitates). Since complete isothermal solidification is not always achieved, non-isothermal solid is also observed. A schematic of characteristic microstructure is shown in Figure 18.

Figure 18. Microstructure schematic (Adapted from [Su et al, 1999])

Actual WGB microstructures are more complex, as shown in Figure 19, with different precipitate morphologies, non-homogeneous distributions and voids (porosity). Referring to the micrographs from the figure, the microstructural features that will be considered in this investigation and discussed in the rest of this section are:

1. γ -Ni solid phase
2. Insert precipitates
3. Non-isothermal solid
4. Interfacial isothermal solid
5. Base alloy precipitates
6. Porosity

Figure 19. Representative superalloy WGB Microstructures [Fortuna, 2002], [Wu et al., 2001], [Pallos, 2001]

2.6.1 γ -Ni Solid Phase

The superalloy characteristic γ -Ni matrix phase is present in the final microstructure of WGB from three sources:

- The fraction of the filler powder that did not undergo dissolution
- Isothermally solidified solid formed at the braze temperature

- Eutectic structures formed from cooling liquid.

The γ -Ni phase formed in the insert region isothermally. This phase, indicated by marker (1) in Figure 19, appears as a globular light-colored structure. While these could be mistaken for the filler powder particles, it is important to note that this phase consists of both the fraction of the filler particles that did not undergo dissolution in addition to solid fraction that formed isothermally. Thus, these “globules” are often much larger than the filler powder particle size.

Compositionally, this phase is mostly Ni with some Co, Cr and refractories (Mo, Nb, Ta) and γ' formers (Al, Ti). The solid that forms isothermally will be leaner in the refractory elements (since the liquid at the braze temperature is mostly reflective of the braze alloy Ni-Cr-B composition).

A distribution of small precipitates is often observed in this phase. These are not to be confused with the strengthening γ' precipitates which are of a much smaller size scale and were not considered in this investigation. The precipitates that form inside the γ -Ni “globules” correspond to borides that form due to boron saturation and will be discussed in the following next section.

2.6.2 Insert Precipitates

This microstructural feature is the focus of this investigation and includes all of the precipitates that form in the braze insert except for the Ni_3B precipitates that form in the non-isothermal solid.

Within the insert, two distinct precipitate types can be discerned. The first precipitate type forms a fine globular dispersion within the $\gamma\text{-Ni}$ solid (arrow marker in Figure 20) [Jahnke and Demny, 1983] and is the least detrimental to final properties. These precipitates form at the brazing temperature due to B saturation of the $\gamma\text{-Ni}$ [Lal et al, 2000]. The same study suggests the precipitates may form prior to the presence of a liquid phase due to solid-state diffusion between braze and filler alloy powder particles.

Figure 20. Precipitate formation inside filler powder particle [Lal et al, 2000]

Most investigators agree that for WGB of IN-738 with a Ni-Cr-B braze alloy these precipitates are borides with high Cr content, however, there is no consensus on the predominant phase stoichiometry. Su et al reported a predominant Cr(B,C) stoichiometry [Su, Chou, Chang, Liu, 1999]. Other authors [Jahnke and Demny, 1983] reported that the first precipitates to form are Cr_5B_3 , but as the matrix becomes Cr depleted, the CrB stoichiometry becomes more prevalent. In addition to Cr, these precipitates will also have refractory (Mo,W) content [Lal et al., 2000], [Tandon and German, 1994].

The second boride type forms between and adjacent to the γ -Ni globules and is the most detrimental to final properties. These borides are larger than the intragranular precipitates and form in a script-like phase. Compositionally, they are similar to the intragranular precipitates, precipitating initially as Cr_5B_3 and reverting to CrB as Cr becomes depleted. This can lead to a significant depletion of Cr in this region, greatly decreasing the corrosion resistance [Jahnke and Demny, 1983]. They are also W and Mo rich.

2.6.3 Non-isothermal solid

As its name implies, this microstructural feature is solid that did not form isothermally at the brazing temperature, but formed as the liquid between the γ -Ni “globules” cooled [Lal et al., 2000]. The microstructure of the non-isothermal solid is identical to that shown in the insert region in TLP brazing when isothermal solidification is not achieved (Figure 9).

The non-isothermal solid forms in the space between the γ -Ni “globules” and is often accompanied by porosity and intergranular precipitates.

The non-isothermal solid consists of a γ -Ni matrix with a fine Ni_3B precipitate distribution with the characteristic appearance shown in Figure 21. However, it is important to highlight that the eutectic reactions discussed in Section 2.2.2 predict the formation of Cr_5B_3 and CrB in association the γ -Ni/ Ni_3B and this has been observed by [Su, Chou, Chang, Liu, 1999]. Thus, when blocky borides are observed in the interface between the γ -Ni “globules” and the non-isothermal solid, it is difficult to determine if the boride formed isothermally or non-isothermally.

Figure 21. Eutectic (γ -Ni/ Ni_3B) structure [Lal et al, 2000]

The presence of non-isothermal solid is detrimental to the final properties and should be minimized. The interface between these γ -Ni “globules” and the non-isothermal solid is known to be a preferential crack propagation path [Su, Chou, Chang, Liu, 1999].

Additionally, the lower solidus temperature in this region makes it more prone to incipient melting when the finished component is reheated.

2.6.4 Interfacial Isothermal Solid

This phase is the same as the isothermally solidified γ -Ni phase that forms in the insert region, both with respect to composition and solidification mechanics [Chasteen and Metzger, 1979], [Tung and Lim, 1995]. The microstructural feature deserves separate mention because it represents the metallurgical bond between the insert and the base alloy. If this layer is not visible, it means no erosion of the base alloy occurred during brazing and will likely lead to a weak bond between the base and the insert material.

2.6.5 Base Alloy Precipitates

Precipitates will form in the base alloy through the same mechanism as in TLP bonding, namely, diffusion of MPD from the insert into the base alloy. The precipitates can form intragranularly close to the interface or intergranularly further away from the interface, since grain boundaries act as preferential diffusion paths. Similar to the insert precipitates, a small precipitate intragranular distribution is preferable to long, interconnected precipitate chains at the grain boundaries. Studies of TLP brazing of IN-718 showed both intergranular and intragranular precipitation up to 200 μm away from

the insert/base interface. At greater distances, precipitation is intergranular only [Wu et al., 2001].

2.6.6 Porosity

While unintentional, porosity is a common occurrence in WGB microstructures that merits discussion as a microstructural feature. When porosity is present, smaller distributed voids are preferred over large, continuous voids (cracks).

The formation of large voids is indicative of defects in the braze insert (e.g. inhomogeneous filler and braze powder distribution, low braze ratio, cracks or discontinuities, etc...), while smaller voids are associated with solidification shrinkage [Tung and Lim, 1995], [Lim et al., 1995], [Lugscheider, et al 1988]. Small voids have also been associated with the size distribution of the powders used. Small powder size improves homogeneity in the microstructure, but results in small interstice volumes. If the capillary paths are too narrow, braze alloy may not be able to penetrate, leading to the formation of small, distributed voids [Zhang, Zhang, Sun and Liu, 2000]. A powder mesh size of -200/+300 has been reported as ideal for WGB.

Solidification shrinkage can occur when isothermal solidification is not complete before cooling. The solid phase will form a continuous rigid structure trapping small volumes of liquid. If the density of the liquid phase is lower than that of the resulting solid phase(s), when the liquid cools voids will form in association to the non-isothermal solid.

Another important implication resulting from the porous nature of the braze insert is that the volume of the braze insert will decrease after brazing. This has major implications when trying to fill a volume of fixed size, since the insert will shrink. Reported values for the reduction in volume of the braze insert are from 10-20% [Chasteen and Metzger, 1979], [Tandon and German, 1994]. To compensate for shrinkage of the insert, it is necessary to place a braze reservoir adjacent to the insert to deliver make-up material. This will result in an increase in braze ratio and is compensated by using a braze insert of lean composition, relying on the infiltrated braze alloy to raise the braze ratio past the point of maximum allowable porosity.

2.7 WGB Technology - Opportunities for Improvement

Currently, WGB is a commonly used repair technology for superalloy components. Particularly with respect to gas turbine parts, WGB is used in the restoration of surfaces that are prone to erosion, heavy oxidation and foreign object damage [Stankowski, 2002]. The use of a very rigid insert has been suggested as a technique to manufacture complete components [Lorenz et al, 2004]. The technology's limitation is the mechanical properties (e.g. LCF resistance, stress-rupture strength) that result from the final microstructure, specifically, the heterogeneity. This investigation will consider improving the microstructure homogeneity through the insert structure and composition.

Chapter 3. Objectives and Scope

3.1 *Objectives*

This investigation is the first phase of a research program with the intent of developing a WGB repair process that will produce a joint with mechanical properties, specifically TMF resistance, equaling those of the base metal. This current research phase had the following goals:

1. Establish a procedure to produce a WGB joint suitable for future mechanical testing by
 - a. defining a standard geometry, and
 - b. determining the optimal delivery method for the filler and braze alloys
 - c. determine an optimum braze ratio range
2. Characterize the microstructure resulting from the WGB process

3.2 Scope

3.2.1 Alloying system

The alloying system used in this investigation is summarized in Table 9 and has been studied by other researchers [Jahnke and Demny, 1983], [Su, Chou, Chang, Liu, 1999].

The base alloy was a PH-Ni-base superalloy (IN-738LC). The filler alloy composition matched the base alloy while the braze alloy was a near-eutectic Ni-Cr-B alloy. Detailed compositions are given in Table 10. Some tests were carried out using different base alloys, however, the filler and braze alloys were always the same.

Table 9. Alloying System Selection Criteria

Alloy	Commercial Name / Description	Selection Criteria
Base	IN-738LC	Industrially relevant alloy which has been characterized extensively
	PH Ni-base superalloy	Difficult to weld composition make it a candidate for brazing repair High Cr content makes it critical to minimize and/or control precipitate formation Low C content to reduce carbide and carboboride phases
Filler	NI-284 Superalloy gas atomized powder	Composition matching the base alloy IN-738
Braze	NI-276 Ni-Cr-B gas atomized powder	Ni-based like the base alloy to ensure wetting Boron as the MPD with the highest diffusivity High Cr content to match the properties of the base and filler alloys Near eutectic composition for quick melting and good infiltration of the braze insert

3.2.2 Sample Configurations

Two groups of samples were made: drop-samples intended to characterize the effect of braze ratio alone on the final microstructure and joint samples intended to characterize the microstructure in an actual joint configuration. The joint configurations had two geometries – a variable-gap geometry in which the joint gap was not pre-determined and a fixed gap geometry in which the joint gap was controlled.

For the drop samples, the braze insert was a slurry that dried in place consisting of braze and filler alloy powders with a binding. Several slurry compositions were tested with varying braze ratios.

The inserts for the joint samples were manufactured through tape casting. Tape casting (also known as knife-coating or doctor blading) is a process used in ceramic component manufacturing and is thoroughly reviewed in [Mistler and Twina, 2000]. Tape strips of varying thicknesses and braze ratios were cast and later stacked to form individual inserts. By combining tapes having different braze ratios, a layered structure could be created which was thought would be beneficial for interface bonding.

3.2.3 Sample Evaluation

Sample evaluation was limited to metallographic analysis and no mechanical testing was performed. The metallographic evaluations included visual inspection, optical microscopy, SEM microscopy and EDS analysis.

Chapter 4. Experimental

4.1 *Materials*

IN738-LC was used as the base alloy, although some trials were carried out using IN-713 and 316 stainless steel. The base alloy was supplied as as-cast 20 mm (3/4”) rods. The filler alloy powder was NI-284 (composition similar to IN-738) while the braze alloy powder was NI-276 (BNi-9 composition), both manufactured by Praxair Surface Technologies and with a nominal sieve size of -325. The detailed compositions of the alloys used are given in Table 10.

Table 10. Material Elemental Analysis (Wt%)

	Ni	Co	Cr	W	Mo	Nb	Ta
IN-738LC	Bal.	8.68	16.06	2.76	1.70	0.84	1.92
NI-284	Bal.	8.62	15.9	2.49	1.75	0.85	1.68
NI-276	Bal.	0.01	14.74				
	Al	Ti	C	B	Zr	Hf	
IN-738LC	3.40	3.44	0.12	0.010	0.04	0.03	
NI-284	3.38	3.27	0.10	0.011	0.06		
NI-276			0.01	3.51			

Nicrobraz Cement 520 (Wall Colmonoy) was used as a binding agent to consolidate the powders in the braze insert. The binder is a liquid plastic, consisting of a polymer material in solution with a volatile solvent. The dry powders are mixed with the liquid binder and the solvent is allowed to evaporate, leaving a dry solid.

4.2 Sample Production

The steps necessary for the preparation of each sample type are shown sequentially in Table 11. Slurry preparation and tape casting will be discussed individually before moving on to the assembly process for each sample type. Finally, the brazing cycle will be reviewed.

Table 11. Sample production steps

Step	Sample Type		
	Drop	Variable-Gap	Fixed-Gap
Slurry preparation	Y	Y	Y
Tape casting		Y	Y
Insert assembly		Y	Y
Base alloy grinding			Y
Surface preparation	Y	Y	Y
Insert application	Y	Y	Y
Reservoir addition			Y
Furnace brazing	Y	Y	Y

4.2.1 Slurry Preparation

All samples started with the preparation of a slurry containing braze and filler alloy powders and a liquid binding agent. Slurry was prepared by weighing the braze and filler

alloy powders individually to achieve the desired braze ratio and placing both powders in a beaker. Binder was added gradually while the mixture was mixed manually using a stainless steel spatula. The slurry was mixed for approximately 5 minutes until the powders were blended homogeneously and the slurry had adequate viscosity.

The amount of binder added varied from batch to batch. Primarily, the amount of binder required varied with the braze ratio as shown in Figure 22. For slurries with low braze ratio, the required binder amount was approximately 0.6 mL/10g while high braze ratio slurries required as much as 1.1 mL/10g. It was critical to add binder gradually since viscosity would increase rapidly after a critical binder volume.

Figure 22. Proportion of binder to powder used in slurry preparation

Another factor that affected the amount of binder required was that after opening the main binder container, solvent would begin to evaporate, changing the binder's

properties. When working with binder that had been open for a significant length of time, more binder was required to achieve the desired slurry consistency.

Slurry was used immediately after the desired viscosity and homogeneity were attained, either poured directly into drop-samples or cast into tape form for the joint samples.

4.2.2 Drop Sample Assembly

Drop samples, consisted of a radial hole (0.25" DIA x .200") drilled into a 3/4" base metal rod as shown in Figure 23. Prior to final sample assembly, the base metal rod was degreased in an acetone ultrasonic bath. Immediately after degreasing, each hole was filled with slurry of fixed composition. These samples were used to determine the characteristic microstructure of known compositions. The slurry compositions used for the drop samples is given in Table 12.

Figure 23. Drop Sample Geometry

Table 12. Drop Sample Compositions

Sample	Filler (g)	Braze (g)	Total (g)	r_B (%)
VFR7-1A	8.9455	3.8477	12.7932	30
VFR7-1B	10.0298	2.5275	12.5573	20
VFR7-1D	8.9455	3.8477	12.7932	30
VFR7-1G	8.8113	5.8756	14.6869	40

4.2.3 Tape Casting and Insert Assembly

Slurry was cast into thin strips (tape) by moving an open-bottomed spreader slurry reservoir over a non-adherent surface (teflon), depositing a thin film which was allowed to dry (Figure 24). Slurry flow out of the reservoir occurred through a machined notch of

determinate size (blade gap) as seen in Figure 25. Several slurry reservoirs were fabricated from square aluminum pipe with 5 mm wall thickness and with blade gaps varying from 0.4 to 1.5 mm to produce tape with varying thicknesses.

Figure 24. Schematic of tape casting process

Figure 25. Photograph of slurry reservoir, dry tape and substrate

Tape casting was used to manufacture the individual layers that made up each braze insert as well as the braze reservoir for the samples that required it. Tape casting consisted of the following steps (illustrated in Figure 26):

1. The teflon substrate and slurry reservoir were washed with soapy water, rinsed with fresh water and finally rinsed with acetone.
2. The slurry was prepared as described in Section 4.2.1 and poured directly into the slurry reservoir without allowing it to settle or deaerate (Detail A).
3. The spreader box was slid manually along the Teflon substrate at a steady rate (~1" per second) (Detail B) until either the slurry inside the reservoir was exhausted (Detail C) or the spreader box reached the end of the substrate surface.
4. The slurry was left to dry under natural convection to form tape. Drying time depended on the tape thickness as well as the "freshness" of the organic binder, with the binder "freshness" having the most significant impact and drying times ranging from one hour to one day. The tape was considered dry when it would detach from the Teflon substrate when the substrate was bent. (Detail D)
5. After the tape was dry, it was scored and sectioned using a sharp knife into roughly 25mm x 20mm rectangles (Detail E).
6. The average thickness of each section was measured using a flat-faced micrometer
7. Tape was stored in plastic bags until insert assembly (Storage periods ranged from hours to months with no appreciable change in tape handling properties).

Figure 26. Schematic of the tape casting process

While multiple slurry reservoirs with varying blade gaps allowed production of tape of different thicknesses, it is important to point out that dry tape thickness is not equal to blade gap thickness as illustrated in the photograph of Figure 25. Reported values for the ratio of tape thickness to blade gap range from $1/3$ to $2/3$ [Mistler and Twiname, 2000]. In some cases, the final tape thickness is thicker than the blade gap due to welling of the slurry as it exits the reservoir.

This presented some problems in producing tape of the desired size, since the shrinkage was not easily predicted. Overall, it was found that thinner tape had less shrinkage than thicker tape as shown in Figure 27. In general, a 25% reduction in thickness was expected. The dry tape thicknesses produced ranged from 0.38 to 1.65mm. Another inconsistency encountered with tape production was that the tape thickness would be higher at the start of the tape strip when compared to the end. Because of this, it was necessary to produce tape in much higher quantities than strictly required for insert production.

Figure 27. Dry tape average thickness versus blade gap

Insert assembly was relatively straight-forward. Tape strips were selected, brushed with a small amount of binder and stacked to form the insert. The insert was allowed to dry 5 to 10 minutes before further handling.

The advantage of assembling inserts from multiple tape strips is that it allowed varying the insert composition. This was used to assemble inserts where the middle layer had a lower braze ratio than the outside layers, with the idea that the interface layers needed a higher braze ratio to ensure a good bond with the base alloy.

4.2.4 Variable-gap Sample Assembly

Variable-gap samples consisted of a braze insert stacked between two base alloy discs (Figure 28). The discs were cut from rod stock using a liquid cooled abrasive grinding saw. After sectioning, the disc surfaces were ground with 120 grit sandpaper and degreased with acetone in an ultrasonic bath for 15 minutes. Immediately after degreasing, the mating surfaces were brushed with a thin layer of binder and the insert placed between the two discs.

With this configuration, the base alloy pieces were free to move relative to each other. The purpose of this joint geometry was to establish a comparison point against the fixed-gap samples in order to study the shrinkage of the braze inserts. All of the samples were brazed with the insert in the horizontal position.

Figure 28. Variable-Gap Sample Geometry and Photograph

The variable-gap sample configuration and composition is summarized in Table 13. It was necessary to substitute IN-713 for IN-738 as the base alloy in some samples due to material availability. This did not have a noticeable effect on microstructure. Also worth mentioning is the insert configuration for samples VFR3-1, 2 and 3. Instead of assembling the insert from individually cast tape sections, the insert was manufactured by casting the second and third tape layers over the previous layer as shown in the photograph of Figure 29. Therefore, it was not possible to measure the thickness of each layer and the braze ratio was calculated based on the nominal gap size used during tape casting.

Table 13. Insert Composition and Layer Configurations (Variable-gap Samples)

Sample	Insert Layer Structure			Insert Total		Base Alloy
	<i>Layer</i>	<i>r_B</i> (%)	<i>Thickness</i> (mm)	<i>r_B</i> (%)	<i>Thickness</i> (mm)	
VFR3-1	1	100		43*	1.5*	IN-738
	2	0				
	3	100				
VFR3-2	1	33		43*	1.5*	IN-738
	2	50				
	3	33				
VFR3-3	1	75		43*	1.5*	IN-738
	2	25				
	3	75				
VFR6-5	1	75	0.26	42	1.46	IN-713
	2	25	0.95			
	3	75	0.25			
VFR6-6	1	75	0.35	44	1.98	IN-713
	2	25	1.21			
	3	75	0.42			
VFR6-7	1	43	1.53	43	1.53	IN-713
VFR6-8	1	43	1.56	43	1.56	IN-713

* Estimated

Figure 29. Typical insert layer detail for VFR3 samples

4.2.5 Fixed-gap Sample Assembly

The fixed-gap samples were designed to produce a joint configuration suitable for future mechanical testing. The geometry consisted of a slot cut into a rod of the base metal, as shown in Figure 30. The fixed volume is representative of actual repair applications where a fixed volume needs to be filled with the repair material.

Figure 30. Fixed-Gap Sample Geometry

Producing the notch in the base alloy was not trivial, since machining IN-738LC requires specialized equipment due to its high hardness. After determining that the cutting tools were suffering an excessive attrition rate, it was decided to manufacture the notch by grinding instead of machining.

The notch was produced by partially sectioning the base metal rod using a manually controlled water cooled abrasive grinding saw, with the notch depth controlled using a go/no-go gauge. The cutting disc used was a Buehler SamplMet2 Abrasive Cut-Off saw with a 4110 blade, which had a nominal thickness of 0.070" (1.8 mm).

Switching from machining to grinding the notch meant that it was not possible to attain the square notch geometry shown in (Figure 30). The notch had the typical geometry illustrated in Figure 31 due to varying shape and wear rates of the grinding disc. The geometry feature that should be highlighted is the tapering of the notch towards the root.

Figure 31. Fixed-Gap Sample Notch Profile Detail

The final procedure in the sample assembly was the placement of the brazing insert in the notch. The inside surfaces of the groove were lightly ground with 120 grit sand paper, followed by cleaning in an acetone ultrasonic bath for 15 minutes. The insert was then placed using the steps outlined below and shown graphically in Figure 32:

1. Layers of tape were stacked tightly so the insert would remain in place and with care not to apply excessive force that would crumble the insert. Due to the variations in tape and notch thicknesses it was necessary to match each notch and insert individually.
2. After selecting a suitable combination of tape sections the layers were stacked using a small amount of binder, creating the insert. The notch surfaces were then lightly brushed with binder and the insert was set in place. The assembly was allowed to dry, usually taking around 15-30 minutes (middle insert in Figure 33)

3. Excess insert material was removed using a sharp blade to make the insert flush with the rod surface (inserts on the left side of Figure 33).
4. For samples with a braze reservoir, the outer surface of the assembly was brushed with binder and a strip of thin tape was wrapped around the sample (inserts on the right side of Figure 33). The tape used in this step was prepared immediately prior to installation so it would be pliable enough to wrap around the rod.
5. Samples were allowed to dry for 15 – 30 minutes and then stored in plastic bags before processing in the vacuum furnace.

A photograph of the final sample is shown in Figure 33:

Figure 32. Steps in assembling fixed-gap samples

Figure 33. Multiple fixed-gap samples on single rod before brazing (VFR7-2 A-E from left to right)

The configuration for all the fixed-gap samples is summarized in Table 14. The experimental variables for these samples were the insert layer structure and braze ratio, effect of having a braze reservoir and joint orientation. For samples with a horizontal orientation (i.e. the notched rod was standing upright) the rods were of short length in order to avoid toppling. For the vertical samples, the ends of the bar were supported on IN-738 racks. Both sample orientations are shown on the right side of Figure 34, with the vertical samples in the centre and two smaller horizontal samples on the sides. Due to material availability, some trials used base alloys other than IN-738LC.

Table 14. Fixed-gap Sample Insert Composition

Sample	Insert Layer Structure			Insert Average		Braze Reservoir	Joint Orient.	Base Alloy
	Layer	r_B (%)	Thick. (mm)	r_B (%)	Thick. (mm)	r_B (%)		
VFR5-1	1	76	0.43	49	1.84	80	Horiz.	IN738LC
	2	25	0.98					
	3	76	0.43					
VFR5-2	1	76	0.36	47	1.93	No reserv.	Horiz.	IN738LC
	2	25	1.11					
	3	76	0.46					
VFR5-3	1	76	0.44	51	1.84	No reserv.	Vert.	IN738LC
	2	25	0.91					
	3	76	0.49					
VFR5-4	1	76	0.44	47	2.01	80	Horiz.	316 SS
	2	25	1.16					
	3	76	0.41					
VFR5-5	1	76	0.48	49	1.87	80	Vert.	316 SS
	2	25	0.98					
	3	76	0.41					
VFR5-6	1	76	0.54	55	1.88	No reserv.	Vert.	316 SS
	2	25	0.78					
	3	76	0.56					
VFR6-1A	1	55	0.42	26	1.89	No reserv.	Vert.	IN738LC
	2	0	0.99					
	3	55	0.48					
VFR6-1B	1	55	0.18	20	1.00	80	Vert.	IN738LC
	2	0	0.63					
	3	55	0.19					
VFR6-2A	1	55	0.29	17	1.87	No reserv.	Vert.	IN738LC
	2	0	1.3					
	3	55	0.28					
VFR6-2B	1	55	0.33	19	1.96	80	Vert.	IN738LC
	2	0	1.3					
	3	55	0.33					
VFR6-3A	1	75	0.31	30	1.65	No reserv.	Vert.	IN738LC
	2	0	1					
	3	75	0.34					
VFR6-3B	1	75	0.34	29	1.71	80	Vert.	IN738LC
	2	0	1.06					
	3	75	0.31					

Sample	Insert Layer Structure			Insert Average		Braze Reservoir	Joint Orient.	Base Alloy
	Layer	r_B (%)	Thick. (mm)	r_B (%)	Thick. (mm)	r_B (%)		
VFR6-4A	1	75	0.31	34	1.61	No reserv.	Vert.	IN738LC
	2	0	0.88					
	3	75	0.42					
VFR6-4B	1	75	0.33	30	1.79	80	Vert.	IN738LC
	2	0	1.08					
	3	75	0.38					
VFR7-2A	1	0	1.1	0	2.17	No reserv.	Vert.	IN738LC
	2	0	1.07					
VFR7-2B	1	30	0.39	11	2.23	No reserv.	Vert.	IN738LC
	2	0	1.4					
	3	30	0.44					
VFR7-2C	1	30	0.41	12	2.01	No reserv.	Vert.	IN738LC
	2	0	1.2					
	3	30	0.4					
VFR7-2D	1	30	0.37	10	2.11	80	Vert.	IN738LC
	2	0	1.39					
	3	30	0.35					
VFR7-2E	1	0	1.14	0	2.27	80	Vert.	IN738LC
	2	0	1.13					

4.2.6 Furnace Brazing Cycle

Samples were brazed in a vacuum furnace (Figure 34) with radiant electrical heating elements located at the National Research Council's Institute of Aerospace Research. To prevent damage of the furnace, the samples were placed on a ceramic base to contain any braze alloy drips.

Figure 34. NRC Vacuum Furnace (left) and vertical and horizontal orientation samples ready for brazing (right)

With the samples inside the furnace, the chamber was evacuated and filled with argon. The argon was then evacuated and the procedure repeated three times to ensure the absence of oxygen in the brazing atmosphere. After the final purge, the furnace was evacuated to 3×10^{-6} torr and the brazing cycle started.

The cycle used was based on those described by [Idowu, Richards and Chaturvedi, 2005], [Lugscheider et al, 1989], [Su et al, 1999], [Chasteen and Metzger, 1979], [Lim et al., 1995]. The steps in the cycle were as follows and plotted in Figure 35:

1. Ramp to the binder evaporation temperature ($10^{\circ}\text{C}/\text{min}$).
2. Hold at the binder evaporation temperature (600°C for 2 hours) - The manufacturer's reported evaporation temperature for the binder was 540°C .
3. Ramp to the sintering temperature ($10^{\circ}\text{C}/\text{min}$)

4. Hold at the sintering temperature (1000°C for 1 hour).
5. Ramp to the braze temperature (20°C/min): This is done at the furnace's maximum heating rate in order to minimize solid-state diffusion between the braze alloy and the filler alloy.
6. Braze hold (1200°C for 20 minutes)
7. Cool sample (10°C/min to below 500°C) - Sample was furnace cooled from the brazing to below 500°C in order to avoid sample aging during cooling. Below 500°C, the furnace heating elements were turned off and the sample cooled to room temperature.

Figure 35. Time-temperature plot for the brazing cycle

The temperature inside the vacuum chamber was monitored using the thermocouples that can be seen on the left-hand side of the photograph on the right in Figure 34.

4.3 Sample Evaluation

The finished samples were examined visually for external defects (incomplete fill, slumpage or surface voids) and then sectioned across the joint for metallurgical analysis using an abrasive grinding disc (Buehler 10-4127-010 alumina abrasive wheel 0.032" thick). The sectioned samples were prepared for optical microscopy, the main evaluation method since it permitted quick screening of a large number of samples for both integrity and microstructure. The preparations steps were as follows:

1. Section using abrasive grinder
2. Mount in black Bakelite
3. Grind using 120 grit bench-grinder
4. Manually grind in successive 240/320/400/600 grits
5. Clean samples by immersing in acetone in ultrasonic cleaner for 20 min
6. Polish samples using 6 μm diamond paste
7. Clean samples by immersing in acetone in ultrasonic cleaner for 20 min
8. Polish samples using 1 μm diamond paste
9. Clean samples by immersing in acetone in ultrasonic cleaner for 20 min

Samples were examined both in the as-polished and etched conditions. The etched samples were etched using Marble's reagent (10g CuSO_4 + 50mL HCl + 50mL H_2O), which attacked preferentially the matrix phase. Selected samples underwent further

evaluation using an SEM (JEOL 6400 digital Scanning Electron Microscope with a Link Systems eXL Energy Dispersive X-ray Analyser).

Samples were evaluated under two criteria: joint integrity and suitability for future mechanical testing and characterization of the microstructure.

Chapter 5. Results and Discussion

5.1 *Joint Integrity*

One of the objectives of this investigation was establishing a method for producing WGB samples suitable for mechanical testing, preferably with the standard tensile-testing geometry. The fixed-gap sample configuration was the only sample type evaluated for joint integrity.

For a sample to be considered successful, the notch volume had to be filled without large voids and with a minimum number of defects at the insert/base interface. Pass/fail criteria (Table 16) were established with the following considerations:

- Fill – there has to be sufficient insert material at the centre of the rod to allow the metal rod to be machined to the typical tensile test geometry.
- Insert defects – there had to be no defects directly related to the sample assembly process. As reviewed in Section 2.6.6, large voids (and cracks) are often the result of braze insert defects (either material discontinuity or locally high braze ratio). The definition of large voids was established in terms of the maximum capillary gap that can be infiltrated by BNi-9 in conventional brazing (100 μm).

- Interface defects – this criteria was the most subjective. Large voids were considered unacceptable for the same reason as the previous criteria. Small voids in a chain pattern were considered detrimental since they would represent an easy crack initiation/propagation location. A void chain pattern was defined as any void distribution with several voids in close proximity.
- Microstructure – microstructure was evaluated thoroughly at another level of analysis. As subsequent discussion will show, there was a distinct microstructure associated with regions of predominantly non-isothermal solidification. While some non-isothermal solidification is expected during WGB, regions of extensive non-isothermal solidification are clearly not representative of the WGB microstructure. For the sample to be suitable for mechanical testing, there could be no presence of extensive non-isothermal solidification.

Table 15. Joint Integrity Evaluation Criteria

Criteria	Evaluation Method	Pass Criteria
Fill	Area analysis after sectioning across the notch	> 90% area filled
Insert Defects	Void measurement using microindentation microscope	No voids > 100 μ m diameter No cracks longer than 100 μ m
Interface Defects	Void measurement using microindentation microscope	No voids > 100 μ m diameter No cracks along the interface No chains of small voids
Microstructure	Evaluation of fill area microstructure	No regions with predominantly non-isothermal phases

The as-brazed fixed-gap samples (Figure 36) were sectioned along the plane indicated in the figure and evaluated for joint integrity. The results are given in Table 16.

Figure 36. As-brazed fixed-gap samples before sectioning (VFR5-1 through 6 from left to right)

Table 16. Joint Integrity Evaluation

Sample	Fill (%)	Insert Defects (Number x max. diameter)	Interface defects	Microstructure (Location of predominantly non-isothermal phases)	PASS
VFR5-1	100	Void - 1 x 750µm	Small void chain - one side only	Throughout	N
VFR5-2	73	None	None	50% of notch - all on one side of the joint	N
VFR5-3	58	Voids - 3 x 400 µm	None	Notch root	N
VFR5-4	100	None	Small void chain - one side only	At the interface on both sides	N
VFR5-5	95	Voids - 4 x 500 µm	None	Throughout except notch root	N
VFR5-6	78	None	None	Throughout except notch root	N
VFR6-1A	73	Void - 1 x 1800 µm	None	None	N
VFR6-1B	93	Crack - 5mm along notch	None	Notch root and end only	N
VFR6-2A	62	None	Complete lack of bonding on one side Small void chain on opposite side	None	N
VFR6-2B	90	Crack - 6mm along notch	Small void chain - one side only	Throughout	N
VFR6-3A	78	None	Small void chain - one side only	Throughout	N
VFR6-3B	87	Void - 1 x 650 µm	None	Throughout	N
VFR6-4A	69	Crack - 5mm along notch	None	Throughout	N
VFR6-4B	98	Void - 1 x 1800 µm	Small void chain - one side only	Throughout	N
VFR7-2D	95	Void - 1 x 300 µm	None	Notch end only	N
VFR7-2E	100	Void - 1 x 250 µm	None	None	N
Pass (%)	44	31	63	19	0

The most notable result from this table is that no successful samples were produced according to the self-imposed specifications. However, there are trends in each of the evaluation criteria that should be evaluated.

5.1.1 Fill

The results confirm that complete fill of the joint volume can only be achieved when using a braze reservoir, as seen by comparing the top row of samples in Figure 37 (with reservoir) to the bottom row samples (no reservoir). The incomplete fill in absence of a reservoir is due to the inherent porosity of the braze insert (Section 2.5.1).

Figure 37. Fill of VFR6 Fixed-Gap Samples

Referring to the results summarized in Table 17, only one sample with a braze reservoir failed to achieve complete fill (VFR6-3B), and even for that sample, the failure to achieve complete fill was marginal (87% versus 90%).

Table 17. Fill Results

Sample	Fill (%)	Reservoir	Insert r_B (%)
VFR5-1	100	Y	49
VFR5-4	100	Y	47
VFR7-2E	100	Y	0
VFR6-4B	98	Y	30
VFR5-5	95	Y	49
VFR7-2D	95	Y	10
VFR6-1B	93	Y	20
VFR6-2B	90	Y	19
VFR6-3B	87	Y	29
VFR5-6	78	N	55
VFR5-2	73	N	47
VFR5-3	58	N	51
VFR6-3A	78	N	30
VFR6-1A	73	N	26
VFR6-4A	69	N	34
VFR6-2A	62	N	17

Another result that should be highlighted is that the sample with no braze alloy in the insert (VFR7-2E) achieved complete fill. This demonstrates that if the insert structure is sound, all of the braze alloy can be delivered to the notch from the braze reservoir.

It is also worth noting that in some samples with incomplete fill and no braze reservoir, there were distinct discontinuities in areas that had the desired microstructure and areas that solidified non-isothermally. This means that eliminating the braze reservoir to improve homogeneity is not a reasonable strategy.

Lastly, it should be noted that complete fill was achieved with samples brazed in either the vertical or the horizontal position (VFR5 samples), confirming that capillary flow in

this system is not strongly depending on the direction of gravity. This is verified by other authors [German et al., 2005] where for short bonding times (1-10 minutes) gravity has no effect on fill or final microstructure.

5.1.2 Insert Defects

While cracks were observed in some inserts, the most common insert defect was the presence of large voids. As mentioned previously, these large voids are likely the result of defects in the braze insert prior to brazing. Voids that form in the non-isothermally solidified region are likely the result of complete melting in the region damaging the insert capillary path structure.

One likely cause for these insert defects are inconsistencies in the braze ratio throughout the insert. Recalling that the slurry used for tape casting was mixed manually and without quantitative evaluation of viscosity or homogeneity, it is possible that the braze ratio was not consistent throughout each individual tape section. There is also the possibility of the insert braze ratio being too high overall, but this would have resulted in a predominantly non-isothermally solidified microstructure.

Another cause for void formation in the insert region is the loss of capillary paths due to damage to the insert structure during assembly. It was difficult to find tape section combinations that would provide adequate fill of the notch volume as outlined in the

sample assembly procedure (Section 4.2.5). The reason for this was two-fold, the notch geometry and the tape section thicknesses.

Recalling that the notch geometry had a taper, it was difficult to provide complete fill without a similarly tapered braze insert. This resulted in the defects shown in the top two photographs in Figure 38. On the left is an insert that is too narrow to provide complete fill at the notch end, while on the right is an insert that was too thick to completely fit into the root region. Applying binder to the notch surfaces reduced these defects, but excess binder would dissolve the insert back into slurry and eliminate the layered structure. Another defect was that when the insert was trimmed prior to application of the braze reservoir, material would sometimes accidentally be removed from inside the notch volume, as shown in the bottom photograph.

Figure 38. Insert Assembly Process Defects

The other problem encountered was the tape section thickness, which was hard to control, both in terms of average value for the tape strip as well as in the consistency throughout each tape strip. This should not have been surprising since out of the four factors identified as critical to providing consistent thickness (slurry viscosity, blade gap, casting speed and reservoir height [Stankowski, 2002] , [Mistler and Twiname, 2000]) only blade gap was controlled quantitatively.

5.1.3 Interfacial Defects

Attaining a defect free interface was the least exclusive selection criteria. As can be seen in Table 18, defect free interfaces were achievable with a wide range of insert interface layer braze ratios (from 76% to 0%). What should be highlighted is that the presence of interfacial defects can be associated to the presence of a non-isothermal structure for all but one of the samples. Additionally, the void chains formed on one side of the interface only. It is speculated that as the insert cooled, one side of the insert was pulled away from the base alloy creating void chains.

Table 18. Interfacial Defects Summary

Sample	Interface defects	Microstructure (<i>Location of predominantly non-isothermal region</i>)	Insert interface layer r_B (%)	Insert r_B (%)
VFR5-2	None	50% of notch - all on one side	76	47
VFR5-3	None	Notch root	76	51
VFR5-5	None	Throughout except notch root	76	49
VFR5-6	None	Throughout except notch root	76	55
VFR6-1A	None	None	55	26
VFR6-1B	None	Notch root and end only	55	20
VFR6-3B	None	Throughout	75	29
VFR6-4A	None	Throughout	75	34
VFR7-2D	None	Notch end only	30	10
VFR7-2E	None	None	0	0
VFR5-1	Small void chain - one side only	Throughout	76	49
VFR5-4	Small void chain - one side only	At the interface on both sides	76	47
VFR6-2B	Small void chain - one side only	Throughout	55	19
VFR6-3A	Small void chain - one side only	Throughout	75	30
VFR6-4B	Small void chain - one side only	Throughout	75	30
VFR6-2A	No bond on one side Small void chain on opposite side	None	55	17

The only sample that had interfacial defects without exhibiting regions of non-isothermal microstructure was VFR6-2A. It should be noted that the insert for this sample had a lean composition, no braze reservoir and exhibited a homogeneous structure throughout. Also, sample shrinkage occurred homogeneously on one side only, as shown in Figure 39. This indicates poor bonding due to low overall braze ratio, providing an indication of the lower limit for the braze ratio.

Figure 39. Fill pattern for VFR6-2A

5.1.4 Microstructure

The results summarized in Table 16 indicate that the presence of regions with predominantly non-isothermally solidified phases was the most common factor leading to unsuccessful samples. Notably, most of the samples that had this microstructure throughout, either due to a high braze ratio, the presence braze reservoir or both. The non-isothermal solidification in those samples was a result of composition and not directly related to the sample manufacturing process.

Table 19. Composition of samples with extensive non-isothermal regions

Sample	Microstructure (<i>Location of predominantly non-isothermal region</i>)	Reservoir	Insert r_B (%)
VFR5-6	Throughout except notch root	N	55
VFR5-5	Throughout except notch root	Y	49
VFR6-3A	Throughout	N	30
VFR6-4A	Throughout	N	34
VFR5-1	Throughout	Y	49
VFR6-2B	Throughout	Y	19
VFR6-3B	Throughout	Y	29
VFR6-4B	Throughout	Y	30

More important for producing successful samples is identifying the reasons for the formation of these non-isothermal regions in samples with low braze ratios. A feature that was observed throughout was that the non-isothermal structure formed preferentially at the notch root and the notch end. This is likely due to the insert defects described in Figure 38, where lack of insert material at either the notch root or end led to regions with more liquid at the braze temperature.

Although undesirable, the formation of the non-isothermal phases at the notch end and root can be tolerated to a certain extent when machining mechanical testing samples, since only the core of the notch material will be retained in the sample.

5.2 Microstructure Characterization

When discussing microstructure, it is important to reiterate that the goal was to produce the most homogeneous microstructure possible. The following sections will describe the

different microstructures observed and will consider two distinct regions: the insert region and the interfacial region.

The microstructures in the insert region will be discussed first, and since the starting point for this investigation was the metal powders forming the insert, their morphology is presented first.

5.2.1 Powder Characterization

The powders used (Figure 40) had the average particle sizes in the 10 to 40 μm range, as summarized in Table 20. Although braze and filler powders were of the same nominal sieve size (~ 325), the braze powder particles were found to be distinctly larger than those of the filler powder and of more uniform size.

Figure 40. Powder Morphologies (Optical)

Table 20. Average Powder Particle Size and Morphology

Powder	Average Particle Size* (μm)	Size Distribution	Particle Shape
Braze (NI-276)	38	Homogeneous	Spherical
Filler (NI-284)	10	Heterogeneous	Spherical
* Det. by optical analysis			

The braze powder particles had a regular spherical shape and a homogenous size distribution with an average particle size of 38 μm . The filler powder had a more heterogeneous size distribution ranging from 40 μm to less than 7 μm . The filler powder can be characterized as finer than the braze powder, with an average particle size no greater than 10 μm . This explains the correlation observed between braze ratio and required binder volume observed during slurry preparation (4.2.1)

5.2.2 Insert Region Microstructures

The joint configurations studied resulted in a wide variety of microstructures which typically contained the same microconstituents in varying morphologies and phase fractions. This section presents an overview of the observed microconstituent morphology and composition, detailed discussion will be presented in later sections. The microconstituents observed (marked in the optical micrograph of Figure 41 and the SEM EDS analysis of Figure 42) are identified as follows:

1. γ -Ni solid phase - This includes the unmelted fraction of the filler powder particles as well as isothermally solidified matrix phase. This phase corresponds

to the characteristic superalloy γ -Ni matrix. EDS analysis determined high Ni and Co content and moderate levels of Mo, W, Cr. Note that this phase appears as “globules” with a 30-60 μm size range, much larger than the filler powder particle size (10 μm).

2. Non-isothermally solidified phases – This feature occupies the space between the γ -Ni globules and consists of a fine, regular, precipitate dispersion in a matrix phase, shown in detail in Figure 43. The matrix phase is identified as γ -Ni and the precipitates as Ni_3B (per Section 2.6.3). It should be noted that the γ -Ni phase that forms the matrix of the non-isothermal solid is similar to the isothermally solidified γ -Ni with the difference that the non-isothermal γ -Ni has higher Ti and lower Cr content. For easy reference, this structure will be referred to as the “eutectic” structure. Because it formed between the γ -Ni globules, the characteristic size of this phase was highly variable.
3. Large borides – These precipitates formed in between γ -Ni globules in three distinct morphologies: dendritic, network and discrete, as illustrated by Figure 44. The size of these precipitates varied widely with morphology, from 10 μm in the distributed form to over 600 μm in the dendritic form. These precipitates had similar compositions in all samples, high in Cr, Mo and W and apparently devoid of Ni, Co and Ti.
4. Small borides – These small (1 μm range) precipitates formed a fine distribution within the γ -Ni globules. Their size made analysis difficult, but they appear to be Cr-rich and Ni-poor in a similar fashion to the large boride precipitates.

5. Unidentified precipitates – These precipitates were not discernible under optical microscopy and were only revealed after EDS mapping. They are approximately 1 μm in size and precipitate within the eutectic structure and at the interface between the eutectic structure and the γ -Ni solid phase. These precipitates had high Mo and W content, but unlike the borides described above, were high in Ti and low in Cr.
6. Porosity – The porosity considered here is different from the macroporosity described in Section 5.1.2 and considers those voids smaller than 100 μm that are almost unavoidable in the WGB microstructure.

Figure 41. Typical Microconstituents (VFR7-1D / Optical / Unetched)

Figure 42. EDS Mapping of Typical Microconstituents (VFR6-4A / 500x / Etched)

Figure 43. Eutectic Structure Detail (VFR6-4A / Optical / Etched)

Figure 44. Boride Morphologies (Borides appear as dark phases)

Figure 45. Typical porosity (VFR6-5 / Optical / Unetched)

The fact that the large borides form with different morphologies is important to this investigation. The different boride morphologies can be used as indicators of the final properties based on the considerations of Section 2.4.1. Based on this, the dendritic boride morphology was the most detrimental to overall properties, while the discrete morphology distribution was the most desirable. Each boride type was associated with a particular solidification sequence, as will be explained in the following sections. The advantage of using the boride phases as indicators is that a large number of samples can be screened using optical analysis alone.

Throughout the following discussion of insert microstructures, the phase markers will be kept consistent with the preceding micrographs.

5.2.2.1 Non-isothermal Solidification Microstructure

This type of microstructure was the one considered in the pass/fail criteria of section 5.1.4. Chapter 5. The characteristic microstructure is shown in Figure 46 and Figure 47 with the composition analysis shown in Figure 47. The distinguishing features of this microstructure are γ -Ni dendrites (1), eutectic phases with large volume fraction (2) and long borides (3). These borides were so large that they could be detected by the naked-eye after polishing the sample cross-section.

The presence of dendritic borides was associated with non-isothermal solidification. The evidence of this is the presence of γ -Ni in dendritic instead of globular form, as shown by the distinctive “maltese cross” pattern (Marker 1 in Figure 46 and Figure 47) associated with conventional, non-isothermal superalloy solidification. Note that the dendritic designation is not meant to imply that the borides are dendritic in nature, but that the particular boride type forms in association with γ -Ni dendrites.

The length and characteristic shape make it unlikely that these γ -Ni dendrites formed from an agglomeration of filler powder particles. Another indication that the

solidification process was non-isothermal is the lack of the fine precipitates (marker 4 in Figure 41) known to form from B saturation of the original solid phase (Section 2.6.2)

Preferential nucleation of the γ -Ni dendrites occurred at the insert/base interface (top of Figure 46), on the surface of the base alloy remained solid throughout the brazing cycle. The dendrites are rich in W and Mo, and have a Cr content higher than the eutectic but lower than the boride precipitate. The dendrites are Al and Ti poor.

The dendritic borides tended to precipitate within the core of the host dendrite. This is logical since the dendrite cores are known to be rich in Cr and refractory boride-forming elements [Ojo, Richards, Chaturvedi, 2004]

Figure 46. Dendritic Borides (Top – Optical , Bottom SEM)

Figure 47. Elemental mapping of a typical dendritic boride SEM (VFR6-3A Unetched)

The most significant impact of non-isothermal solidification is the sharply defined compositional gradients evident in Figure 47 which are detrimental to achieving homogenous microstructure.

Another detrimental characteristic of this microstructure type is the macroporosity that can form in association with it as described in 5.1.4. For non-isothermal solidification to occur, the local braze ratio has to be greater than the maximum allowed by the considerations in section 2.5.1.

Since this microstructure was observed in samples also with isothermal solidification structures, it is likely that the fabrication method for the inserts resulted in heterogeneous braze ratios within the same insert. It is important to highlight that the highest braze ratio composition (40%) tested did not result in this microstructure type.

5.2.2.2 Isothermal Solidification with Network Borides

The intermediate microstructure between non-isothermal solidification and isothermal solidification with a discrete boride distribution was the formation of an isothermally solidified structure where the borides tended to form networks of varying lengths between γ -Ni globules as shown in Figure 48 (EDS Maps in Figure 49). Besides the boride networks, this microstructure type can also be characterized as having a lower

presence of eutectic structures than the non-isothermal microstructure, but higher than the discrete boride distribution microstructure.

The network borides formed between γ -Ni globules and generally cluster a group of particles together. As can be seen in the Figure 48, a single network boride can span an extensive length of the overall joint. The networks had varying sizes in terms of the number of particles that are clustered and generally did not exhibit a preferred distribution direction. Another interesting feature is that these borides never formed adjacently to the eutectic structure and were always completely surrounded by γ -Ni globules.

It is postulated that these borides form in a manner analogous to the base alloy grain boundary precipitates (Section 2.6.5). After a dense structure of isothermal γ -Ni forms, the MPD diffusion from the remaining isolated liquid regions will occur preferentially along γ -Ni phase boundaries where individually formed γ -Ni globules meet. Although a detailed analysis was not conducted, evidence of this preferential MPD diffusion may be found by studying element depletion in the γ -Ni phase along the network path.

If these network borides form due to preferential diffusion paths in the isothermally solidified γ -Ni phase, then there is a possibility of modifying their morphology by changing the number/size of the γ -Ni globules, such as increasing the number of nucleation sites by using nano-sized powder or seeding with a refractory powder or

reducing the number of individual globules, and hence grain boundaries, by using a filler powder with a large size.

Figure 48. Optical micrograph of boride networks (VFR7-1D / Unetched)

Figure 49. SEM EDS Map of Boride Network (VFR7-3A / Unetched)

5.2.2.3 Isothermal Solidification with Discrete Borides

This microstructure is thought to be the least detrimental for overall properties. Shown in Figure 50, this microstructure has a small eutectic fraction, indicating almost complete isothermal solidification. No massive borides form and the boride distribution reduces available paths for crack propagation and decreases compositional segregation as seen in the EDS map of Figure 51.

Figure 50. Discrete boride microstructure (VFR3-3 Optical Unetched)

Figure 51. EDS Map of Typical Discrete Boride Microstructure (VFR7-2E Unetched)

5.2.2.4 Effect of Braze Ratio on Microstructure

The previous sections have described a variety of microstructures. The relationship between the microstructure types and the braze ratio will be discussed in this section.

One of the problems associated with the use of a braze reservoir, from an experimental perspective, is that it is difficult to accurately determine the actual braze ratio, since the mass of liquid infiltrating the insert is unknown. The drop samples were prepared specifically to determine the characteristic microstructure for defined braze ratios.

Three discrete braze ratios were studied, 20, 30 and 40%. It is evident that increasing braze ratio made the microstructure evolve to more complex large boride structures as shown in Figure 52. At a braze ratio of 20%, the borides form in the discrete distribution. At 30% braze ratio, the volume fraction of eutectic phases and boride size increases, with microstructure transitioning between discrete and network boride distributions. At the maximum braze ratio studied the borides transition from a network morphology to dendritic-type borides (although γ -Ni dendrites are not readily apparent).

Figure 52. Microstructure progression with increasing braze ratio

It is important to point out that even at the 20% braze ratio, the borides that precipitated tended to form network structures as seen in Figure 53. A sample with a higher estimated braze ratio (VFR3-1 at 43%) exhibited the distributed boride microstructure (Figure 54). Clearly, while braze ratio is important it is not the only factor determining the final microstructure.

Figure 53. Drop sample microstructure at 20% braze ratio (VFR7-1B / Optical / Unetched)

Figure 54. Insert microstructure at estimated 43% braze ratio (VFR3-1)

5.2.3 Interface Microstructure

Unlike the variety of microstructures found in the insert region, the base/insert interface consistently showed the same features in all the tests carried out, independent of insert structure or braze ratio. The notable features are shown in an optical micrograph in Figure 55 and in an EDS element map in Figure 56. They can be categorized in the following manner:

1. Insert
2. Precipitate free zone (PFZ)

3. Base alloy fine precipitates
4. Unaffected base alloy
5. Grain boundary precipitates

Figure 55. Typical Interface (VFR3-3 / Optical / Unetched)

Figure 56. EDS Mapping of Typical Interface (VFR6-4A / Etched)

The insert microstructure was described in the previous sections. The unaffected base alloy was the depth below the interface at which no new precipitates are observed. The interface is defined as the area between the insert and the unaffected base alloy.

Just away from the insert is the first feature of interest in the interface microstructure, the precipitate free zone (PFZ). The PFZ is believed to develop through the solidification of the γ -Ni phase by nucleation at the base alloy and growth into the insert region. This can be evidenced by examining a sample where the base alloy and filler powder composition did not match. An interface EDS Map is shown in Figure 57 for sample VFR6-6, which used IN-713 as the base alloy. Compared to IN-738LC (and the filler powder) IN-713 has lower Cr and Co contents and a higher Mo content. The EDS analysis clearly shows that the PFZ zone has a higher Cr and Co content than the base alloy, evidence that this region formed from the insert liquid.

Figure 57. EDS Mapping of Interface with a IN-713 Substrate (VFR6-6 / Unetched)

Below the PFZ is a region of the original base alloy grain structure but with a fine precipitate distribution. These precipitates form with the same mechanism as the small borides described in 5.2.2, namely, saturation of the γ -Ni matrix with B diffusing from the liquid.

The last interfacial feature is the grain boundary precipitates, which can be clearly seen in Figure 55, Figure 58 and Figure 57. Grain boundaries are preferential diffusion paths and it is clear from the figures that boron diffused preferentially along these paths and formed a large number of precipitates.

An unexpected result was the difference in composition of the borides between the IN-738 (VFR6-4A) and the IN-713 (VFR6-6) base alloy samples. While the expected boride precipitation behavior of high Cr content was observed in the IN-738 sample, the IN-713 GB precipitates are Cr-poor and Mo-rich. This indicates that borides will precipitate preferentially with Mo rather than Cr. This property could be used to promote a controlled boride precipitation through the insert by the use of Mo additions. Even if this does not reduce the boride phase fraction it will reduce the problem of Cr depletion.

While the interfacial features were found to be the same for all samples, the depth of these features varied as a result of the insert microstructure immediately adjacent to the interface. This can be illustrated by comparing Figure 56, an interface that corresponds to a discrete precipitate insert microstructure, with Figure 58, where the corresponding

insert microstructure was a transition between network and dendritic borides. Clearly, the depth of the affected base material is greater in the case of the sample with the transition microstructure (higher braze ratio).

Figure 58. EDS Mapping of Interface in a Newtwork/Dendritic Boride Joint (VFR6-4A / Etched)

Chapter 6. Conclusions

While the goal of achieving a method for producing samples suitable for mechanical testing was not achieved, with some additional improvements it is likely that successful samples can be achieved consistently. In terms of the critical process variables that were identified, the following conclusions can be drawn:

- Complete fill of the notch volume cannot be achieved without a braze reservoir
- The braze insert does not need to have braze alloy content to produce complete fill. The braze alloy can be provided from the braze reservoir exclusively.
- Joint orientation (vertical or horizontal) does not affect fill characteristics.
- The slurry and tape casting processes used do not have enough quantitative control to reliably produce defect free inserts
- A layered insert structure is not necessary to achieve a good interfacial bond
- High braze ratio at the base/insert interface is not a sufficient condition for good bonding
- Braze ratio must be higher than 17% to achieve good bonding with the substrate
- Non-isothermal solidifications structures were often found to be associated with other defects such as voids.

With respect to microstructure, it was shown that there is a distinct evolution in boride morphology that matches an increasing braze ratio. Multiple sample configurations could be screened quickly by evaluating their microstructure and identifying the boride distribution. While braze ratio is a determining factor in final microstructure, some inserts were able to achieve acceptable microstructures with braze ratios that were unsuccessful in other samples.

In terms of future work, the following items are recommended:

- Review and improvement of the insert manufacturing process. The repeatability of the slurry preparation should be ensured by a quantitative evaluation of slurry viscosity. Tape casting can also be improved using lab-scale tape casting equipment.
- Conduct microscopy (e.g. optical, SEM) characterization of the insert structure as a quality control process prior to brazing.
- Determine if the temperature control of the vacuum furnace is adequate for producing repeatable microstructures.
- Investigate the use of seeding nucleation sites for γ -Ni globules (e.g. refractory powders)
- Investigate the influence of braze and filler powder particle size and size distribution.
- Investigate the effect of standard heat treatments (e.g. aging of IN-738) on the final microstructure.

Chapter 7. References

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Appendix A – Sample Configurations

Master List of Sample Configurations

Sample	Type	Joint Orientation	N° of Insert Layers	Reservoir	Substrate
VFR3-1	Variable	Horiz.	1	N	IN-738
VFR3-2	Variable	Horiz.	1	N	IN-738
VFR3-3	Variable	Horiz.	1	N	IN-738
VFR5-1	Fixed	Horiz.	3	Y	IN-738LC
VFR5-2	Fixed	Horiz.	3	N	IN-738LC
VFR5-3	Fixed	Vert.	3	N	IN-738LC
VFR5-4	Fixed	Horiz.	3	Y	316 SS
VFR5-5	Fixed	Vert.	3	Y	316 SS
VFR5-6	Fixed	Vert.	3	N	316 SS
VFR6-1A	Fixed	Vert.	3	N	IN-738LC
VFR6-1B	Fixed	Vert.	3	Y	IN-738LC
VFR6-2A	Fixed	Vert.	3	N	IN-738LC
VFR6-2B	Fixed	Vert.	3	Y	IN-738LC
VFR6-3A	Fixed	Vert.	3	N	IN-738LC
VFR6-3B	Fixed	Vert.	3	Y	IN-738LC
VFR6-4A	Fixed	Vert.	3	N	IN-738LC
VFR6-4B	Fixed	Vert.	3	Y	IN-738LC
VFR6-5	Variable	Vert.	3	N	IN-713
VFR6-6	Variable	Vert.	3	N	IN-713
VFR6-7	Variable	Vert.	1	N	IN-713

Sample	Type	Joint Orientation	N° of Insert Layers	Reservoir	Substrate
VFR6-8	Variable	Vert.	1	N	IN-713
VFR7-1A	Drop	N/A	N/A	N/A	IN-738LC
VFR7-1B	Drop	N/A	N/A	N/A	IN-738LC
VFR7-1C	Drop	N/A	N/A	N/A	IN-738LC
VFR7-1D	Drop	N/A	N/A	N/A	IN-738LC
VFR7-1E	Drop	N/A	N/A	N/A	IN-738LC
VFR7-1F	Drop	N/A	N/A	N/A	IN-738LC
VFR7-1G	Drop	N/A	N/A	N/A	IN-738LC
VFR7-2A	Fixed	Vert.	2	N	IN-738LC
VFR7-2B	Fixed	Vert.	3	N	IN-738LC
VFR7-2C	Fixed	Vert.	3	N	IN-738LC
VFR7-2D	Fixed	Vert.	3	Y	IN-738LC
VFR7-2E	Fixed	Vert.	2	Y	IN-738LC