Gas Turbine Blade Hardfacing Process Development and Microstructural Study of the Interface

by

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Abstract

Extending the life of gas turbine blades by preventing wear damage that could lead to cracking or material loss poses many cost benefits. Hardfacing is a weld overlay method that can provide the necessary wear resistance in critical regions on a turbine blade. The development of an efficient and sound hardfacing process for specific material combinations of IN738 with wear resistant alloys Stellite 694 and Tribaloy T-800 using plasma transferred arc (PTA) welding was the objective of this research. Taguchi design of experiment methods were used to optimize processing parameters for PTA hardfacing, resulting in a set of predicted parameter level settings for each material combination. In addition to the process development, the interface microstructure was studied for varying degrees of mixing/dilution. Using analytical techniques such as differential scanning calorimetry, scanning electron microscopy, and energy dispersive spectroscopy, mixtures of different ratios of hardfacing alloy to substrate alloy were examined for any occurrence of deleterious phases. Mixtures resembling a high dilution rate (50 wt% of hardfacing material in the substrate) exhibited microstructures that indicated the presence of both topologically close-packed and adverse carbide phases.

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Chapter 1: Introduction

Surfacing methods have developed into a viable means of extending turbine blade life by providing a thermal barrier or wear resistance. While many advanced surfacing processes are quite expensive, the cost of replacing turbine blades is such that surfacing options are still attractive. This thesis investigates the development and optimization of a plasma transferred arc (PTA) hardfacing process to apply a wear resistant weld overlay coating on an Inconel 738 (IN738) substrate. The need for this process arises from the potential for wear damage on the tip shroud Z-section of gas turbine blades, an area of contact between adjacent blades. The arrow in Figure 1-1 points out the Z-section on an IN738 turbine blade.

Figure 1-1 Tip Shroud Z-Section

PTA hardfacing is an established surfacing method in the petroleum industry for wear prevention using powder consumables to produce smooth and consistent weld overlay deposits. Typical powder deposition applications involve large, mainly steel substrates whereas wire consumables are used with turbine blade components due to a claimed higher density of the deposit. The development and optimization of a PTA hardfacing process, using powder consumables, for IN738 turbine blades could result in a more cost efficient process, in addition to the life-extension benefits of a wear resistant coating. Major hurdles on the path to a defined process are optimizing the PTA processing parameters, for the specific material combinations involved, to produce a high quality deposit, and to determine the effect on the interfacial microstructure where two very different alloys are joined in a metallurgical bond.

This thesis begins by presenting a review of relevant literature and background information on the materials and manufacturing methods for gas turbine blades, potential hardfacing processes with an emphasis on PTA hardfacing, and some fundamentals of the Taguchi design of experiments method. These topics are covered in Chapters 2 through 4. Chapter 5 reviews the materials involved in the project and details the equipment and procedures followed to generate experimental results. PTA parameter optimization follows an innovative technique for design of experiments based on the Taguchi method. Based on concepts reviewed in Chapter 4, the Taguchi design of experiment is presented in Chapter 6 specific to this research. Here, the experimental set-up is defined along with the pertinent criteria for evaluating the results of experimental trials. Following on from Chapter 6, experimental results and discussion are presented in Chapter 7, including general preliminary observations, results and analysis from the parameter optimization

experimental trials, and the investigation into interface microstructures. This chapter is complemented by high resolution micrographs to illustrate the material effects and to complement other analytical results. The remaining chapters close out the research work with discussion, conclusions, and future work in Chapters 8, 9, and 10 respectively.

Chapter 2: Gas Turbine Blade Design and Manufacturing

Gas turbine engine operating efficiency is highly dependent on the material capabilities of components in the hot sections of the engine. Specialized materials and manufacturing methods are continually developed for parts such as turbine blades that experience extreme operating environments. These components/materials are designed to withstand high stresses and temperatures with minimal effect on the geometry and integrity of the blade, Figure 2-1. With all these capabilities, turbine blades become very expensive to manufacture and hence replace. This chapter examines the property and material requirements, manufacturing methods, and common materials for gas turbine blades.

Figure 2-1 Typical Gas Turbine Blade

2.1 Mechanical Property Requirements

The service environment experienced by gas turbine blades is demanding in many respects. High rotational speeds are the source of both centrifugal stresses and vibration. The hot gas flow introduces aerodynamic loads and high temperatures to the blades and can also be the origin of thermal fatigue, corrosion, erosion, and combined forms of degradation.

Turbine blades experience large temperature gradients, therefore dictating the need for thermal shock resistance. It is common practice for jet aircraft operating in northern climates to allow engines to idle in cold temperatures before shutdown to reduce the temperature gradients experienced by engine components. During operation, centrifugal loads are coupled with the high temperatures to cause susceptibility to creep. Resistance to creep must be designed into the blade microstructure. Other primary failure considerations include high cycle fatigue (HCF), present in the form of vibration, stress rupture, and corrosion. Low cycle fatigue (LCF), caused by thermal cycles and centrifugal loading from start-stop, is a secondary consideration. Figure 2-2 (Bannantine et al, 1990) shows how elastic and plastic strain components combine to give an overall life prediction based on crack initiation. Elastic strain is derived from stress conditions related to HCF while the plastic strain component is a function of the LCF. Further beneficial properties include a low coefficient of thermal expansion (CTE) and high thermal conductivity, necessary during high temperature operation to limit thermal strain. Manufacturability is also desired because of the high aerodynamic performance that depends on shape and tight tolerances.

Figure 2-2 Strain Life Curve

2.2 Material and Microstructure Requirements

Superalloy microstructure can be quite complex, but is strictly controlled to provide optimum mechanical properties depending on the service application. The evolution of superalloy microstructures is depicted in Figure 2-3 (Sims et al, 1987). The basic microstructure of nickel superalloys is a face-centred cubic (FCC) γ phase with γ' coherent precipitates (second phase particles), carbides, and occasionally other phases. Shape of the γ' phase precipitates has an effect on the creep properties; rod or plate-like morphologies, combined with γ saturation in molybdenum, demonstrate superior creep characteristics (Sims et al, 1987).

Figure 2-3 Superalloy Microstructure Development

Metal-carbide (MC) precipitates can contribute to creep resistance when at very low levels. However, these particles can decrease both ductility and fracture toughness. Degenerates of MC carbides can form and decrease material properties. Carbides of the forms $M_{23}C_6$ and M_6C should be avoided as they can form topologically close-packed (TCP) phases on grain boundaries. Alloying with hafnium forms fine HfC carbides instead of $M_{23}C_6$ and M_6C precipitates, increasing grain boundary sliding and oxidation resistance (Artemev, 1995).

Grain size and structure plays an important role in the strength and temperature capabilities of a turbine blade material. Grain structure is usually dependent on the manufacturing method used to fabricate the blade. Shown in Figure 2-4, conventionally cast or equiaxed blades have small to medium sized grains all of similar size and shape,

directionally solidified blades have large, columnar grains, and single crystal blades have no grain boundaries (one grain) (Sims et al, 1987). Section 2.3 examines manufacturing methods in more detail. Table 2-1 shows the relationships between grain size, strength, and creep resistance.

Figure 2-4 Cast Microstructures

Table 2-1 Grain Size and Associated Properties

Grain Size	Strength	Creep Resistance
Decreasing (↓)	Increases (↑)	Decreases (↓)
Increasing (↑)	Decreases (↓)	Increases (↑)
Single Crystal	Dependent on loading direction	Good (highest operating temperatures)
Fine (prevents dislocations)	Good (structural elements below 700°C)	Susceptible to grain boundary sliding

Grain boundaries can be a source of weakness in a material due to grain boundary embrittlement. There is a decrease in ductility, creep rupture time, and formability caused by concentrations of sulphur and phosphorus at the grain boundaries. The addition of boron and zirconium to the alloy solves this problem by replacing the unwanted elements at the grain boundaries, increasing creep resistance. However, in single crystal blades, tertiary creep is delayed because no voids can form (that are typically formed at grain boundaries). Therefore, there is no need for alloying elements to strengthen the grain boundaries and the melting temperature is increased, hence the operating temperature rises as well.

2.3 Manufacturing Methods

Trends in gas turbine blade manufacturing have moved from forged to cast products. Precision forgings are still an economical means of producing blades that do not require the high temperature capabilities of a first stage turbine nozzle or blade. A forged turbine blade begins as a cylinder of round stock, which is usually heated prior to the forging process. The specific type of forging process is dependent on alloy properties such as ductility, crystal structure, yield strength, and melting point and can include rapid hammering or slow pressing/rolling (Treager, 1996). Forgings produce a fine grain structure with good ductility and strength. More advanced alloys that have higher temperature capabilities are less conducive to forging because their formability and hot working window are reduced by the addition of refractory metals.

After forging, machining is required to crop excess material, introduce a means for cooling (holes and channels), and to finalize geometry under tight tolerances.

Conventional machining involves the use of traditional tools to remove material such as mills, lathes, drills, and grinders. For superalloys that have very high hardness and toughness properties, less traditional methods must be applied. For these materials electrical, chemical, and electro-chemical processes have been developed. These processes employ spark discharge, chemical reagents, and anodic dissolution to remove material from the workpiece in a very precise manner. Energy beam methods such as electron beam and laser beam machining can provide accurate material removal and drilling by vaporizing the base material.

2.3.1 Casting Processes

Evolution in casting technology has facilitated higher engine operating temperatures and improved performance. The complex geometry of some turbine blades required for internal air-cooling and high-performance airfoils is accomplished by investment casting (also known as the lost-wax process), Figure 2-5 (Johnson Matthey Ltd., 1999). A mould is created around a wax or plastic replica of the component, the wax is then melted out and the mould can be filled with the molten alloy. This process requires little or no machining once the casting is removed from the mould. Conventional casting using this method produces an equiaxed structure with reasonable temperature capabilities and high strength in all loading directions. For this type of microstructure, typical in-service defects include:

- Increased precipitation of grain boundary carbides
- Grain boundary void formation by creep
- Precipitation of TCP phases
- Interaction between casting and applied coating.

Figure 2-5 Investment Casting Process

Factors affecting this sort of blade deterioration are total cycles, engine internal conditions, and deviations in the manufacturing process (Persson and Persson, 1992).

Degradation typically progresses from trailing to leading edge at the mid-span of the blade. Creep is the key limiting factor as voids can form at a relatively early stage in the blade life.

Directional solidification (DS) was the next step in casting technology. DS castings produce components with long, columnar grains that run along the length of the casting direction. The base of the casting mould is chilled and drawn slowly down to promote the growth of the columnar shaped grains. Turbine blades manufactured using this technique only have grain boundaries parallel to the loading axis and therefore voids due to creep are less likely to form as these voids form on grain boundaries perpendicular to the loading axis.

Single crystal (SX) turbine blades are cast the same way as DS blades. In order to achieve a single grain in the blade portion of the casting (actual part to be entered into service) either a grain selector or a seed crystal can be placed at the base of the casting at the chill plate. The normal method for turbine blade production is to use a helical grain selector, or “pigtail”, designed into the mould. The helix only allows one grain to emerge at the top that will then propagate throughout the remainder of the blade as the casting solidifies.

The development of powder metallurgy has allowed new alloys to be created that could not necessarily be made using conventional techniques. Two methods exist for alloying powdered metals: mechanical, where powders of pure elements are mixed to provide the desired concentrations; and prealloyed, where the powder is created from an atomized liquid (Durand-Charre, 1997). Turbine blades can be manufactured using powdered metals in a process called hot isostatic pressing (HIPing). HIPing requires

temperatures up to 1200°C and pressures of 193 MPa (Treager, 1996) to consolidate the powdered metal into a component with uniform microstructure. The process can also be used to rejuvenate damaged blades by fixing cracks and filling voids.

2.4 Superalloys

Superalloys are high technology materials tailored to advanced applications where resisting extreme temperatures, corrosion, and stresses are among the design criteria. Three main categories exist: Iron-nickel-based, cobalt-based, and nickel-based. Generally, nickel-based superalloys are the material of choice in gas turbine blade applications and it is this class of materials that will be the major focus in this section.

2.4.1 Iron-Nickel-Based Alloys

The iron-nickel-based category consists of three subclasses that arrange the alloys according to their hardening/strengthening mechanisms. The first subclass contains precipitation of γ' and γ'' intermetallic compounds within an FCC matrix. This is the most important class of Fe-Ni-based superalloys, an example being IN718, which is strengthened by γ' and γ'' -Ni₃Nb. The CRM (corrosion-resistant material) series is the second subclass; deriving hardening from carbides, nitrides, and carbonitrides and some solid solution strengthening with refractory elements. The final subclass depends mainly on solid solution strengthening and are based on modification of stainless steels by increased additions of Cr and Ni. All Fe-Ni-based superalloys, with the exception of the CRM series, are generally used in the wrought condition (Donachie, 1984), however, an

increasing number of Fe-Ni-based alloys are being cast to reduce the amount of required machining.

2.4.2 Cobalt-Based Alloys

Unlike Fe-Ni- and Ni-based alloys, Co-based alloys are not generally precipitation hardened. These alloys rely heavily on solid solution strengthening from Cr, W, and Mo and carbide formation. Cobalt undergoes an allotropic transformation from HCP to FCC at higher temperatures. The FCC form is often stabilized by alloying elements, however occasional affinity for transformation may still exist. There are significant microstructural differences within the Co-based alloy group depending on whether the alloy is cast or wrought. In either case, Co-based alloys are typically considered to be less ductile than their Fe-Ni- and Ni-based counterparts. Due to the fact that in turbine blade applications Co-based alloys are mostly used as wear resisting coatings, this class of superalloys will be covered more extensively in Chapter 3.

2.4.3 Nickel-Based Alloys

Nickel-based superalloys have a maximum service temperature up to 1200°C (Rolls Royce, 1996) and new alloys are being developed to increase this temperature limit. Extreme temperature is not the only factor that must be considered when designing a turbine blade alloy. Chromium is a very common alloying element due to its ability to create a protective oxide layer on the surface of the component lending to excellent corrosion resistance. Other common alloying elements include titanium, aluminum, cobalt, and molybdenum, which are also added for strengthening purposes, to provide either solid solution or precipitation hardening. The following sections describe specific

elemental roles in Ni-based superalloys, carbide behaviour and morphologies, and common topologically close-packed (TCP) phases.

Elemental Roles

The alloying elements used in Ni-based superalloys have specific roles contributing either to strengthening or tolerance of high temperature operating environments. The elements are divided into classes and subclasses, including the matrix, γ' precipitate, and grain boundary classes; and the carbide and oxide scale subclasses. Belonging to the matrix class are Ni, Co, Cr, Mo, and W, providing the γ matrix with solid solution strengthening. The γ' class (Ni_3X) consists of Nb, Ta, Ti, and Al, with Ti and Al the most common precipitate formers with nickel. The grain boundary class exists for equiaxed and DS alloys to strengthen grain boundaries by displacing deleterious elements. Small additions of C, B, and Zr accomplish this task. The carbide subclass contains Cr, Mo, W, Nb, Ta, and Ti, all of which can combine with C to form desirable carbide phases, which will be discussed further in the next section. Finally, the oxide scale subclass, composed of Cr and Al, is important for high temperature and corrosion protection.

Carbides

Carbon levels in Ni-based superalloys are typically between 0.05 and 0.2 wt%. Carbon combines with the refractory and reactive metals found in the carbide subclass to form MC carbides, a beneficial carbide phase. These carbides can be susceptible to decomposition, during heat treatment or in service, into M_{23}C_6 and M_6C , less desirable phases that segregate to grain boundaries. It is possible to roughly identify the different

carbides based on their morphology and location. MC carbides form during solidification and are often evenly distributed at inter- and intra-granular and often interdendritic sites. They have an FCC structure that is not generally aligned with the matrix and tend to exhibit a coarse random cubic or script morphology. Degenerative carbides, such as $M_{23}C_6$, form at lower temperatures than MC carbides, hence the possibility of formation during processing and service. $M_{23}C_6$ carbides are abundant in alloys with moderate to high Cr content and have a complex cubic structure that is similar to the TCP σ phase. Preferential sites are along grain boundaries with irregular blocky particles or plate morphologies. σ plates have been known to nucleate from $M_{23}C_6$ particles (Sims et al, 1987). The properties and behaviour of σ and other TCP phases in Ni superalloys are given in the next section.

TCP Phases

Poor compositional control of high-temperature alloys can lead to a susceptibility of forming “basket-weave” type nets of close-packed layers of atoms during heat treatment or, more often, in service. Known as topologically close-packed, or TCP, phases, they align with the octahedral planes of the FCC matrix and appear as thin linear plates nucleating on grain boundary carbides. While appearing to grow directly from these carbides, or sometimes from γ' precipitates, TCP phases actually form from the Ni austenite matrix since most carbides and γ' precipitates develop first (Sims et al, 1987).

The most common TCP phases in Ni-based alloys are σ and μ , which are normally considered detrimental. As mentioned above, σ phase is similar in structure to $M_{23}C_6$ carbides and has a formula of $(Cr, Mo)_x(Ni, Co)_yC_6$ where x and y vary between 1-

7. Figure 2-6 shows two experimental alloys that have developed σ plates after thermal exposure at 950°C and the $M_{23}C_6$ carbides from which they nucleated (Chen et al, 2002). The σ plates exhibit a high hardness and are susceptible to low temperature brittle fracture. Their morphology and hardness are a cause of crack initiation and propagation. Other detrimental effects include lowering the high temperature rupture strength and a change in the rupture mechanism. Rupture strength is lowered due to a degradation of solid solution strengthening as the σ phase drains refractory metal content from the γ matrix. The rupture mechanism is changed from intergranular fracture to intersigmatic fracture, with propagation along the σ plates. With an increase of weight percent of Mo and W above 7%, the formation of σ shifts to μ . The μ TCP phase demonstrates similarities to the M_6C carbide phase.

Figure 2-6 σ -Phase Plates in Two Similar Experimental Alloys

2.5 Electron Hole Theory and PHACOMP

The stability of an austenitic matrix can be characterized by the electron vacancy theory embodied in the PHACOMP numerical calculation algorithm (Sims et al, 1987).

PHACOMP has been used very successfully in both the modification of chemical compositions for Ni- and Co-based alloys to avoid the formation of TCP phases, and in checking for the possibility of TCP phases after composition altering processing as in Calliari et al (2001) and Day et al (2003).

Work done by Pauling in the 1930's first identified electron vacancies, or holes, for important elements in the first period of transition metals. These elements are characterized by occupation of electrons in the 4s orbital while only partial filling of the 3d shell has taken place. Additionally, there is only a 3% change in atomic diameter between Cr and Ni (includes Cr, Mn, Fe, Co, Ni). Pauling theorized that within the d-orbital the different spins ($+\frac{1}{2}$ and $-\frac{1}{2}$) could be divided into fractions of bonding and non-bonding orbitals. Bonding orbitals are able to hybridize with p- and s-orbitals forming metallic bonds. Those d-orbitals not used in bonding then become available to participate in other events (Murphy et al, 1968). Pauling then assumed the number of required hybrid (spd) electrons for bonding in Cr, and calculated the amount "left over" based on the 6 available 3d and 4s orbital electrons. Since the atomic diameter remains almost constant for Mn, Fe, Co, and Ni, Pauling considered the requirement for spd bonding electrons to be unchanged from that for Cr. Therefore, the number of non-bonding electrons increases for each element as more electrons become available in the 3d and 4s orbitals. Finally, he compiled density of states diagrams for non-bonding 3d orbitals, revealing vacancies or "electron holes," Figure 2-7 (Murphy et al, 1968).

Figure 2-7 Electron Vacancies from Density of State Diagrams

The electron hole numbers, denoted by N_v , that Pauling found are assumed to be the same for other elements within the same periodic table group. There is some debate, however, on the accuracy of doing this as the effective valence seems to increase as the period increases. Other discrepancies appear when combining pure elements into alloys and it has been suggested that N_v may be compositionally dependent.

2.5.1 Applying Electron Hole Theory

Determining a mean electron hole number, $\tilde{N}_v$, for an alloy system can give an indication whether a TCP phase will form. Several factors must be known before a definitive answer can be given. Firstly, the alloy composition and subsequently the composition of the austenite matrix are required. This, in turn, requires a sound knowledge of precipitated phases, borides, carbides, γ' , etc, to determine the amount of available remaining austenite constituents. The mean electron hole number is then found by summing terms that are a combination of the elemental atomic fraction (m) and the electron hole number (N_v) for that element:

$$\tilde{N}_v = \sum m_i (N_v)_i \quad (2-1)$$

Finally, the susceptibility for TCP phase formation is determined by comparison of $\tilde{N}_v$ to an accepted safe limit. Typically, for Ni-based alloys, the composition is prone to form a TCP phase if $\tilde{N}_v \geq 2.45 \sim 2.50$. The safe limit for Co-based alloys is slightly higher.

Three assumptions must be made in order for the process to work properly:

- All expected phases are formed and the alloy is close to equilibrium;
- The TCP phase that might precipitate will form from the matrix only; and
- The calculated mean electron number is a linear function of the matrix composition.

The PHACOMP process incorporates the above application of the electron hole theory into a computer implemented algorithm.

Chapter 3: Hardfacing of Gas Turbine Blades

3.1 Purpose of Hardfacing: Wear Resistance

Wear exists when two solids under load experience relative motion at their counterface. Many types of motion can lead to wear, including unidirectional, reciprocating, sliding, or rolling, or any combination thereof. Typically, a higher wear resistance can be found in harder materials, with the drawback of lower fracture toughness. Desirable properties for high temperature wear resistance include high hot hardness, resistance to oxidation at temperature, and a high thermal diffusivity (Glaeser, 1980). For intense operating environments, Ni-based superalloys have inferior wear or galling resistant properties compared to Co-based alloys (Vikstrom, 1994). Galling is a condition whereby excessive friction between mating parts results in localized welding; subsequent spalling and a further roughening of the rubbing surfaces may follow. This condition is more severe on castings due to their coarser microstructures (Ocken, 1995). For these reasons, Co-based alloys are applied extensively as coatings to Ni-based superalloys for wear resistance (Drapier et al, 1975). Co-based alloys can be tailored for various wear applications by increasing the carbon content and adding more solid solution strengthening elements to better maintain the high temperature hardness (Bhansali, 1980).

3.1.1 Wear of Metals

Different types of wear can result from varying the type of motion and the materials. The main types of wear are adhesive, abrasive, fretting leading to fatigue wear, chemical (corrosive), and erosive wear (Sarkar, 1976). At the macro scale, properties

such as hardness and surface roughness and the presence of debris or contamination will affect the tribological properties of the system. On the micro scale, asperity interaction, surface and sub-surface crack initiation and propagation, and particle formation (debris) are all factors that affect the surface performance. An extensive review of contact mechanisms and tribology of coatings is found in Holmberg et al (1998).

Adhesive and abrasive wear are the dominating mechanisms in industrial metallic wear situations and can exist in conjunction with any of the other mechanisms mentioned in Section 3.1. Often, the instigating wear process will not be the same mechanism as the one that causes the majority of damage or even failure, making failure analysis quite difficult.

Adhesive Wear

Adhesive wear predominantly occurs as a result of bonding at the asperity tips and subsequent material transfer. There are many theories regarding mechanisms for material detachment, i.e. once a bond has been made between asperities, how does a fragment form? In many instances, the interfacial bond will be weaker than bonds within the material and so no wear or transfer will take place. However, due to weakness or defect within the top layer of material, occasionally the internal bond will be broken, causing a section of material to become attached to the mating surface, as in Figure 3-1.

Figure 3-1 Material Transfer in Adhesive Wear

Another possibility is that plastic shearing of successive layers will take place coupled with propagation of a shear crack, producing wedge-shaped transfer fragments. The fragments adhere to the mating surface, remain on the original surface, or bond to a previous fragment, eventually becoming agglomerated into a loose wear particle (Bhushan, 2002).

Systems that feature unlubricated metal-metal contact and where friction and wear are concerns, dissimilar metals are favoured for mating surfaces. This is because friction and wear are reduced with dissimilar metals compared to self-mated metal pairs. Choosing metals that are metallurgically incompatible results in less solid solubility between the materials, reducing adhesive properties. A further advantage can be found by using hexagonal close-packed (HCP) metals, such as Co, because they possess fewer slip planes than cubic materials. In addition, only one counterface requires the HCP structure to provide the same friction and wear performance as with two HCP mating surfaces.

Abrasive Wear

Abrasive wear encompasses wear by plastic deformation and through brittle fracture where significant cracking and release of particles are observed. Plastic

deformation can be caused by a significant difference in counterface hardness whereby the asperities of the harder metal causes plastic flow of the softer surface, in the form of ploughing, wedge formation, or cutting. Also possible is 3-body abrasion, which has the same effects but is caused by hard particles within the interface introduced by an outside source or through a prior wear mechanism that has generated oxidized wear debris. Figure 3-2 illustrates the mechanisms of abrasive cutting and 3-body abrasion. Abrasive wear by plastic deformation is easily identified using SEM analysis as clear scratch marks aligned with the direction of motion.

Figure 3-2 Plastic Deformation in Abrasive Wear

With very brittle materials, asperity contact above a critical load will result in brittle fracture as lateral cracks develop and grow up to the free surface. Clusters of growing cracks act to isolate small “plates” of material that are removed from the bulk when the cracks have reached the surface. This type of abrasive wear is shown in Figure 3-3.

Figure 3-3 Brittle Fracture in Abrasive Wear

3.1.2 High Temperature Wear

At high temperatures contacting metals oxidize, influencing wear properties and causing significant changes in the wear rate. Oxides act to reduce metal-metal contact within a system, resulting in a transition from severe to mild wear. The oxide wear debris is mostly contained within the contact layer, forming compact, load-bearing layers that act to reduce the coefficient of friction. A more rapid reduction is realized at higher temperatures. At these higher temperatures, a glaze can form from sintered wear debris, further reducing friction and allowing very low wear rates. Subsequent debris can be re-compacted into the glaze, maintaining this protective layer (Stott, 2002), see Figure 3-4, adapted from Stott (1998).

Figure 3-4 Development of Protective Oxide Layers from Wear Debris

It should be noted that glaze formation is dependent on the counterface material. For example: Nimonic 80A (Ni-based superalloy) exhibited glaze formation and good wear resistance with an Incoloy 800 counterface (Fe-Ni-based superalloy); but Nimonic 80A with a Stellite 6 (Co-based alloy) counterface showed very poor wear resistance because of the absence of glazes (Wood et al, 1997).

3.2 Cobalt-Based Alloys

Co-based alloys were originally designed for castings in wear resistant applications and have subsequently been used as hardfacing alloys applied to components

requiring added wear resistance. From a microstructural point of view, there are three types of Co-based hardfacing alloys.

The first type is a Co solid solution, which typically contains <0.4% carbon. The solid solution type Co alloys are scarcely used as wear resistant coatings; rather, they are used for good corrosion and thermal fatigue resistance.

The second type of Co-based hardfacing alloys contains significant amounts of carbides with Cr, W, and C intentionally added into the matrix for increasing and retaining high temperature hardness. The microstructure for a composition of Co-1.2C-29Cr-4.5W exhibits a hypoeutectic dendritic structure of $M_{23}C_6$ carbides ($M = \text{Cr, W, Co}$) and cobalt solid solution (Wu and Redmond, 1994). Other phases, such as Cr_7C_3 and Co_3W , have also been found in Stellite 6, for example, in addition to the primary $M_{23}C_6$ carbides (Wang and Li, 2003). The addition of Si in Stellite 6 has resulted in formation of μ and σ phases (Kuzucu et al, 1998), a hybrid form of type 2 and type 3 Co-based wear resistant alloys. Stellite 694, examined in this study, is one of the carbide containing Co alloys with a greater W content than Stellite 6. The refractory elements such W and Mo, while known for their effective solid solution hardening effect, contribute to the formation of MC, $M_6\text{C}$, and intermetallic phases such as $\text{Co}_3(\text{Mo, W})$ (Przybylowicz and Kusinski, 2000).

A third type of Co-based hardfacing alloy uses intermetallic phases, such as Laves phase $(\text{Co, Mo})_2\text{Si}$, to impart wear resistance. The most common alloys in this category are those in the Tribaloy series; the T-800 alloy used in this study falls within this group. Coatings containing Laves phase are unique in terms of its high temperature wear resistance, as the Laves phase retains its stability up to 788°C. Hence, these materials

have found wide application in coating components for high temperature wear resistance. However, the alloy is quite brittle due to the presence of the Laves phase and the weldability is quite poor.

3.2.1 TCP Phases in Cobalt-Based Alloys

While some TCP phases are desirable in Co-based alloys, such as the Laves phase discussed above, others such as σ , μ , and π can cause a loss of strength and ductility in the alloy. Close attention must be paid to alloy compositions in order to avoid these TCP phases; formation will occur if the solubility limit of the austenite matrix is exceeded by additions of refractory elements and Cr. In fact, σ phase will form if the combined Cr and W content is over 37 wt%. As in Ni-based alloys, σ plates are coherent with $M_{23}C_6$ carbides and appear as needles when examined. Segregation of elements such as Cr and Mo to the grain boundaries cause TCP initiation in these areas, often nucleating on carbides also present at the grain boundaries. The σ plates grow from the matrix and not the more stable γ' phase, which is often displaced as the σ plates grow. Differentiation between the plate-like $M_{23}C_6$ and σ phases is important, but difficult at low magnifications. At higher magnifications, $M_{23}C_6$ will show a composition of short, aligned rods or particles and may etch differently than the σ plates (Sims et al, 1987).

3.3 Coating Application Methods and Equipment

Surfacing techniques are used to apply protective coatings to new turbine blades and to repair damaged blades by filling voids and rejuvenating geometry. Thermal spray coating is a process that produces a protective surface with no metallurgical link to the

base material, mechanical interlocking is the primary form of adhesion. Hardfacing (fusion surfacing, weld surfacing), on the other hand, relies on a metallurgical bond between the coating and the substrate. While this method may influence the microstructure of base materials, it can provide better wear resistance, if the dilution can be controlled.

Surface preparation is very important when applying coatings since the surface condition will dictate adhesion of the coating and any contamination can cause diminished mechanical properties in service. Typical steps include: Degrease, grit-blast with steel grit (or equivalent), and a thorough degrease (hot alkaline bath, trichloroethylene, methyl ethyl ketone, acetone). The coating is also affected by the base material in the following manners (Gross, 1968):

- Composition – diffusion can occur between the base material and the coating, the surfacing material is not necessarily the same as the substrate
- Mode of surface preparation – there are varying techniques and processes
- Degree of surface roughening
- Temperature the substrate reaches during the process.

3.3.1 Arc Welding Processes

Two arc welding processes are used in this study to apply hardfacing materials; gas tungsten arc welding (GTAW, also known as tungsten inert gas welding or TIG) and plasma transfer arc (PTA) welding. A brief description of TIG welding is provided below, followed by a more in-depth section on PTA welding since it is the focus of this research project. A comparison of the two processes will identify the key differences and some possible advantages of one process over the other.

Tungsten inert gas welding was originally named “TIG” because of the use of argon as a shielding gas and tungsten as the electrode. The term “GTAW” is becoming more prevalent stemming from the use of gas mixtures other than argon and helium. TIG welding brought about a change in arc welding practices as the electrode is not consumed by the process and no flux is required. The tungsten electrode can act as either the cathode or anode, and can be used with AC or DC depending on the metal to be welded. An arc is struck between the tungsten electrode and the work by scratching or touching and pulling away of the electrode. Heat from the tungsten arc ionizes the protective gas in the gap producing intense heat and light in a conical shaped arc. The weld pool is protected from oxidation by the shielding gas, typically argon, but helium and various gas mixtures can be used for specialized applications. Deposition of hardfacing coatings using a TIG welder requires the coating alloy to be in wire form, it is then manually fed into the arc/weld pool to form a bead on the work surface. Figure 3-5 illustrates the torch configuration and equipment set-up (US Army, 1993).

Figure 3-5 TIG Torch and Set-Up

Plasma transfer arc welding is very similar to a TIG set-up with one major exception: the arc is transferred to the workpiece via plasma instead of just between the tungsten electrode and the substrate. Plasma is a gas that is ionized through high temperatures becoming an excellent conductor. The plasma can then efficiently transfer an arc to the workpiece. A tungsten cathode is still used, however, it is located within a nozzle and is ignited by a pilot arc established between the electrode and the nozzle. PTA welding requires an additional gas to the shielding gas called the plasma gas. This gas, usually argon, is forced through the constricting nozzle creating a column of plasma as it

passes the tungsten electrode. The plasma column delivers the arc to the workpiece with a relatively small arc diameter, providing intense heat in only a small area (Deloro Stellite, 1992). The arc shape is cylindrical and is less dependent on the working distance. Adjusting the voltage and current, as well as plasma gas flow rate changes the weld characteristics. Internal cooling of the nozzle and surrounding components is required and accomplished by circulating water through the nozzle. A plasma torch can be much more complex than a TIG torch, and for that reason it is typically mounted on an automatically controlled gantry. A common addition is the capability to deposit powdered metal provided by a hopper mounted above the torch and introduced into the plasma stream with another inert gas. A typical PTA torch (Joining Technologies, 2004) and equipment set-up (US Army, 1993) are shown in Figure 3-6, note that the powder gas stream is not pictured.

Figure 3-6 PTA Torch and Set-Up

Depending on the specific plasma arc welding process, two to three gas streams are required. The plasma (or orifice) gas and shielding gas are always necessary for operation, and a transfer gas for powdered metal may also be used, as mentioned above.

In many cases, the same gas is used for all applications. Argon is the most common plasma gas providing an easy arc start. For higher heat input, helium is added to the argon. Shielding gas is selected based on the substrate material:

- Welds in Al, carbon steel, Cu – argon, helium, or argon-helium mixtures
- Welds in low alloy steels, stainless steels, Ni alloys – same as above plus argon-hydrogen mixtures may be used.
- Welds in Ti – argon, helium, or mixtures

Reactive metals, such as titanium, must not be welded using nitrogen or hydrogen mixtures as the plasma or shielding gas as this may result in porosity (Praxair, 2002).

3.3.2 PTA Hardfacing

The deposited material starts in powdered form allowing deposit alloys to be simply premixed in powder form by combining the weight percentages of the pure substances. This also means that PTA can apply materials that cannot be easily formed into wires or rods; or that are not metallic at all, cermets (WC, TiC, etc.) can be used to create super-hard layers. Some powder deposit delivery systems involve varying an induced vibration in the powder tank to change the powder feed rate. A delivery gas then supplies the powder to the torch. Care must be taken when working with alloying elements of differing densities. Nickel and titanium, for example, have densities of 8.9 g/cc and 4.5 g/cc respectively. By varying the vibration, the amount of each element being delivered can also vary (Owa et al, 2002).

Cracking can occur when deposit and base materials differ in thermal expansion coefficients and thermal gradient, causing residual stresses during cooling. Differences in

thermal conductivity also contribute to cracking by magnifying the difference in thermal shrinkage (Owa et al, 2002).

3.3.3 Advantages of PTA over TIG

Plasma processes were developed after TIG welding had been established as an advanced welding process for materials not able to be welded using classical techniques. Therefore, in many senses, PTA welding could be considered an improvement to the TIG process and offers the following advantages (Henthorne and Chadwick, 1982):

- Electrode cannot contact the weld pool
 - Low risk of tungsten inclusions
 - Electrode does not become contaminated
- Increased weld speeds, less distortion, smaller HAZ
 - No post-weld heat treatments required
- Arc is stable at very small currents
 - Allows thin sections to be welded
- Improved visibility and access because the arc length is less critical owing to the cylindrical shape of the arc, as opposed to conical for TIG
- Deposit material is in powder form
 - Costs reduced, more versatile weld bead geometry

3.3.4 Laser Powder Fusion

Laser powder fusion (LPF) is often used in welding and cladding applications for aero-engine components. Fine metallic powder is injected into a CO₂ gas or solid-state YAG laser beam; the particles undergo rapid melting and are deposited onto the

substrate, Figure 3-7 (Krause, 2001). In a single pass, the thickness of the deposit can be adjusted between 0.005 in and 0.030 in (0.13 mm and 0.76 mm) with beads as narrow as 0.020 in (0.5 mm). LPF accomplishes weld build-up with little heat transfer; the heat input is an order of magnitude lower than conventional techniques such as TIG. Therefore, there are less residual stresses and a small HAZ. Table 3-1 provides a comparison of the hardfacing methods discussed in this section (Kaser, 2002).

Figure 3-7 Laser Powder Build-Up Welding

High precision is attainable with the laser process with CNC capabilities allowing depositions of near net shape. Further refinement of the welding parameters based on individual part geometry and alloy composition can provide DS and even SX

microstructures within the deposit. While these processes are still in an early stage, research on the required parameters is ongoing (Krause, 2001).

Table 3-1 Technology Comparison

Technology	Pass Height	Pass Width	Productivity
Manual TIG	0.100" – 0.200" (2.54 mm – 3.05 mm)	0.300" (7.62 mm)	4-5 blades per hour
PTAW	0.040" (1.016 mm)	0.040" (1.016 mm)	6-10 blades per hour
LPF	0.020" (0.51 mm)	0.060" – 0.075" (1.52 mm – 1.91mm)	25-30 blades per hour

3.3.5 Thermal Spray Coatings

Thermal spray processes are included here as they are a viable means of imparting wear resistant coatings to gas turbine components. However, a distinction must be made between thermal spray coating (TSC) and the previously discussed processes: TSC is not a welding method and is therefore not technically a hardfacing process.

A thermal spray process is composed of two key steps; melting of a wire or powder material and transport (spraying) of the molten material to the workpiece. Solidification of the molten droplets is instantaneous upon contact with the part resulting in predominantly mechanical bonding, although metallurgical interaction may also be possible, particularly during post-spraying thermal processes. Thermal spraying offers a wide range of possible deposit materials, high deposition rates, and equipment mobility. Figure 3-8 (Sulzer Metco, 2004) shows a typical thermal spray set-up that can be applicable to any of the following processes:

- Combustion wire spraying
- Combustion powder spraying

- Electric arc wire spraying
- Plasma arc spraying (powder)
- High velocity oxy-fuel (HVOF) spraying
- Controlled atmosphere chamber spraying (with any of the above)

Figure 3-8 Thermal Spray Process

3.4 Interface and Deposit Microstructures

The microstructure of a PTA deposit varies from the fusion zone to the upper-section near the surface. Near the deposit base, or interface, dendrites form perpendicularly to the interface; the centre-section of the deposit is dendritic with equiaxed crystals; and the upper-section has a mixed structure with finer equiaxed crystals. These features indicate a very rapid cooling rate.

Chapter 4: DOE and the Taguchi Method

DOE, or design of experiments, was introduced as a statistical method used to investigate the effects of multiple factors simultaneously. Originally developed for the agriculture industry in the late 1920s, DOE based on the full factorial method found limited application in an industrial setting due to academic developments that made advanced DOE methods impractical for production-based engineers. On the other hand, the Taguchi method, or Taguchi approach, is a modification of the standard DOE process based on improved techniques for organizing experiments and analyzing the results. It incorporates a new way of thinking for quality improvement during the design process and dictates the mode of application for established DOE techniques. This chapter will outline the fundamentals of experimentation for process improvement/optimization, provide a breakdown of the Taguchi method with a comparison to traditional approaches, and will present the necessary techniques for analyzing results.

4.1 Experimentation

Two types of experiments exist: investigative and demonstrative (Roy, 2001). Investigative experiments are those planned using a DOE method and are intended to give a result pertaining to the effects of factors or designs on performance. A demonstrative experiment is one carried out to validate the results of a development process before production/implementation proceeds.

Challenges faced by test engineers when designing investigative experiments are twofold. The first hurdle is defining the evaluation criteria and describing how performance will be assessed. Often, a statistical method is required to properly measure

performance since a direct comparison is not feasible when groups of products/processes are being compared to one another. The second challenge is deciding the form of the experiment, whether physical or virtual. In the situation where a valid simulation tool exists for a process, the same methods of experimentation and analysis can be applied as for real hardware but with significant advantages in cost. In either case, costs can still be excessive, necessitating a course of action that yields a maximum amount of data from a minimum amount of experiments.

4.1.1 Terminology

A *factor* is any input that is controllable in an experiment and has an influence on the subsequent performance of the product or process. Factor is a general term used to describe the inputs, causes, variables, parameters, ingredients, or constituents of an experiment, usually denoted by letters for simplification. Proper project definition will avoid confusion when dealing with factors that could be considered inputs for some experiments but outputs for another. For example, hardness is an output for a heat treating experiment but an input for a cutting tool experiment.

Factors fall into one of two categories based on the degree of control possessed by the experimenter. A continuous factor can always be quantified and is fully adjustable through a continuous set of values. Continuous factors include temperature, time, pressure, and part thickness in a curing process experiment. A discrete factor is associated with “types” or pre-existing settings. The same curing process experiment could have oven type, material system, and tool material as discrete factors. It should also be noted that temperature for this experiment could be a discrete factor if the oven only has *low*, *medium*, and *high* settings.

Levels are contained within factors and refer to the settings under study and are represented by numbers (1, 2, 3...). Usually, all the factors in an experiment will have the same number of levels, most commonly two or three, however, some set-ups allow a combination of levels to be examined. Determining the number of levels to include in an experiment depends on the time and money available and the desired detail of the results. A greater number of levels will require more tests to be run, but will also allow analysis of non-linear behaviour.

A measurement of performance is termed a *result*. To be useful for experimental analysis, results must be quantified, even if they are inherently qualitative. Measured values can be directly manipulated but pass/fail or good/bad type results must be converted to numerical values. A 0 to 10 scale is useful in these circumstances when qualitative performance must be defined. Where multiple objectives exist, the result remains a single value, but is composed of a combination of several evaluations. This result is termed the *overall evaluation criterion* (OEC).

With a numerical result, an indication of direction is required to give meaning to the number. The *quality characteristic* (QC) is used to define how we interpret the number and enables comparison of performance, i.e. how to pick the best result from a set of values. Knowledge of the project and requirements of the product/process are essential when assigning the QC as there are three possibilities: *Bigger is better*, *smaller is better*, and *nominal is best*.

4.2 Taguchi Methodology and Process

At the heart of the Taguchi approach is a new philosophy towards product/process quality and optimization. By dealing with quality issues at every stage of development, and not just inspecting parts at the end of production, considerable savings can be realized in terms of less scrap and fewer customer returns. For an existing process, optimization to achieve a higher consistency of performance, or quality, means that variation from a target value must be minimized and the standard deviation of the population be reduced. A fundamental philosophy in the Taguchi approach is that a reduction in variation will improve quality. The Taguchi method “provides a systematic approach to the study of the effects of various production parameters in a limited number of experimental trials and hence limited budget and resources” (Antony et al, 2001). Antony and Antony (2001) provides a series of nine steps to follow for any Taguchi experiment:

- Step 1: Formulation of the problem – the success of any experiment is dependent on a full understanding of the nature of the problem.
- Step 2: Identification of the output performance characteristics most relevant to the problem.
- Step 3: Identification of control factors, noise factors, and signal factors (if any). Control factors are those which can be controlled under normal production conditions. Noise factors are those which are either too difficult or too expensive to control under normal production conditions.

Signal factors are those which affect the mean performance of the process.

- Step 4: Selection of factor levels, possible interactions, and the degrees of freedom associated with each factor and the interaction effects.
- Step 5: Design of an appropriate orthogonal array (OA)
- Step 6: Preparation of the experiment.
- Step 7: Running of the experiment with appropriate data collection.
- Step 8: Statistical analysis and interpretation of experimental results.
- Step 9: Undertaking a confirmatory run of the experiment.

4.3 Design and Evaluation

Most industrial processes are composed of multiple variables (factors) with multiple levels or settings that influence the outcome of the process. There are three principle methods of examining these factors to determine the optimal levels, or combination of levels. Studying one factor at a time is an option, but only in a simplified case. For a complex process that involves a number of factors, this method would take too much time and the subsequent results would not be representative of how the factors interact. A full factorial method was developed to test all possible level combinations amongst the factors. This is certainly a thorough exercise but is not generally feasible due to time and budget constraints. As an example, an experiment involving 4 factors with 3

levels each would require 81 (3^4) individual tests to satisfy the requirement of all possible combinations. Taguchi developed a set of standardized orthogonal arrays (OA) that utilized a fractional factorial method to reduce the number of tests but still provide insight into the process. With this approach, the experiment with four 3-level factors requires the use of an L-9 OA, meaning only 9 tests need to be performed instead of 81.

4.3.1 Orthogonal Arrays

An orthogonal array is selected once the number of factors and levels for an experiment have been decided. The OA defines the number of trials (left most column) to be carried out and prescribes the combination of levels for each trial. Orthogonality requires the columns of the array be balanced; hence, each column has an equal number of levels distributed in an orderly fashion (not random) and there is also an equal number of level combinations between any two columns. These features are illustrated in the L-9 OA in Figure 4-1 (details in Chapter 6). Note how in every column each level (1, 2, or 3) appears three times and that in any two columns every possible number combination appears once.

Figure 4-1 L-9 Orthogonal Array

	A	B	C	D
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

4.3.2 Evaluation

An experiment with a single performance objective can be quite simple to evaluate when the QC is known. An experiment testing a heat treatment process may simply require high hardness, and consequentially the trial that produced the highest overall hardness may be selected for the optimized process. With multiple performance objectives it is often necessary to formulate an overall evaluation criterion (OEC) that acts as a single result for each trial. It is recommended that, initially, results be evaluated based on each objective separately and then compared to determine if the same optimum factor levels are indicated. If this method fails to produce the ideal factor levels, the performance evaluations for each trial are combined into a single index, the OEC, and evaluation is completed according to the QC of the OEC. A method for determining the

OEC for a set of data is given in Roy (2001) and this task is performed for the present research in Chapter 6.

The next level of analysis involves calculating the relative influence of each factor, the confidence interval (CI) on main effect of factors, and an error term that incorporates the influence of additional outside factors and experimental error, using analysis of variance, or ANOVA. ANOVA is a very well known procedure with techniques given in Roy (2001) and Ross (1988) to determine statistically significant factors.

4.4 Industrial Applications

Recent successful applications of the Taguchi method show how powerful this tool is for process optimization. For instance, five submerged arc hardfacing parameters are tested at two levels using an L-8 array (Tarng et al, 2002); optimal process conditions are found using ANOVA to provide a decrease in dilution, and an increase in deposition rate and hardness. An L-8 OA was also used in a study to optimize plasma- and detonation gun-sprayed alumina coatings for wear and hardness (Saravanan et al, 2000). ANOVA calculations for both spray methods led to the identification of key parameters that contributed to denser coating microstructures and hence improved hardness. Research on plasma surface hardening of steel (Yang, 2001) determined optimal plasma arc parameters to improve hardness depth results from 0.08-0.37 mm to 0.35-0.37 mm, increasing the average depth by more than twofold. The experiment employed a 4-factor 3-level L-9 orthogonal array.

Chapter 5: Research Objectives and Description

5.1 Scope

This thesis is the result of Materials and Manufacturing Ontario (MMO) funded research in partnership with Deloro Stellite. The main focus of the research was to determine PTA hardfacing parameters for two blade-hardfacing alloy material systems through Taguchi DOE methods. Other tasks were identified to complement this main goal and develop a better overall understanding of deposit/substrate interaction and performance. These objectives are as follows:

1. Establish baseline PTA parameters suitable for a small-size substrate component.
2. Identify possible deleterious interfacial phases by mixing the substrate and hardfacing materials at varying ratios and analyzing the resulting alloys using differential scanning calorimetry, scanning electron microscopy, energy dispersive spectroscopy, and X-ray diffraction.
3. Obtain performance data such as hardness in deposit and HAZ regions, extent of cracking, interface bonding, and porosity in the deposit to support the DOE methods of determining optimized PTA hardfacing parameters.

The remainder of this chapter describes the materials, methods, and equipment that were instrumental in this research. Subsequent chapters will detail the experimental design based on Taguchi methodology and present the results of microstructural, mechanical, and statistical analyses.

5.2 Research Materials

Materials required to conduct the research presented here encompassed a representative turbine blade material and two established hardfacing alloys. While the three alloys are widely used in various applications, this was the first attempt to combine these materials with each other using the PTA hardfacing process.

5.2.1 Turbine Blade (Substrate) Material

The blade material chosen for this study was Inconel 738, a widely used nickel-based superalloy with nominal elemental composition of (max. wt. % values) 0.20C, 16.30Cr, 2.00Mo, 2.80W, 9.00Co, 1.10Nb, 2.00Ta, 3.70Ti, 3.70Al, 0.05Fe, 0.015B, 0.15Zr and balance is nickel. The material is commonly referred to as IN738 or IN738 LC, where the LC stands for “low carbon” and the compositions of C, B, and Zr are modified for improved castability of large sections. IN738 is strengthened primarily by γ' -Ni₃Al and MC carbide precipitates in a γ matrix. The carbides can greatly affect the mechanical properties of the material, frequently growing in interdendritic and grain boundary regions. Factors controlling the growth of these carbides are the following: microsegregation levels of the carbide forming elements, morphology of the γ matrix, and local solidification time. For the coarse dendritic growth characterized by the vacuum investment casting process used to manufacture IN738 turbine blades, a script-type morphology MC carbide is produced (Liu et al, 1994). See Figure 5-1 and Figure 5-2 for examples of carbides and γ' phase found in the IN738 used for this study.

Figure 5-1 Carbides in IN738

Figure 5-2 γ' Phase in IN738

Both yield strength and ductility of IN738LC are temperature dependent. From room temperature, yield strength decreases up to 450°C; strength then increases to a maximum in the region between 750-850°C, where any further increase in temperature again produces a decline in strength (Bettge et al, 1995). Elongation differs greatly between room temperature and high temperature regions, with an elongation at yield of 2% at room temperature and 12% at 750°C. Elongation continues to increase with temperature to 950°C; above this temperature dislocations can bypass γ' precipitates by climb processes and the γ' morphology becomes unstable, undergoing directional coarsening of the precipitates. Fracture mechanisms include linking of pre-cracked carbides and decohesion of the carbide/matrix interface, occurring at higher temperatures. The principal fracture mode associated with these mechanisms is intercrystalline fracture since most carbides are present at grain boundaries (Bettge et al, 1995).

The γ' phase volume fraction for polycrystalline IN738, in the aged condition, is usually between 40-45% and provides the material with comparable temperature-dependent deformation mechanisms as single crystal superalloys with high (60-80%) γ' volume fractions (Bettge et al, 1995). Processes that involve local melting and rapid solidification, such as grinding or welding, can dissolve the γ' precipitates leading to a γ' depletion zone (Osterle et al, 1994). A post process heat treatment is required to partially restore the material's properties.

5.2.2 Substrate Form

IN738 was used in three different forms during this research. Initial testing of the TIG and PTA processes were carried out on the airfoil section of actual industrial gas

turbine blades. Two investment cast, artificially aged IN738 turbine blades received from Microfusione Stellite in Italy were used, an example of a partially prepared (cut) blade is shown in Figure 5-3.

IN738 in fine powder form was needed for controlled composition analysis using differential scanning calorimetry, and for preparing larger castings of mixed alloy composition for X-ray diffraction. The pre-alloyed powder was of the type used for laser powder deposition.

Figure 5-3 IN738 Turbine Blade

PTA parameter testing was carried out using small, 5/8" thick, rectangular (1.25 x 4.50 in) IN738 coupons. These coupons were cut from an investment cast block (8.50 x 4.50 x 1.25 in) cast at Deloro Stellite, as described in Section 5.3.3, and were subsequently heat treated at 1120°C for 2 hrs 40 min, secondary aged for 24 hrs at 845°C, and ground on the intended hardfacing surface to remove burs and oxides. The coupons

could accommodate 2 deposits, one from each end towards the middle, hence nine plus one spare were prepared. Figure 5-4 depicts three test coupons, two have received their hardfacing deposit while the third awaits parameter programming of the PTA equipment.

Figure 5-4 IN738 Test Coupons in Process

5.2.3 Hardfacing Deposit Materials

Two commercial Co-based hardfacing alloys, Stellite 694 and Tribaloy T-800, are being considered for wear protection of IN738 turbine blades in this research. Wear resistance of the two alloys is derived from two very different microstructural features. Stellite 694 has a high carbon and tungsten content, relying on carbides and solid solution strengthening, while T-800 has a high molybdenum content and silicon, leading to a high

volume fraction of an intermetallic Laves phase. Stellite 694 and Tribaloy T-800 were provided in the form of plasma transferred arc (PTA) hardfacing pre-alloyed powders; compositions are given in Table 5-1. An example of the powder granule size and shape is given in Figure 5-5 for Stellite 694, while T-800 exhibits similar features.

Table 5-1 Compositions of the Hardfacing Alloys (wt. %)

	Stellite 694	Tribaloy T-800
C	0.85	0.08
Si	1	3.4
Mn	1	0
Cr	28	17.5
Ni	5	1.5
W	19.5	0
V	1	0
Fe	3	1.5
B	0.1	0
Mo	0	28.5
Co	Bal	Bal

Figure 5-5 PTA Hardfacing Powder (Stellite 694)

5.2.4 Interface Representative Alloys

To investigate the effects of different deposit/substrate dilution levels, a controlled compositional study was undertaken. Representative interface mixture samples were created by mixing each hardfacing alloy with the substrate (IN738) alloy; the mixing ratios are detailed in Table 5-2. The powders were mixed mechanically in small quantities and specimens were cast in alumina crucibles during DSC analyses. Two heating cycles up to 1500°C, under an inert argon atmosphere, were used to ensure complete mixing, thus creating “new” alloys. The DSC process is described in Section 5.3.1.

Table 5-2 Sample Mixture Ratios

	wt. %	wt. %	wt. %
Sample #	694	IN738	T-800
1		100	
2	100		
3	5	95	
4	25	75	
5	50	50	
6	75	25	
7			100
8		95	5
9		75	25
10		50	50
11		25	75

5.3 Analytical Methods and Equipment

The following sections describe the methods and equipment used to produce all test pieces for this research and any apparatus used for specimen analysis.

5.3.1 Differential Scanning Calorimetry

Thermal analyses were performed using a Netzsch DSC 404C Pegasus (Netzsch-Geratebau GmbH, Germany), as shown in Figure 5-6. Alumina crucibles were used for both reference and sample containers, the reference crucible was left empty. Lids were also used to prevent possible elemental loss during the process. Powder mixture samples were measured into 35-40 mg specimens. Masses of each specimen and both crucibles are recorded before and after each DSC run. With the specimen and reference crucibles in place, the furnace cavity is evacuated to high vacuum using a foreline (mechanical) pump and a turbomolecular drag pump. Testing under vacuum can be done; however, an inert

atmosphere was used to prevent the loss of more volatile elements, in addition to the use of the lid. Argon at 50 mL/min is used to backfill and maintain a slightly positive pressure within the furnace; it is also used as the furnace protective gas. Heating and cooling are controlled through a TASC 414/4 controller (Netzsch-Geratebau GmbH, Germany) linked to PC based software, where the segments of the heating/cooling program can be set. The heating/cooling rate was set to 20°K/min with a 10 minute hold at the 1500°C maximum and a 5 minute hold at 400°C between the two cycles. After the second cycle, the cooling rate is not controlled below 400°C. Output to Netzsch data analysis software produces plots of DSC signal (mW/mg) versus temperature (°C). Of utmost concern in this research is the second cycle cooling curves, which provide an indication of the number of phases precipitated in the substrate/hardfacing alloy mixture based on the number of exothermic peaks. A simplified operating manual was prepared by the author to support other students' work with the DSC, it is included in Appendix A.

Figure 5-6 Carleton DSC Facility

a) Controller b) Power Supply c) PC Interface d) Gas Flow Meter e) Furnace/Specimen Holder f) Vacuum Controller g) Digital Scale

5.3.2 Castings using the DS Facility at Carleton University

Based on compositional analysis results of the mixed alloy DSC specimens, it was decided that XRD would be performed on the “worst-case” mixtures to identify all precipitated phases. The 50/50 mixtures were selected due to the abundance of phases present in both the Stellite 694/IN738 and Tribaloy T-800/IN738 mixtures. XRD requirements for sample size (area) precluded the use of inherently small DSC specimens; necessitating the casting of larger samples for the 50/50 mixtures to ensure at least 1 cm² of flat, prepared surface area could be produced by sectioning.

To cast an appropriate sized sample a conical, 99.8% alumina crucible with a 10 mL capacity was used, illustrated in Figure 5-7. A high purity alumina is required to prevent contamination of the metal in molten state. Constituents of the castings were as follows:

- 694/738 casting – 22.71 g IN738 + 22.72 g Stellite 694
- T-800/738 casting – 23.20 g IN738 + 23.43 g Tribaloy T-800

Figure 5-7 Dimensions of Alumina Casting Crucible

The DS furnace at Carleton University is a modified Bridgman-type (Saari, 1999) and was chosen to cast these samples because of availability, temperature and vacuum/inert gas capability, and for its capacity to rapidly solidify the castings by withdrawal into the cool zone. The furnace was originally manufactured by Centorr Vacuum Industries of Nashua, NH. It consists of a water-cooled furnace chamber with an upper hot zone and lower cool zone separated by adjustable baffles. The tungsten mesh heating elements in the hot zone provide maximum operating temperatures of 2200°C for the top heater and 2000°C for the lower heater. The casting charge sits on a copper chill plate that can be raised and lowered with the pedestal travel system; a programmable ball screw actuator arrangement. This system allows the mould to be moved in and out of the hot zone at a maximum speed of 30"/min (762 mm/min). Low vacuum is attained by

“roughing” the chamber with a Leybold D30A dual-stage rotary vane mechanical pump. High vacuum can be achieved, in the range of 10^{-5} torr (1.33×10^{-2} Pa), using an Edwards CR160/700 cryo-cooled Diffstak Mk 2 diffusion pump. High vacuum was desirable, before casting proceeded, to rid the chamber of oxygen that can react with titanium contained in IN738. At 1000°C the chamber was backfilled and slightly pressurized with argon to discourage the evaporation of aluminum, also a component of IN738. Heating then continued up to 1500°C where the temperature was held for 15 minutes before rapid (maximum rate) withdrawal of the mould into the cool zone. Figure 5-8 through Figure 5-10 show the Carleton DS facility, furnace chamber schematic, and mould set-up respectively.

Figure 5-8 Carleton University DS Facility

Figure 5-9 DS Furnace Chamber Schematic (Saari, 2003)

Figure 5-10 Casting Mould Set-Up

5.3.3 Deloro Stellite Investment Casting

Vacuum investment casting of the IN738 substrate blocks was completed by Deloro Stellite located in Belleville, ON. The investment casting process (*c.f.* Section 2.3.1) is composed of many steps, detailed in Table 5-3 specific to Deloro Stellite. Final casting was accomplished using a Radyne Limited induction melt furnace contained within a chamber evacuated by a Balzers Turbopump.

Table 5-3 Investment Casting at Deloro Stellite (Day, 2002)

Step	Comments
Permanent Die	Permanent dies are machined either in-house or by the customer for casting wax moulds. The dies can be quite complex, encompassing several parts to create the desired internal cavity to cast the wax. For this reason, the dies are usually very expensive and are carefully stored for future use.
Wax Mould Casting	The die is assembled by hand and inserted into a machine that seals the die and injects the proper amount of wax. The wax solidifies very quickly and is removed from the die immediately. The die must be completely disassembled to remove the wax mould, it then reassembled for another injection.
Wax Mould Assembly	To maximize process efficiency, several wax moulds can be attached to a rack, or "tree," to cast several parts at the same time. At this time, the sprue, runners, risers, and any test coupon moulds are added to the wax tree.
Ceramic Mould	A ceramic shell is gradually built up around the wax mould by dipping the wax tree in a slurry then in fine sand. This is repeated several times while increasing the coarseness of the sand and viscosity of the slurry. The fine slurry and sand provides a good surface finish while the coarser material provides more strength to the mould. The finished moulds are hung on a rail system to dry.
Wax Melting	Ceramic moulds are placed in an autoclave to harden the ceramic shell while melting and draining the wax from the internal cavities. Wax can be reclaimed and reused.
Pre-Casting	Before molten metal can be poured into the ceramic shells, a filter is installed in the sprue to control the turbulence of the liquid metal and to filter oxides and slag that can form on exposed molten surfaces. In addition, any porosity in the shell is noted and repaired with a putty-like material. Finally, the shell is pre-heated in a furnace just prior to casting to minimize the thermal gradient between the molten metal and the walls of the mould.
Casting	Casting is accomplished with one of three pouring options: either via a spout in a medium sized tilting furnace, by a smaller furnace that flips inverted, minimizing the molten metal exposure to air, or under vacuum within a chamber.
Post-Casting	Once the metal has solidified and cooled, the ceramic shell is broken off and the cast part is cut from the tree and machined.

5.3.4 Heat Treatment

Heat treatment of the IN738 PTA hardfacing coupons was carried out in two stages to accomplish the artificially aged service condition. The initial treatment of 1120°C for 2 hrs 40 min required the assistance of the National Research Council (NRC) Structures, Materials, and Propulsion Laboratory, for use of the Model FC700 Oxy-Gon Industries Inc. front-loading vacuum furnace. Precipitation hardening was performed at Carleton University after waterjet cutting (Section 5.3.5) of the coupons. Since the aging treatment at 845°C for 24 hrs does not produce appreciable surface scale, it did not require a controlled atmosphere and the process could be completed using the Type 51442 Lindberg furnace (max. temp. 1200°C) in the Mechanical and Aerospace undergraduate materials laboratory, Figure 5-11.

Figure 5-11 Lindberg Heat Treatment Furnace

5.3.5 Coupon and Sample Cutting

Cutting operations, at times, posed some problems due to the high toughness or poor machinability of the materials under study. Choosing the appropriate process and equipment required consideration of cost, lead time, and how cutting operations would affect the material. Two types of cutting operations were conducted during this project: sectioning for microstructural samples, and cutting of a cast IN738 block into test coupons.

Coupon manufacturing involved cutting the 5/8" thick test coupons from the cast IN738 block. Several options, such as EDM (electro-discharge machining) and abrasive cutting, were available but time and budgetary constraints resulted in the choice to waterjet cut the coupons at Accu-Fab Metal Products Limited, of Carp, ON. Their Esab Cutting Machine waterjet was operated at 55 000 psi with a 0.030" nozzle diameter; 60/80 garnet grit was added to the water stream. A test cut was required to determine the optimal traverse speed of 0.5"/min.

Sectioning of metallographic samples should be done with adequate means to prevent heat and gouging damage to the surface of interest. For this reason, high-speed abrasive cutting with no cooling or lubricant and band-saw cutting are not recommended. Abrasive cutting for samples used in this research was done at Carleton University using a Buehler Samplimet 2 (Figure 5-12a), which is water cooled, and a Buehler Isomet 1000 (Figure 5-12b), which employs an oil reservoir to provide cooling and lubrication to the abrasive blade.

Figure 5-12 Abrasive Cutting Equipment

Sample sectioning of the PTA hardfacing coupons immediately following deposition was necessary to allow interface examination with the SEM. This cutting was done at Deloro Stellite using their Flow International Corporation BENGAL waterjet cutter.

5.3.6 Hardfacing Operations

TIG samples were produced in the engineering machine shop at Carleton University. A Miller Syncrowave TIG torch is manually operated by an experienced welder, however the hardfacing process was new to the machine shop staff. A small plate of IN600 was used as a practice material for the welder to test the weld parameters and the behaviour of the hardfacing materials. The test weld of one bead of each material was carried out under the following conditions:

- Set current = 115 A
- 14-18 VDC straight polarity

- Shield gas flow rate = 30 CFH
- 1/8" tungsten electrode
- Pre-heat on hot plate = 200°C
- Arc start on carbon block (not on base material)

As a result of this test, the welding parameters were confirmed and approximate welding speeds were found to be 0.142 in/s for T-800 and 0.187 in/s for Stellite 694. Welding of the test samples was accomplished on the flat surface of the low-pressure side of one sample blade. Three beads each of Triballoy T-800 and Stellite 694 were applied to the tip and root areas of the blade respectively to form one larger finished bead. Upon completion of the weld, the blade was placed on a hot aluminum slab and transported in an insulated container to a furnace for stress relieving. The welded blade was treated at 550°C for one hour and air-cooled.

All PTA hardfacing operations were performed at Deloro Stellite using a Starweld 300A PTA system outfitted with a Model 600 PTA surfacing torch, both manufactured by Stellite Coatings, Goshen, IN. The Starweld system features an industrial power console that also houses the water refrigeration unit, both specifically designed for the PTA process. A control pendant allows the operator to program and manually manipulate the torch motion and welding parameters. Used in conjunction with the Starweld system, the Model 600 torch is designed for the nominal parameters given below:

- Current: 20 – 250 Amps
- Voltage: 15 – 45 Volts DC
- Water: 0.6 – 1.1 gal/min @ 60 – 90 psi (70°F)
- Powder Gas: 1.18 – 4.01 L/min (2.5 – 8.5 ft³/hr) @ 30 psi

- Shield Gas: 7.5 – 18.88 L/min (16 – 40 ft³/hr) @ 30 psi
- Plasma Gas: 1.18 – 4.01 L/min (2.5 – 8.5 ft³/hr) @ 30 psi
- Electrode Setback: 0.125" (using provided tool)

Some settings that are required for PTA hardfacing do not greatly affect the quality of the deposit or are one-time settings and henceforth are not included in the parameter study. Table 5-4 provides the factors that were constant for all hardfacing processes.

Table 5-4 PTA Set-Up: Constant Parameters

Condition/Parameter	Value
Nozzle	1/8"
Electrode	1/8" 2% thoriated tungsten
Shield gas	8 L/min
Initial current	40 A
Torch offset distance	~3/16"
DC Volts	20 V
Powder gas	1.55 L/min*
	1.76 L/min*
	2.10 L/min*
Oscillation width	1/4"
Oscillator	Dwell left = 0.007
	Dwell right = 0.007
	Speed = 0.67
Shield gas post flow	8 s
Power downslope	8 s
Powder downslope	4 s
Carriage start delay	1 s
Carriage displacement (length of deposit)	2.25"

*Dependent on plasma gas setting (low plasma gas corresponds to low powder gas setting).

The following figures illustrate important aspects of the PTA surfacing equipment, such as the nozzle, CNC gantry, and powder feed system, as labelled.

Figure 5-13 Gantry Mounted PTA with Powder Hopper and Torch Detail

Figure 5-14 PTA Control Console

Figure 5-15 PTA Pilot Arc Ignition

5.3.7 Metallographic Preparation

Bakelite was used to mount most metallographic samples in a Buehler Pneumet I mounting press:

- 25 mL red Bakelite phenolic powder
- Cure at 4000 psi for 20 min

Sample preparation involved wet grinding using 240-, 320-, 400-, and finally 600-grit abrasive paper on a Buehler Handimet grinder, followed by polishing using an Ecomet III polisher with an alumina/water mixture, and finally etching for ~30 seconds with Marble's Reagent (Vander Voort, 1984):

- 10 g CuSO_4 + 50 mL HCl + 50 mL H_2O

Before any of the specimens could be viewed under a microscope, each was cleaned in a methanol-water solution using a Bransonic 220 ultrasonic cleaner for approximately 5 minutes, preventing residual contamination from the etching reagent.

5.3.8 Metallographic and Compositional Analysis

Carleton University

Phase and morphology characterization at low magnification was done using an Olympus PME 6 optical microscope (Olympus Optical Co. Ltd., Japan). Microanalysis was carried out using a JSM-6400 digital scanning electron microscope (JEOL USA Inc., Peabody, MA) at the Facility for Cryo Scanning Electron Microscopy and Microanalysis, both in backscatter (BEI) and secondary electron (SEI) modes. Qualitative compositional analysis was performed on the same specimens using a LINK eXL energy dispersive X-ray LZ-4 microanalysis system (Oxford Analysis Instrumentation, Eynsham, Oxford,

UK), at a voltage of 20 kV. Specific areas of interest were selected to acquire an EDS spectrum to verify compositional differences between visually identified phases.

NRC – IMTI

SEM analysis of hardfacing cross-sections from the PTA parameter test coupons was conducted at the NRC Integrated Manufacturing Technologies Institute (IMTI) in London, ON. XRD on the 50/50 cast alloy mixtures was also performed at this location.

The Hitachi S-3500N scanning electron microscope is fully PC controlled and capable of both secondary and backscattered electron imaging modes. The SEM was typically operated at a voltage of 25 keV, but specific parameters such as voltage, magnification, imaging mode, and micron scale, specific to each image, can be found in the text overlay at the bottom of each micrograph.

XRD was performed by Dr. Jianyin Chen, research officer at IMTI. A Phillips X'Pert X-Ray Diffractometer was used with the following conditions:

- Start Angle: 15°
- End Angle: 90°
- Step Size: 0.02°
- Time per Step: 2.00 s
- Scan Speed: 0.01°/s
- Mo Tube: 60 kV, 40 mA

XRD results, including spectra for both Stellite 694 and Tribaloy T-800 mixtures with IN738 and tables of identified phases, are included in Appendix B.

LEO

Several samples were sent to LEO Electron Microscopy Group in Germany for SEM, EDS, and WDS analysis as a demonstration of their equipment capabilities. This testing was arranged by Meridian Scientific of Stittsville, ON. High resolution imaging of hardfacing deposit and interface microstructures was completed with a Gemini Supra 55VP, while EDS spectrums and WDS mapping of interfacial regions were prepared on a Gemini Supra 50VP.

5.3.9 Hardness Testing

Microhardness testing was performed using a Lietz Wetzlar Vickers microhardness tester. The apparatus employs a 5X magnification lens to locate ideal areas in the microstructure for testing, an adjustable bed equipped with micrometers, a diamond shaped indenter, and a 40X magnification lens to the indents for measurement. The eyepiece is capable of swivelling to facilitate measurement of both diagonal dimensions of the indent. These dimensions were measured using a scale in the eyepiece of the tester, the values are recorded, averaged, and a hardness value is then found by consulting a conversion table. For all tests conducted in this study, a 300g load was used.

Macrohardness testing of the PTA hardfacing test coupons, for use in the statistical analysis for process optimization, was performed with an Ames Precision Hardness Tester using the Rockwell C scale. This equipment was preferred over the microhardness tester as a material's bulk property is commonly measured in an industrial environment as part of process control procedures.

Chapter 6: Taguchi DOE for PTA Process Parameters

The Taguchi method for experimental design requires significant planning before testing can take place. This planning stage encompasses the identification of factors and levels to be tested and the subsequent choice of an orthogonal array, to defining the evaluation criteria and quality characteristic (QC). This chapter will detail and summarize the planning and setup for the PTA hardfacing fractional factorial test trials.

6.1 Process Factors and Levels

The first step in the experimental planning is to identify all the factors (parameters) associated with the PTA hardfacing process. Table 6-1 provides a comprehensive overview of PTA parameters, any of which could be used as factors in the test trials.

Table 6-1 PTA Hardfacing Parameters

PTA Parameters	Comments
Electrode material	2% thoriated tungsten
Electrode diameter/nozzle size	1/8" (12-150 A), 5/32" (25-175 A), 3/16" (50-250 A)
Powder feed rate	Continuous factor, units = g/min
Powder carrier gas flow rate	Continuous factor, units = L/min (setting dependent on plasma gas flow rate)
Powder carrier gas	Typically argon
Power (welding current)	Continuous factor, units = amps
Traverse speed	Continuous factor, units = percentage (CNC controlled)
Plasma gas flow rate	Continuous factor, units = L/min
Plasma gas	Typically argon
Shielding gas flow rate	Continuous factor, units = L/min (can be set constant)
Shielding gas	Typically argon
Pre-heat	Discrete factor, yes/no (with selected temperature)
Slow cool	Discrete factor, yes/no
Base material (substrate)	IN738 coupons, service condition (age hardened)
Powder Type (deposit material)	Discrete factor, Stellite 694 or Tribaloy T-800 (tested separately)
Offset distance	Continuous factor (set constant at ~3/16")

With this list compiled, the most significant factors must be selected as the factors to be tested in the study. This selection is based on information from a PTA Training Manual (Deloro Stellite, 1992) and discussion with an experienced PTA operator to insure the proper parameters are being investigated. Factor levels were chosen based on preliminary test deposits completed the year before to determine nominal values. Again, operator input was a valuable tool for the selection of nominal values and high and low level settings. The choice to use three levels for each parameter was based on the fact that non-linearity and trends can be easily identified, an inability with a two-level setup. Table 6-2 provides the selected test factors and levels used in the experiment.

Table 6-2 PTA Factor and Level Descriptions

	Factors	Level 1	Level 2	Level 3
A	Powder Feed Rate	12 g/min	14 g/min	16 g/min
B	Power (Weld Current)	90 A	94 A	98 A
C	Traverse Speed	33%	44%	55%
D	Plasma Gas Flow Rate	1.2 L/min (low)	1.5 L/min (nominal)	1.8 L/min (high)

6.2 Orthogonal Array

The selection of an orthogonal array (OA) runs in parallel with factor and level definition. By choosing a particular OA, the number of factors and levels that can be tested will be dictated, and vice versa. The two major orthogonal arrays considered for this research were the L-8 and L-9 arrays. An L-8 array would allow seven factors to be studied but only at two levels; whereas the L-9 array allows for a three-level setup with

four factors. Due to the benefits of testing with three levels, the L-9 OA was chosen to generate the test matrix. An L-9 OA can be seen in Table 6-3.

Table 6-3 L-9 Orthogonal Array

	A	B	C	D
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

The numbers 1 through 9 in the left-hand column denote trial numbers; however, it is not necessary to complete the trials in this order. To reduce the chance of error caused by re-setting the plasma gas flow rate for each trial, the like settings were arranged together to minimize adjustments to this parameter. Table 6-4 provides the final test matrix followed during experimental trials at Deloro Stellite.

Table 6-4 Test Matrix

Trial Condition 1			Trial Condition 6		
Powder Feed Rate	1	12 g/min	Powder Feed Rate	3	16 g/min
Power (Weld Current)	1	90 A	Power (Weld Current)	1	90 A
Traverse Speed	1	33%	Traverse Speed	3	55%
Plasma Gas Flow Rate	1	1.2 L/min (low)	Plasma Gas Flow Rate	2	1.5 L/min (nominal)
Trial Condition 2			Trial Condition 7		
Powder Feed Rate	2	14 g/min	Powder Feed Rate	1	12 g/min
Power (Weld Current)	2	94 A	Power (Weld Current)	3	98 A
Traverse Speed	3	55%	Traverse Speed	3	55%
Plasma Gas Flow Rate	1	1.2 L/min (low)	Plasma Gas Flow Rate	3	1.8 L/min (high)
Trial Condition 3			Trial Condition 8		
Powder Feed Rate	3	16 g/min	Powder Feed Rate	2	14 g/min
Power (Weld Current)	3	98 A	Power (Weld Current)	1	90 A
Traverse Speed	2	44%	Traverse Speed	2	44%
Plasma Gas Flow Rate	1	1.2 L/min (low)	Plasma Gas Flow Rate	3	1.8 L/min (high)
Trial Condition 4			Trial Condition 9		
Powder Feed Rate	1	12 g/min	Powder Feed Rate	3	16 g/min
Power (Weld Current)	2	94 A	Power (Weld Current)	2	94 A
Traverse Speed	2	44%	Traverse Speed	1	33%
Plasma Gas Flow Rate	2	1.5 L/min (nominal)	Plasma Gas Flow Rate	3	1.8 L/min (high)
Trial Condition 5					
Powder Feed Rate	2	14 g/min			
Power (Weld Current)	3	98 A			
Traverse Speed	1	33%			
Plasma Gas Flow Rate	2	1.5 L/min (nominal)			

6.3 Evaluation Criteria

The quality of a hardfacing deposit is evaluated by: hardness in the deposit; existence and extent of cracking; microstructural features in the deposit, interface, and substrate; and metallurgical bond at the interface. To evaluate the deposits, these attributes must be measured and/or quantified in some way to allow for numerical manipulation of the results. The measurements, or values, are the evaluation criteria (EC)

and are accompanied by a quality characteristic (QC) to allow interpretation of the numbers (i.e. bigger is better, smaller is better, or nominal is best).

Deposit hardness can be directly measured but is not the only important hardness value. It is also desirable to minimize any effects on the substrate material, suggesting that a hardness test of the HAZ would also be beneficial. Combining these measurements into a net hardness increase value (deposit hardness – HAZ hardness = net increase) simplifies the results and provides a bigger is better QC, which is useful later in the analysis.

A practical means of quantifying crack development was discovered and implemented during the experimental trials. Solidification and thermal strain cracking occurred during and immediately following deposition of the hardfacing deposit, resulting in an audible “tink” during crack initiation/propagation. The number of audible cracks were recorded for each test coupon and subsequently these values were subtracted from 10 to produce a bigger is better QC.

The remaining two evaluation criteria are subjective evaluations of the weld bead, both visually and in the cross-section using SEM. The visual evaluation was performed by an experienced PTA operator using a 0-10 scale with a bigger is better QC. The cross-sectional evaluation was accomplished with SEM analysis of the interface region at magnifications of 25X, 300X, and 1000X. The 0-10 scale was used with a bigger is better QC. A score of 0 was considered “very poor” with either no bonding or extensive intermixing and a large amount of visible cracking. A score of 10 was considered “excellent” with a good metallurgical bond, little evidence of intermixing, and minimal cracking.

6.4 Overall Evaluation Criterion

In the case where multiple evaluation criteria exist, it is useful to combine the values to produce an overall evaluation criterion (OEC), one number to represent the results for each test trial. The OEC can then be used for direct comparison of the trial conditions and in later ANOVA analyses.

Evaluation criteria values were tabulated in an Excel spreadsheet allowing for simple calculation of the OEC value for each trial using Equation 6-1.

$$\sum_{i=1}^n \frac{ECvalue_i - MIN_i}{MAX_i - MIN_i} \times weight_i \quad (6-1)$$

Where:

- n = number of evaluation criteria (4)
- $ECvalue$ = the measured or assigned value for a specific EC (such as hardness increase for trial condition 3)
- MIN = minimum value in an EC set of values
- MAX = maximum value in an EC set of values
- $weight$ = weighting assigned to the specific EC (see Section 6.4.1)

The above equation is for a bigger is better quality characteristic, hence the reasoning behind forcing all evaluation criteria to have this QC, only this equation was needed to calculate the OEC values and not a combination of equations. The calculation is repeated for each trial condition to produce a total of 9 OEC values for each material combination.

6.4.1 Weighting of the Evaluation Criteria for OEC Calculation

The OEC calculation given in Equation 6-1 allows for the use of a weight factor to categorize critical evaluation criteria. Weights are selected in terms of percent and should add up to 100. For this research there are four evaluation criteria, two with measured values and two with assigned subjective scores. It was determined that the measured quantities, audible cracks and hardness increase, should have higher weights based on their criticality in the overall quality of the hardfacing deposit. It was also determined that internal appearance (cross-sectional SEM analysis) provided more data pertaining to weld quality than the external appearance, and as such, received a higher weighting. Assigned weights are summarized in Table 6-5.

Table 6-5 Evaluation Criteria Weights

Evaluation Criteria	Weight
External Appearance	15%
Internal Interface Quality	25%
Audible Cracks	30%
Hardness Increase	30%
Total	100%

Chapter 7: Experimental Results and Discussion

This chapter presents the experimental results and related discussion from various aspects of the completed research. General results are given first to provide an insight into the materials studied in the research and problems present in current hardfacing techniques. This is followed by results of hardness testing and details of the PTA process optimization. The chapter concludes with results and discussion regarding the alloy mixture microstructural analyses.

7.1 General Results

7.1.1 Microstructure of the Research Alloys

Initial microstructural investigations were carried out for the three types of materials involved in this research, namely IN738, Tribaloy T-800, and Stellite 694. It was important to become familiar with the common microstructural features of these alloys as later work would focus on the effects of mixing the hardfacing alloys, T-800 and 694, with the blade alloy IN738, simulating mixing and dilution from hardfacing processes.

IN738

Sections of an investment cast IN738 turbine blade were cut for a preliminary analysis of the microstructure. Several micrographs were taken from different locations on the blade and at varying magnifications to understand the typical structure of the IN738 material. Figure 7-1 shows some common microstructural features that characterize IN738: large equiaxed dendritic grain structure, jagged grain boundaries,

high γ' volume fraction, and grain boundary carbides. Differences were observed between the root and tip sections of the blade. The tip exhibits a much finer dendritic structure compared to the root of the blade. Also, casting defects were more prevalent at the tip of the blade; more and larger interdendritic voids and porosity were found towards the blade tip. In addition to the defects found in the turbine blade samples, large amounts of porosity were observed in the centre section of the block cast to produce the PTA hardfacing coupons. Figure 7-2 illustrates these voids with a digital camera image of the central region of a sectioned cast block and a micrograph of a turbine blade tip sample.

Figure 7-1 Typical IN738 Microstructure at Increasing Magnifications

Figure 7-2 IN738 Casting Defects (Voids and Porosity)

Stellite 694

Microstructure of the Stellite 694 hardfacing alloy is shown in Figure 7-3, along with the DSC curve for this material in Figure 7-4. Evident in Figure 7-3 are three main constituents; matrix (α , Co base) (dark grey), primary carbides distinguished by white contrast (later confirmed to be WC), and a eutectic $\alpha + MC$. The DSC curve indicates four peaks that occur at various temperatures in the cooling cycle; hence a fourth microstructural constituent may exist in Stellite 694. It can be speculated that another type of carbide is present, such as a $M_{23}C_6$, M_7C_3 , or MC type, which could be precipitated from α -Co in addition to the primary WC during cooling. Peaks in the heating (upper) curve show the allotropic transformation characteristic for cobalt in the powder form as well as two melting peaks indicating the presence of two distinct phases in the Stellite 694 powder.

Figure 7-3 Multi-Phase Microstructure of Stellite 694 (DSC Cast Form)

Figure 7-4 Stellite 694 DSC Curve

Tribaloy T-800

Unlike Stellite 694, the Tribaloy T-800 alloy shown in Figure 7-5 reveals only matrix and Laves intermetallic phases. The identification of Laves phase is based on both the composition of the alloy, Mo and Si content, and past reported research (Wu and Redman, 1994). Blocky Laves phase constituents can become quite bulky, growing larger during casting than the initial powder size (~50 μm) and forming dendritic-type structures when conglomerated. The low magnification micrograph in Figure 7-6 illustrates this fact, showing the entire DSC cast specimen surrounded by the Bakelite mounting material (black).

Figure 7-5 Microstructure of Tribaloy T-800 (DSC Cast Form)

Figure 7-6 Low Magnification of Tribaloy T-800 (DSC Cast Form)

7.1.2 TIG vs. PTA Hardfacing

Several differences are evident when comparing TIG and PTA deposit and interface microstructures and morphology. TIG deposit (weldmetal) microstructure is more disordered and random in terms of dendrites and has finer dendritic features than those produced by PTA, indicating faster cooling rates. Since TIG hardfacing is done using welding wire, multiple passes are required to build-up the desired deposit width, resulting in noticeable differences in the microstructure between individual passes. This randomly distributed microstructure is not present in PTA deposits because an oscillation can be programmed into the CNC gantry to produce the required deposit width in a single pass. TIG hardfacing is also susceptible to contamination as the surface of the welding wire can become contaminated during handling. Again, PTA hardfacing avoids this issue by using a powder consumable, eliminating direct handling of the deposit material by the operator. The following figures (Figures 7-7, 7-8, 7-9, and 7-10) illustrate the differences found between TIG and PTA deposit microstructures for both Stellite 694 and Tribaloy T-800.

Figure 7-7 Optical Micrographs of TIG & PTA Stellite 694 Deposits

Figure 7-8 Optical Micrographs of TIG & PTA Tribaloy T-800 Deposits

Figure 7-9 SEM Micrographs of (a) TIG & (b) PTA Stellite 694 Deposits

Figure 7-10 SEM Micrographs of (a) TIG & (b) PTA Tribaloy T-800 Deposits

Performance of a hardfacing deposit is highly dependent on the interface between the deposit and substrate. The ideal interface will incorporate metallurgical bonding while minimizing the degree of intermixing between the hardfacing and substrate alloys. It is in this area that PTA hardfacing claims to vastly outperform TIG processes by reducing dilution between the two working alloys to as little as 5%. By comparing TIG and PTA interface microstructures, it is evident that the PTA process can produce a much narrower interfacial region, depending on the welding parameters. It is also clear from many optical and SEM micrographs, for both TIG and PTA deposits, that cracking seems to be concentrated in this interfacial region, either within the substrate, along the interface, or, less often, into the deposit. Figures 7-11 to 7-14 show that the PTA hardfacing process (with Stellite 694/Tribaloy T-800 on IN738) exhibits promising results but still requires work to optimize the processing parameters in order to improve the bonding while minimizing the effect on the substrate, reduce/eliminate cracking, and maximize the deposit hardness (covered in later sections).

Figure 7-11 Optical Micrographs of Stellite 694 TIG & PTA Interfaces showing Limited Dilution/Intermixing with PTA

Figure 7-12 Optical Micrographs of Tribaloy T-800 TIG & PTA Interfaces showing Intermixing with TIG and an Inferior PTA Interface

**Figure 7-13 SEM Micrographs of (a) TIG & (b) PTA Stellite 694 Interfaces
Illustrating Banding in TIG Interface and Good Quality PTA Interface**

Figure 7-14 SEM Micrographs of (a) TIG & (b) PTA Tribaloy T-800 Interfaces showing Large Interface Delamination in TIG and Discontinuous Cracks in PTA

7.1.3 Processing Problems Associated with PTA

One of the objectives of this research is to improve the quality of gas turbine blade hardfacing by replacing a TIG process with an optimized PTA process. It is obvious from the microstructural study/analysis in Section 7.1.2 that the PTA hardfacing process has great potential to outperform traditional TIG hardfacing methods. However, there are still some underlying problems with the PTA process that could be solved by parameter optimization.

Cracking and Defects

Cracking can occur as a result of solidification shrinkage, thermal shock (which can take place if the substrate is not pre-heated and has sufficient bulk mass to act as a heat sink), and liquation that occurs when grain boundaries adjacent to the deposit become partially melted in the hardfacing process, resulting in microfissures extending from the HAZ to the dendrite boundaries of the fusion zone (Su et al, 1997). Occasionally, unfused powder is present within the deposit creating defects at the interface between these particles and the surrounding consolidated material, which may act as a crack. Depending on the conditions (hardfacing parameters) and materials used, cracking and defects can occur, and are indicated in the microstructure within the substrate, seen in Figure 7-15, within the deposit, Figure 7-16 and Figure 7-17, or along the interface, Figure 7-18.

Figure 7-15 SEM Micrograph of Substrate Cracking Parallel to the Deposition Direction

Figure 7-16 Optical Micrograph of Cracking in a PTA Deposit

Figure 7-17 SEM Micrograph of Defect in PTA T-800 Deposit Near the Interface

Figure 7-18 SEM Micrograph of PTA T-800 Cracking within the Deposit and Interface

γ' Dissolution

An ideal sequence for PTA hardfacing of precipitation hardened materials would require solutioning of the substrate before deposition, pre-heating, slow cooling, and subsequent heat treatment to restore the microstructure to the proper service condition. This can become a lengthy and costly process, and is not feasible for field service; hence the motivation behind developing hardfacing parameters for processing materials in their service condition. Due to the large heat input and rapid cooling of the substrate material in the region of the deposit, a HAZ will possess a different microstructure than the substrate. Traditionally, a HAZ is identified by exhibiting a higher hardness than the surrounding bulk material in the solution treated condition. By processing the IN738 in the fully aged condition, the HAZ in fact undergoes solutioning and quenching, evident from a lack of γ' precipitates and a lower local hardness. The TIG process, with a higher heat input to the substrate than PTA, would be more susceptible to this problem. Figure 7-19 illustrates this by showing average microhardness results for PTA and TIG deposits and heat affected zones. The TIG HAZ reveals a lower hardness than that for IN738 in service condition. The PTA HAZ average hardness is slightly greater than the baseline IN738 value, indicating that γ' dissolution was likely not a factor. However, the series of micrographs in Figure 7-20 demonstrate how γ' dissolution can occur in IN738 with a PTA deposit, albeit at a very microscopic level. The γ' volume fraction gradually increases from close to zero near the interface (a) to a normal value (~40%) within the bulk (a to h) based on visual inspection. The micrographs were taken such that the bottom edge of an image roughly corresponds to the top edge of the successive image, starting from just below the interface and proceeding into the substrate.

Figure 7-19 PTA vs. TIG Deposit and HAZ Average Microhardness

Figure 7-20 γ' Dissolution in IN738 Incrementally from the PTA Interface (a to h)

7.2 Hardness Testing

The hardness of a material is easy to measure and is a useful indicator of many other properties and attributes. Knowing the baseline hardness of a material is important when later analyzing the effects of a heat treatment or welding process on the substrate material. For this reason, baseline hardness values for a sectioned IN738 turbine blade were established using Vickers microhardness. Rockwell C macrohardness values in the HAZ of the test coupons for process optimization were also measured. Material's wear resistance is closely associated with its hardness, as such, larger values of deposit hardness are, in most instances, advantageous. In order to reduce the standard deviation, Vickers microhardness and Rockwell C macrohardness were taken from the hardfacing coupons and averaged to give hardness values for both Stellite 694 and Tribaloy T-800; average hardness values are given in Table 7-1.

Table 7-1 Average Measured Hardness Values

Material	Average Hardness (HV)	Standard Deviation	Average Hardness (RC)	Standard Deviation
IN738	375	33	36	2.42
Stellite 694	575	36	45	2.65
Tribaloy T-800	618	43	51	4.38

Hardness profiles are taken through the hardfacing deposit and into the substrate to show the hardness distribution across the weld interface. An ideal hardness profile would feature a high, consistent hardness in the deposit with a smooth but sharp drop at the interface to a value equivalent to the substrate baseline hardness. As can be seen in Figure 7-21 and Figure 7-25, the general trend is followed but some deviations from the ideal are present. There are three trends observed from both microhardness and metallographic examination. Curve (a) in Figure 7-21 and Figure 7-22 represent poor bonding between a PTA 694 deposit and the substrate. As there is no intermixing between the deposit and substrate, the hardness in the HAZ is reduced abruptly due to the dissolution of γ' . The same trend is observed with curve (a) in Figure 7-25 and Figure 7-26, but for a PTA T-800 deposit. A favourable PTA 694 deposit/substrate interface is shown in Figure 7-21 curve (b) and Figure 7-23, where an increased HAZ hardness is a result of limited mixing of Stellite 694 with IN738. While the HAZ γ' in this case has also been dissolved, the hardness is not significantly reduced as some percentage of Stellite 694 has diluted into the HAZ and is contributing to the increased hardness value, compared to Figure 7-21 curve (a). Again, a similar trend is observed in Figure 7-25 curve (b) and Figure 7-27 for Tribaloy T-800. Although some degree of intermixing is advantageous in ensuring a good metallurgical bond, excessive intermixing, or dilution, may not be desirable as significant hardness reduction could occur. This is observed when hardfacing IN738 with TIG. Evident in curve (c) of Figure 7-21 and Figure 7-24, hardness reduction in the deposit is a result of excessive intermixing and higher heat input. An increase in HAZ hardness shown by curve (c) in Figure 7-25 is attributed to the

increase in percentage of Tribaloy T-800 in the region. This observation further emphasizes the difference between PTA and TIG.

Figure 7-21 Stellite 694 Harness Profiles

Figure 7-22 SEM Micrograph of PTA 694-06

Figure 7-23 SEM Micrograph of PTA 694-07

Figure 7-24 SEM Micrograph of TIG 694

Figure 7-25 Tribaloy T-800 Hardness Profiles

Figure 7-26 SEM Micrograph of PTA 800-06

Figure 7-27 SEM Micrograph of PTA 800-07

Figure 7-28 SEM Micrograph of TIG T-800

A major difference between PTA and TIG hardness profiles is the sharp dip in hardness at the interface of PTA samples followed by a slight rise and subsequent drop to the nominal substrate value for the PTA specimens; while the profiles for the TIG specimens exhibit a more smooth transition from the deposit to base material. This dip is especially pronounced in the -06 specimens where there is a distinctive line between the weld and base metals.

Also noticeable in the hardness profiles is the robustness of the Tribaloy T-800 hardfacing alloy when considering processing parameters and hardfacing method. This is evident from the narrower band that all three T-800 hardness profiles lie within, compared to the Stellite 694 hardness profiles, especially on the deposit side of the plots.

7.3 PTA Process Optimization

Chapter 6 describes the methods used to evaluate and optimize the PTA hardfacing process for Stellite 694/Tribaloy T-800 on IN738. Included below are evaluation results and the overall evaluation criteria for the 694 and T-800 experimental trials. Example micrographs are given to show how the deposit cross-section and interface change with the processing parameters. The final result of the evaluation and statistical analysis is the prediction of an optimal combination of parameter settings for each material combination and the percent influence each of the studied factors has on the overall quality of the hardfacing deposit.

7.3.1 Process Evaluation

Data obtained for process evaluation was based on visual observation, hardness, and detailed microstructural analysis. All experimental data, including external and internal evaluations of the hardfacing deposits, were quantified to help incorporate that information into the OEC and allow further statistical analysis. Table 7-2 provides all the data used for evaluation of the hardfacing coupons.

Table 7-2 Hardfacing Coupon Evaluation Data

Sample	Audible Cracks	Deposit Hardness (RC)	Subst. Hardness (RC)	External Eval.	Internal Eval.
694-01	2	47.33	38.00	4	4
-02	1	44.00	34.67	4	8
-03	1	44.33	34.00	3	2
-04	0	47.67	36.00	6	4
-05	1	45.33	34.00	5	6
-06	1	48.00	30.00	4	0
-07	0	39.33	34.00	10	10
-08	1	44.33	35.33	5	0
-09	1	44.33	40.00	3	8
T-800-01	2	55.67	38.00	5	4
-02	4	54.67	35.33	4	5
-03	3	49.33	38.33	3	6
-04	1	50.67	35.67	6	5
-05	1	42.67	38.67	5	9
-06	5	56.67	39.33	9	1
-07	5	50.67	36.00	10	8
-08	4	47.67	35.00	4	2
-09	1	49.33	36.67	4	7

It is easy to see from the above table that no one coupon has the best performance in every column, making evaluation very difficult if just looking at the gathered data. By combining the numbers for the evaluation criteria into an OEC value for each sample, direct comparison between the samples is possible in addition to ANOVA calculations for the parameter setting predictions. Table 7-3 gives the hardfacing OEC values for each sample, with the top performing samples highlighted for each material.

Table 7-3 Stellite 694 and Tribaloy T-800 Coupon OEC Values

Sample	Stellite 694 OEC	Tribaloy T-800 OEC
-01	23.12	62.89
-02	48.12	52.14
-03	33.17	44.31
-04	62.54	70.44
-05	49.65	59.29
-06	47.14	38.95
-07	72.19	57.74
-08	29.53	29.72
-09	35.00	67.83

SEM micrographs of the best and worst overall OEC values for both Stellite 694 and T-800 are shown in the figures below. Not all cases necessarily had the best internal appearance score, however, generally it can be seen that the interfacial regions of the high-scoring samples are superior to those of the low scoring samples. Important features are noted on the images.

Figure 7-29 Poor Interface Bonding for Sample 694-01 (OEC = 23.12)

Figure 7-30 Good Interface Bonding for Sample 694-07 (OEC = 72.19)

Figure 7-31 Poor Interface Bonding for Sample 800-08 (OEC = 29.72)

Figure 7-32 Interfacial Region for Sample 800-04 (OEC = 70.44)

Figure 7-32 illustrates that while good bonding between the deposit and substrate is achieved, cracking in the base material can still be detected. Observations such as this indicate the need for further optimization iterations.

7.3.2 Factor Level Prediction

The best way of evaluating the affects of factor levels is by an average response graph. This graph plots the average response of each factor against the relevant levels, yielding the range of response and clearly identifying the level with the maximum value for each factor, hence the desired level setting for that factor. The average response value is found by summing the OEC for each factor at each level setting and then dividing by the number of results for each factor. Figure 7-33 illustrates this process, the summation yields a value of 191.07 for factor A at level 1, which is then divided by 3 (number of

OEC results) to give 63.69 as the average response for factor A1. This calculation is repeated for factors B, C, and D at all three levels, these values are then plotted in the average response graph. See Chapter 6, Table 6-2, for factor definitions and specific level values.

Figure 7-33 Schematic of Average Response Calculation (T-800)

Experiment	Factor A	Factor B	Factor C	Factor D	Result
1	1	1	1	1	62.89
2	2	2	3	1	52.14
3	3	3	2	1	44.31
4	1	2	2	1	70.44
5	2	3	1	2	59.26
6	3	1	3	2	38.94
7	1	3	3	3	57.74
8	2	1	2	3	29.72
9	3	2	1	3	67.82
		SUM		SUM	483.28
A1	191.07			Stand Dev	13.67
A2	141.14			Average	53.70

Figure 7-34 OEC Average Response Graph: Stellite 694

Figure 7-34 shows a wide range of average response values, indicating process sensitivity to factor level changes; and predicts parameter settings of: A1, B3, C3, and D2 to give the best average effect. It should be noted that this method can be used to predict optimal level combinations that were not tested in the experimental trials. The graph also indicates where further improvement may be possible since factors A, B, and C exhibit predicted level settings at the highest and lowest settings, suggesting that further increases or decreases of these level settings may produce superior results.

Figure 7-35 OEC Average Response Graph: Tribaloy T-800

Immediately apparent in Figure 7-35 is that the average responses for T-800 fall in a much narrower range and have higher overall values than for the 694 deposits. This suggests that the T-800 material contributes to a more robust process with less influence of the processing parameters on the overall quality of the hardfacing deposit. Predicted settings for T-800 are: A1, B2, C1, and D2; different from those for Stellite 694 and also

not tested together during the experiment trials. Predicted factor level settings are summarized in Table 7-4.

Table 7-4 First Iteration Optimized Parameter Settings

Factor	Stellite 694		Tribaloy T-800	
	Level	Value	Level	Value
A (Powder Feed Rate)	1	12 g/min	1	12 g/min
B (Weld Current)	3	98 A	2	94 A
C (Traverse Speed)	3	55%	1	33%
D (Plasma Gas F.R.)	2	1.5 L/min	2	1.5 L/min

7.3.3 Simple ANOVA

Sound statistical examination and advanced techniques such as S/N (signal-to-noise) ratio analysis require multiple samples for proper application. In other words, this would necessitate completing the nine experimental trials an additional four times, for each hardfacing material. While this would allow for more complex analyses and better prediction of parameter settings, it is beyond the scope of this study. Simple ANOVA is possible with the experimental data on-hand and produces results for percentage influence of the factors. This provides a comparison between the tested factors showing how sensitive the final deposit quality is to changes in the respective parameters. A low percent influence means that slight adjustments to that factor will not adversely affect the results. Figure 7-36 compares the percentage influence of the factors for each hardfacing material.

Figure 7-36 Comparison of Factor Influence on Hardfacing Deposit Quality

7.3.4 Using the Taguchi Method for Parameter Optimization

The Taguchi method for design of experiments not only presents techniques for experimental set-up and analysis, but also describes an entire philosophy to follow throughout the course of a project. The ideas put forward by Genechi Taguchi on how to manage and work together on quality improvement projects and on quality control itself were quite different from the industry norm. Many of Taguchi's ideas are common practice in industry today, notably the project planning meeting for project definition. This meeting requires the involvement of all personnel levels, from the manager running the project to the engineers designing the experiments to the shop technician operating the equipment. The purpose of this meeting is to have open discussion and input from all levels pertaining to all aspects of the project, and to plan every step from factors to investigate to how the results will be interpreted. From the perspective of an industry-based project group, this method is ideal; however, for the case of a university-based

research project many of the project planning aspects are not applicable or not feasible. Often, the only factor limiting the adherence to Taguchi's philosophy is time/availability of those involved at the industry level. Instead of a day-long meeting to completely define the project, input is sporadic and occasionally contradictory to what has already been planned. This problem could be easily solved by implementing Taguchi methods from project inception and scheduling the multi-level planning meeting well in advance to accommodate the availability of all those involved. Some persuasion may be necessary on the part of the researchers to convince industry representatives of the benefits such a meeting would provide.

Another area where university research falls short of maximizing the potential of the Taguchi method is with respect to the advanced analytical techniques developed to process experimental results. S/N ratio and error calculations are not possible without significant sample sizes of, say, 5 for example. While running process optimization experiments in an industrial manufacturing setting can prove quite simple, due to equipment availability and pressure from management, it is much different for a student trying to fit experimental trials into a busy production schedule. Therefore, only one set of experimental trials was completed (although other factors such as material cost and preparation time come into play). Conversely, the fractional factorial design of experiment method prescribed by Taguchi helps reduce the number of trials that must be completed to get useful data. In this respect, the Taguchi method proved invaluable by reducing the number of required trials by 89%.

Optimized parameter settings predicted by the average response calculations show that the predicted levels for powder feed rate and plasma gas flow rate are the same for

Stellite 694 and Tribaloy T-800. These factors are shown, on average, to have less influence in the hardfacing process, Figure 7-36. Also evident, is the fact that optimization for T-800 is further along than for Stellite 694. Two mid-level settings (weld current and plasma gas flow rate) were found to be at the correct settings based on the peak in the average response graph, compared to only one for Stellite 694. As well, the weld current was found to have the greatest influence on the quality of the hardfacing deposit for T-800, indicating that the material is most sensitive to changes in this parameter. Due to the very low percent influence of the plasma gas flow rate for Tribaloy T-800, and since the average response calculations show this parameter to be optimized, subsequent experimental trials (iterations) should investigate other factors that may contribute a higher influence on the performance of this process. It is here where the effects of pre-heating could be examined.

7.4 Microstructural Analysis of Interface Alloy Mixtures

Mixing or dilution between a hardfacing deposit and the substrate is inevitable because a metallurgical bond is needed to prevent spalling or delamination of the hardfacing material. While the goal is to minimize this intermixing, understanding how various mixture ratios affects the material in and around the interface will prove beneficial for material and process selection. The research on composition-induced microstructural variation encompassed data from differential scanning calorimetry, scanning electron microscopy, and energy dispersive spectroscopy (DSC, SEM, and EDS) for both Stellite 694 and Tribaloy T-800 mixtures with IN738. Appendix C provides a complete set of output plots for all DSC trials.

7.4.1 Stellite 694 Mixtures

All the DSC-cast specimens given in Table 5-2 were examined with SEM/EDS; however, a detailed microstructural study was conducted on the specimen cast with 50 wt% Stellite 694 + 50 wt% IN738 (50/50 694), as this was a likely “worst case scenario.” A vastly different microstructure was observed, seen in Figure 7-37, compared to the 100% Stellite 694 specimen in Figure 7-3.

Figure 7-37 50/50 694 Alloy Mixture at 200X Magnification

There is a high percentage of matrix (Co + Ni solid solution) shown in Figure 7-37 for the 50/50 mixture, and precipitates have primarily script type morphology. A qualitative EDS analysis was done at the location indicated by the arrow in Figure 7-38 and is given in Figure 7-39. The qualitative composition analysis combined with the distinct precipitate morphology, suggests that the script type phase could be (W, Ti)C.

Figure 7-38 50/50 694 Alloy Mixture at 600X Magnification

Figure 7-39 EDS Spectrum of 50/50 694 Script Precipitate

In addition to the script phase, another group of phases with eutectic features were found at higher magnifications, as shown in Figure 7-40. EDS analyses were performed on three different locations with distinctive contrasts (indicated by 1, 2, and 3 in Figure 7-40) and varying degrees of compositional difference were found in the spectra, as shown in Figure 7-41 a, b, and c respectively.

Figure 7-40 50/50 694 at 4000X Magnification

Figure 7-41 EDS Spectra from Indicated Regions in 50/50 694 (a) 1, (b) 2, and (c) 3

(a)

(b)

(c)

Phases 1 and 3 are clearly Cr rich phases that contain W, while phase 2 is a W rich phase with an addition of Cr. While there is some indication of the presence of C, it is not certain at this stage whether these phases are carbides or intermetallics. They do, however, differ significantly from the primary script type phase [(W, Ti)C]. Parallel to the SEM and EDS data, the DSC cooling curve, lower line in Figure 7-42, suggests there

could be four phases in the Co-Ni matrix. The nature of these phases will be discussed further in Section 7.4.3.

Figure 7-42 DSC Second Heating/Cooling Curve for 50/50 694

To demonstrate the gradual change in the microstructure due to the variation in the mixing ratio, optical micrographs at both low and high magnification were assembled to produce Figure 7-43; a graphical view of the change in composition is also given (Figure 7-44). The change in quantity, morphology, and nature of the phases can be observed in the precipitates from left to right in the figure. It also provided an exaggerated view of possible interface microstructures between the surface coating and the substrate.

Figure 7-43 Morphology Evolution Between IN738 and Stellite 694

Figure 7-44 Composition Change Between IN738 and Stellite 694

7.4.2 Tribaloy T-800 Mixtures

With the addition of 50 wt% IN738 into the T-800 alloy (50/50 T-800), the microstructure evolved from blocky shaped Laves in Figure 7-5 to a more eutectic form Laves phase (Figure 7-45) and the emergence of carbides, shown in Figure 7-48.

Figure 7-45 50/50 T-800 Alloy Mixture at 300X Magnification

Figure 7-46 EDS Spectrum from Location 1 (of Figure 7-45)

Figure 7-47 EDS Spectrum from Locations 2, 3, and 4 (of Figure 7-45)

EDS analyses show a typical Co-Ni-Cr solid solution matrix (point 1 in Figure 7-45 detailed in Figure 7-46) and possible Laves phase $(\text{Mo, Si})_x(\text{Cr, Co, Ni})_y$, highlighted by points 2, 3, and 4 in Figure 7-45 with the respective spectrum in Figure 7-47. The presence of carbides was revealed through examination at higher magnification. An arrow in Figure 7-48 indicates the location of the EDS analysis shown in Figure 7-49. These observations of microstructural constituents correspond well with the DSC cooling curve in Figure 7-50, where three distinct peaks are present. While the sequence of formation for these microstructural constituents is not known, the three phases can be identified as carbides, Co-Ni-Cr matrix, and Laves phase.

Figure 7-48 50/50 T-800 at 1000X Magnification

Figure 7-49 EDS Spectrum of Suspected Carbide in 50/50 T-800 (Location 1 in Figure 7-48)

Figure 7-50 DSC Second Heating/Cooling Curve for 50/50 T-800

A further increase in T-800 content up to 75 wt% (75/25 T-800) provided microstructures with a combination of Figure 7-5 (100% T-800) and Figure 7-45 (50% T-800), i.e. blocky Laves (points 2 and 3 in Figure 7-51 and in Figure 7-5) and eutectic Laves (point 4 in Figure 7-51 and Figure 7-45), in addition to a Co-Ni-Cr based matrix (point 1 in Figure 7-51). EDS analyses of all four points indicated on the micrograph correspond to the previous observations, a matrix-type composition in Figure 7-52 and Laves-type composition for points 2, 3, and 4 in Figure 7-53. No carbides were observed with this composition (Figure 7-54); however, the presence of carbides cannot be ruled out. The DSC cooling curve clearly shows the formation of only two phases during cooling (Figure 7-55), which supports the microstructural and EDS observations.

Figure 7-51 75/25 T-800 at 300X Magnification

Figure 7-52 EDS Spectrum from Location 1 (of Figure 7-51)

Figure 7-53 EDS Spectrum from Locations 2, 3, and 4 (of Figure 7-51)

Figure 7-54 75/25 T-800 at 1000X Magnification

Figure 7-55 DSC Second Heating/Cooling Curve for 75/25 T-800

7.4.3 Discussion of Alloy Mixtures

As described in Section 7.1.1, three prominent phases were observed in the cast Stellite 694; namely Co matrix, primary carbides, and eutectic carbides/Co. An allotropic phase transformation at 774°C, as shown in Figure 7-4, suggests that the Co matrix exhibits an HCP structure at room temperature for the pure Stellite 694 alloy. With the addition of IN738, the high temperature FCC structure is stabilized by Ni and Fe and no allotropic phase transformation can be seen for all the mixtures of Stellite 694 and IN738 studied; see Figure 7-42 as an example.

Precipitate morphology was drastically modified when Stellite 694 was mixed with IN738. By comparing Figure 7-3 and Figure 7-37, it can be seen that the total amount of precipitates have been reduced due to the composition changes. Most of the precipitates exhibit a script-type shape with the occasional occurrence of eutectic islands,

such as in Figure 7-40. It is believed from the EDS analysis that the script type precipitates are carbides containing large percentages of W, Ti, and Cr. It is not certain at present whether it is a $M_{23}C_6$, M_7C_3 , or MC type carbide, future work with XRD (X-Ray Diffraction) and WDS (Wavelength Dispersive Spectroscopy) could reveal the nature of the carbides.

While the occurrence of a script type precipitate, particularly a TCP phase, may be detrimental to the mechanical properties of the coated material (Sims et al, 1987), the possibility of the formation of intermetallic phases is of great concern. With the elevated W content in Stellite 694 and the addition of Mo from the substrate alloy IN738, the mixture may be prone to the formation of intermetallics.

The stability of the matrix can be characterized by the electron vacancy theory embodied in the PHACOMP numerical calculation algorithm, discussed in Section 2.5 and detailed in Appendix D. PHACOMP has been used very successfully in the modification of chemical compositions for both Ni- and Co-based alloys to avoid the formation of TCP phases.

PHACOMP calculations, based on the methods described in Murphy (1968), Sims (1987), and Calliari (2001), concluded that the mean electron hole numbers (N_v) are 2.47 and 2.57 for 25% and 50% mixtures of Stellite 694 in IN738 respectively, indicating the susceptibility of forming TCP phases. Further analysis using SEM/EDS suggested the existence of TCP phases. As shown in Figure 7-40, the island shaped constituent in fact contains several microscopic features, i.e. the bright blocky phase (labelled with “1”), the greyish phase (labelled with “3”), and the eutectic natured phase (labelled with “2”). It is speculated that phase 2 (containing W) emanates from phase 3 and depleted it of W, as

seen in Figure 7-41 (c). While both phase 1 and 3 contain similar chemical elements, phase 3 seems to be lower in W due the formation of phase 2. It is likely that both phase 1 and 3 are carbides and phase 2, formed from phase 3, is a TCP phase as predicated by PHACOMP theory.

The possibility of forming a TCP phase in the interface between hardfacing deposit and substrate is of concern when developing a hardfacing process technique. The formation of a TCP phase in the interface could potentially cause a loss of strength and ductility at service temperature, as well as loss of low temperature ductility (Sims et al, 1987). Judicious decision should be made when choosing the process methods (TIG, PTA, or laser) and process parameters in order to avoid or minimize the formation of TCP phases.

Tribaloy T-800, being a Laves type Co alloy, is less susceptible to the detrimental effect of TCP phases. Consideration should have been taken when selecting this hardfacing alloy to allow for reduced ductility. As seen in Figure 7-5, a typical Laves phase in Co matrix is formed during cooling and is well distributed throughout. During deposition of T-800 onto the substrate, intermixing (or dilution) will occur. Reported in one study on laser cladding using T-400 (similar composition to T-800, however with lesser Cr and Si contents) onto a steel substrate, as high as 12% coating material dilution was observed (Przybylowicz and Kusinski, 2000). Due to the dilution of the hardfacing material, the microstructure of the diluted area contains $\text{Co}_3\text{Mo}_2\text{Si}$ Laves phase, deviating from the nominal composition of CoMoSi.

In this study, the mixture with 75% T-800 and 25% IN738 produced microstructure containing primarily two phases, from both Figure 7-51 and Figure 7-54,

being Co-Ni-Cr matrix and Laves phase. The Laves phase is confirmed based on both previous microstructural observation of Tribaloy and EDS spectrum showing a prominent Si peak in Figure 7-53.

With an increase in the IN738 content to 50%, the blocky shaped Laves phase subsides. Instead, both script and eutectic forms of Laves are present, as shown in Figure 7-45. All the grain boundaries are heavily populated with precipitates as demonstrated in Figure 7-48 and the morphology of phase 1 in this figure resembles carbides. EDS analyses of both phases 1 and 2 were carried out; phase 1 contains mainly Ti while phase 2 showed a similar spectrum as Figure 7-47, i.e. Laves phase.

Since T-800 is designed to form an intermetallic phase, the mean electron hole number from the PHACOMP calculation is expected to be high for larger concentrations of T-800 in the mixtures. The 50% T-800 mixture yields a value of 2.60 and the 75% mixture gives a mean N_v of 2.68. Once an alloy transitions to a Co based matrix, the acceptable range for the mean electron hole number increases and phase prediction becomes more complicated with intentional intermetallics and abundant carbides that can nucleate TCP phases. Alternatively, by looking at the impact of dilution from the hardfacing deposit into the substrate, the concentration of the Ni-based alloy is still quite high, and the mean electron hole number will be a better indicator as to the susceptibility of forming deleterious phases. A good example is the 5% hardfacing alloy cases in the mixture study where the mean N_v for both Stellite 694 and Tribaloy T-800 in IN738 is 2.31. The calculated value is below the critical mean electron hole number for IN738 of 2.38 (Sims et al, 1987). This again emphasizes the importance of minimal

dilution/intermixing in the hardfacing process and points towards optimized PTA rather than TIG for hardfacing IN738 with Stellite 694 or Tribaloy T-800.

Chapter 8: Summary and Conclusions

It can be derived from the results presented in Sections 7.1.2 and 7.1.3 that further improvement in the hardfacing process, for both TIG and PTA, can be made. There are two reasons why this is true: a) because the process parameters had not been fully optimized at this stage, and b) because there were purposeful deviations in some common practices. Parameter optimization did show a marked improvement over the preliminary results, with a reduction in cracking and good interfacial bonding that minimized dilution. However, the results from the parameter optimization study were not perfect since, once again, some recommended practices were neglected.

When welding or hardfacing a precipitation hardened material it is common practice to solution heat treat the alloy before proceeding with the welding process. This puts the substrate alloy in its most ductile condition, reducing the chance of cracking due to large thermal strains and residual stresses. Once the welding or hardfacing is complete, and other post-process machining is finished, the component is heat treated to its age hardened state, usually involving another solution treatment followed by aging for several hours (24 hours in the case of IN738). All of these treatments require time, energy, and appropriate facilities. If these steps could be eliminated from the process when PTA hardfacing is used, it would become a much more versatile and attractive option for companies who operate gas turbines to conduct coating/welding and repair in-house. Repair and modification costs would be greatly reduced if blades could be processed in a matter of hours on-site instead of days at another facility. Therefore, it was determined at an early stage of this research that the decision to attempt hardfacing with the test coupons in service condition was justified.

Another normal procedure that was not used was pre-heating of the test coupons (substrate). Pre-heating is dependent on the nature of the deposit and substrate alloys and is not a requirement with many materials. However, working with very brittle materials such as Tribaloy T-800, a lack of pre-heating will certainly result in solidification cracking of the deposit, for example, due to the high temperature gradient experienced as the molten metal contacts the cold substrate. Industrial practice suggests that hardfacing with T-800 will require the base material to be red hot before deposition begins. This is accomplished with a large flame torch, and consequently there is little control over the specific temperature achieved in the substrate and subsequent heat treatments would be required to restore material properties. Therefore, pre-heating was not used in this study and the possibility of eliminating cracking by parameter optimization was attempted. However, extensive cracking suggests that pre-heating may have to be accepted as a part of this hardfacing process, particularly with T-800. New technology such as infrared heaters and high-temperature heating pads may be a solution. Temperature control would be much easier with this type of equipment, providing for heating to just under the age hardening temperature of the alloy, and hence not affecting the microstructure while drastically lowering the temperature gradient experienced during hardfacing. Another benefit of new, localized heating equipment may be the ability to re-precipitate γ' in the narrow band of dissolution just below the weld interface. This would require extensive study.

As detailed in the introduction, the objective of the research in this thesis was twofold: to develop an optimized set of processing parameters for PTA hardfacing with Stellite 694 and Tribaloy T-800 on IN738, and to understand the effect of the hardfacing

process on the microstructure at the deposit/substrate interface. The following conclusions can be drawn from experimentation and analyses conducted:

1. Preliminary hardfacing trials using turbine blade sections indicated that the PTA hardfacing process was superior to a TIG process based on geometry of the bead, interface dilution and bonding, and hardness profiles from the deposit to substrate material.
2. The hardfacing process is sensitive to changes in PTA processing parameters and the proper combination of those parameters. The wide range of deposit quality within the test coupons studied supports this statement.
3. Comparison between the test coupons using multiple criteria was facilitated by calculating an overall evaluation criterion, combining five evaluations and measurements into one number. By combining and averaging OECs of like levels, a prediction of optimal level combinations for each material combination was formulated. The predicted optimized level settings for each material combination were not tested during the experimental trials. This again shows the advantages of using Taguchi DOE because an optimal combination of processing parameters can be developed irrespective of whether those conditions were included in the experimental trials.
4. Average response graphs plotted for both Stellite 694 and Tribaloy T-800 indicate which levels are optimized and which require further testing. A peaked shape on the graph, for a particular parameter, shows that the level

2 setting had the best performance in the trials and any deviation, up or down, of that setting would lower the resulting deposit quality. For Stellite 694 the plasma gas flow rate was optimized, but the weld current and traverse speed indicated that higher settings may provide increased performance, while a lower setting for powder feed rate is suggested. Tribaloy T-800 results show optimized settings for weld current and plasma gas flow rate, while lowering powder feed rate and traverse speed settings could improve the process further.

5. The factors with the greatest influence on deposit quality were weld current and traverse speed for Tribaloy T-800 and Stellite 694 respectively.
6. At this time, under the tested conditions, no combination of parameter settings could produce crack-free test coupons. Further iterations of the optimization, combined with pre-heating of the substrate and slow cooling (recommended practice), could provide better results.
7. Tribaloy T-800 deposits had a higher overall hardness and less scatter of hardness values between different conditions and processes. In this respect, the T-800 alloy would provide better wear resistance and is more robust in terms of hardfacing technique and parameter settings. This is also supported by a greater average OEC for the T-800 test coupons.
8. Alloy mixtures, representing deposit/substrate interfacial regions, with mixture ratios between 25% and 50% (by weight) of the hardfacing component with IN738 are prone to forming deleterious phases and are

generally undesirable. Of these mixtures, the 50/50 Stellite 694 forms a more severe script type phase compared to the eutectic and blocky type structure of the 50/50 T-800 mixture.

9. Data from the DSC cooling curves correlated well with results obtained from SEM and EDS. Knowing the number of peaks on the cooling curve for specific alloy mixtures aided in the search for multiple phases using the SEM.
10. A hardfacing process that minimizes mixing between the deposit and substrate will prove superior. Based on previous data and conclusions, the PTA process outperforms a TIG process in this respect.

Chapter 9: Future Work

A continuation of this research should work towards an industrial application of the PTA hardfacing process using Stellite 694 or Tribaloy T-800 to deposit on IN738 turbine blade Z-sections. Optimized processing parameters have been predicted by statistical methods embodied in the Taguchi approach for design of experiments. It is important to run a validation test using the predicted level settings to determine if the quality is indeed improved over the previous experimental trials. This is a fundamental part of the Taguchi method. A second iteration would then be required to investigate those factors that indicated an optimal setting may be outside the range of the tested levels. Ideally, the goal would be to have peaks for all the plotted lines on the average response graph. At this point, the PTA parameters could be considered completely optimized.

Additional iterations are also required to determine the benefits of pre-heating the coupons before deposition. This is common practice, especially with difficult-to-weld alloys such as IN738 and Tribaloy T-800, and may prove to eliminate cracking altogether when combined with an optimized process.

The consequences of large dilution and intermixing between deposit and substrate are now understood. Evidence of TCP and detrimental carbide phases has been presented, but quantitative analysis using EDS/WDS must be done on actual PTA interfaces to determine the extent of dilution that occurs with an optimized process. In addition, XRD analysis of samples prepared with the determined mixture ratio of hardfacing and substrate alloys would identify the major phases that develop in the interfacial region.

The effects of these deleterious phases on mechanical and thermo-mechanical properties should also be studied.

The development of a mechanical test plan for PTA hardfacing deposits would require a clear definition of the scope of testing, since this is such an extensive field. Critical tests would include wear/abrasion testing, ideally at high temperature to reproduce the specific wear mechanisms present in service. An interesting test at this stage would be to determine whether both contacting surfaces would require hardfacing, or if adequate performance is possible with Co-based and Ni-based mated surfaces. High temperature wear can vary depending on the specific counterface material. Other tests could include adhesion, high temperature as-welded strength, and finally PTA hardfacing of the required geometry (Z-section).

As a final step before industrial implementation of PTA hardfacing with Stellite 694 and Tribaloy T-800 on IN738, an improved quality inspection process should be established. The current method of X-ray radiography requires detailed inspection by highly qualified staff and does not seem conclusive when it comes to identifying cracks caused by processing. A quality procedure may require in-process controls as well as end-process non-destructive testing. Subsequent Taguchi-based experimentation could prove helpful in this area.

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Appendix A: DSC Set-up Procedures

DSC Set-up Procedures

Before starting the procedure detailed below check that:

- Ar supply is on and at the correct pressure (at the tank)
- All equipment is turned on
- The pressure relief valve on the DSC machine is open

- 1) Open the DSC machine by pressing the safety button on the right-hand side and the "Up" arrow button on the left-front panel.

- 2) Ensure the sample and reference crucibles are clean. Use methanol and acetone if necessary.
- 3) Weigh the reference crucible and record the mass (before and after the run).
- 4) Weigh and record the mass of the reference material (if used).
- 5) Insert the reference crucible in the receptacle located on the far side of the DSC sample holder. Brace your hand against the green safety rail.

DSC Set-up Procedures

- 6) Weigh the sample crucible and sample and record the masses.
 - Ensure sample is clean (ultrasonic)
 - Ensure crucible is debris-free
- 7) Weigh the sample and crucible together and record the mass.
- 8) Insert the sample crucible in the near receptacle of the DSC sample holder. Brace your hand against the green safety rail.

- 9) Close the DSC machine by pressing the safety button on the right-hand side and the "Down" arrow button on the left-front panel.

DSC Set-up Procedures

- 10) Introduce protective gas by turning the dial on the right side of the flow meter to an indicated reading of 60 mL/min.

- 11) Introduce purge gas by pressing "purge 1" on the front panel (green LED indicates on) and adjusting the "purge 1" knob on the top of the DSC unit. Check the left side flow meter for a reading of 60 mL/min.

DSC Set-up Procedures

- 12) Close the pressure relief valve on top of the DSC furnace unit by turning 90° clockwise. Immediately stop the purge gas by pressing "purge 1" (LED extinguishes).

- 13) Turn on fore pump (mechanical vacuum pump), by pressing the green switch on the left front of the vacuum console, to evacuate the furnace. Vacuum pressure is indicated on the LCD and by a gauge at the rear of the DSC unit.

- 14) Turn on the turbo pump when the mechanical vacuum pump has reached a steady-state (4.5-5.0 indicated on LCD). Press the button on the right front of the vacuum console below a flashing green light (LED remains illuminated when pump is turned on).

DSC Set-up Procedures

- 15) At this time, set up the measurement program.
- 16) Turn off the turbo pump when the vacuum is no longer changing ($\sim 1.3E^{-4}$). Allow the pump to spin down.
- 17) When the indicated vacuum (LCD) starts to fall rapidly (numbers get larger):
 - Back-fill with purge gas ("purge 1" button and knob on top)
 - At equilibrium, turn off the vacuum pump
 - Pressure will rise just above positive (indicated on the dial gauge)
 - Open the pressure relief valve to a 45° angle (can hear a whistle of escaping gas)
 - Turn "purge 1" knob back to an indicated reading of 60 on the flow meter

- 18) In the measurement program, click START STANDBY. Heating is indicated on the DSC front panel by a green LED.
- 19) When the temperature in the DSC furnace reaches 100°C, click START MEASUREMENT to begin the heating cycle and data collection.
- 20) When the cycle is complete and the furnace has cooled to room temperature, shut off the purge and protective gases. At this time, the sample and reference crucibles may be removed.

Appendix B: Stellite 694 and Tribaloy T-800 XRD Results

Notes regarding the XRD spectra and tables of identified phases contained on the following pages:

- (1) In each XRD spectra figure, the top curve represents the XRD spectrum obtained along the grow-up direction of dendrites, while the lower curve is the spectrum through the cross-section.
- (2) The reliability for the presence of α -Co, gamma (Ni-base), gamma prime (Ni_3Al) phases is relatively high. However, it is difficult to separate their diffracted lines since their lattice constants are very close. SEM/EDS may provide further proofs.
- (3) It should be very cautious to claim the presence of other minor phases. The XRD itself cannot give solid conclusion at this point since the related diffracted lines were weak, broad, dispersed, and/or overlapped with major diffracted lines. SEM/EDS analysis could identify the presence of minor phases and their semi-qualitative chemical composition, which might support/eliminate the presence of some minor phases on the phase list.

50/50 Stellite 694

50/50 Tribaloy T-800

1. Specimen 694 (50/50 694 with IN738)

Phase	PDF#	1	2	3	4	5	6	7	8	9	10	11
α -Co	15-0806			(111)	(200)		(220)	(311)	(222)		(331)	(420)
Ni	04-0850			(111)	(200)		(220)	(311)	(222)		(331)	(420)
Ni ₃ Al	09-0097			(111)	(200)		(220)	(311)	(222)	(400)	(331)	(420)
Cr ₂₃ C ₆	35-0783		(422)	(511)	(531)		(822)					
Cr ₃ C ₇	11-0550			(202)(421)	(601)(402)							
α -CrCo	09-0052			(410)(330)	(222)		(532)					
WC	25-1047	(100)										
Co ₃ W ₉ C ₄	06-0616	(103)	(301)	(203)								
MoSi ₂	41-0612			(103)								

2. Specimen 800 (50/50 T-800 with IN738)

Phase	PDF#	1	2	3	4	5	6	7	8	9	10	12	14	15
α -Co	15-0806			(111)			(200)	(220)		(311)	(222)		(420)	(422)
Ni	04-0850			(111)			(200)	(220)		(311)	(222)		(420)	(422)
Ni ₃ Al	09-0097			(111)			(200)	(220)		(311)	(222)	(400)	(420)	(422)
Ni ₃ Ti	05-0723			(004)		(202)		(220)	(312)	(401)	(008)			
Cr ₂₃ C ₆	35-0783	(420)	(422)	(511)				(822)						
Cr ₃ C ₂	35-0804	(121)		(150)(131)	(240)	(211)	(310)							
Cr ₃ C ₇	11-0550	(321)		(202)(421)	(600)	(511)	(601)(402)	(503)(910)	(552)					
Co ₇ W ₆	02-1091	(110)	(1010)	(116)(021)		(024)	(119)	(0216)						
α -CrCo	09-0052	(311)		(410)(330)	(202)	(420)	(222)	(532)						
χ -Co ₃ W ₉ C ₄	06-0616		(301)	(203)		(302)								
Co ₃ Mo ₂ Si	30-0449	(110)	(103)		(112)	(201)			(220)	(215)	(313)			
Co ₃ Mo	29-0488			(002)		(201)				(222)				

Appendix C: DSC Output Plots

IN738 powder

Netzsch 404.C DSC

date:	30-May-03	initial weight:	36.5	mg	sample	Ref. crucible
run #:	8	final weight:	35.7	mg		ref initial 242.1 lid
sample:	IN738	purge gas:	Argon			ref final 242
condition:	powder	gas flowrate:	50	ml/min		sample crucible
1st vacuum:	1.20E-04 mbar	operator:	James Day			samp initial 244.5 lid
2nd vacuum:	- mbar					samp. Final 244.6
						(includes sample reesidue)

heating program: * note values in red must be entered

	start (°C)	setpoint (°C)	rate (°C/min)	duration (min)	cumulative (min)
room temp:	25	25	N/A	0	0
start	25	100	10	7.5	7.5
initial stdby:	100	100	isotherm	10	17.5
dynamic:	100	1500	20	70	87.5
hold:	1500	1500	isotherm	10	97.5
dynamic:	1500	400	20	55	152.5
emergency T:	1520

start standby:
start measurement:

Stellite 694 powder

Netzsch 404.C DSC

date:	22-May-03	sample	Stellite 694	Ref. crucible	
run #:	6	initial weight:	35.9 mg	ref initial	242.3 lid
sample:	Stellite 694	final weight:	36.6 mg	ref final	242.2
condition:	powder	purge gas:	Argon	sample crucible	
1st vacuum:	8.70E-05 mbar	gas flowrate:	50 ml/min	samp initial	240.4 lid
2nd vacuum:	- mbar	operator:	James Day	samp. Final	241
				(includes sample reesidue)	

heating program: * note values in red must be entered

	start (°C)	setpoint (°C)	rate (°C/min)	duration (min)	cumulative (min)
room temp:	25	25	N/A	0	0
start	25	100	10	7.5	7.5
initial stdby:	100	100	isotherm	10	17.5
dynamic:	100	1500	20	70	87.5
hold:	1500	1500	isotherm	10	97.5
dynamic:	1500	400	20	55	152.5
emergency T:	1520

start standby:
start measurement:

T-800 powder

Netzsch 404.C DSC

date:	25-Jun-03	sample	T-800		Ref. crucible
run #:	1	initial weight:	39	mg	ref initial
sample:	T-800	final weight:	36.4	mg	242 lid
condition:	powder	purge gas:	Argon		ref final
1st vacuum:	9.70E-05 mbar	gas flowrate:	50	ml/min	242.2
2nd vacuum:	- mbar	operator:	James Day		
					sample crucible
					samp initial
					245.9 lid
					samp. Final
					246.1
					(includes sample reesidue)

heating program: * note values in red must be entered

	start (°C)	setpoint (°C)	rate (°C/min)	duration (min)	cumulative (min)
room temp:	25	25	N/A	0	0
start	25	100	10	7.5	7.5
initial stdby:	100	100	isotherm	10	17.5
dynamic:	100	1500	20	70	87.5
hold:	1500	1500	isotherm	10	97.5
dynamic:	1500	400	20	55	152.5
emergency T:	1520

start standby:
start measurement:

log book ref.

file

95%IN738+5%Stellite 694 Powder

Netzsch 404.C DSC

date: 15-May-03
run #: 2
sample: 95IN738+5Stellite 694
condition: POWDER
1st vacuum: 5.50E-05 mbar
2nd vacuum: - mbar

Sample 95IN738+5Stellite 694
initial weight: 38.90 mg
final weight: 37.00 mg
Reference Al2O3
initial weight: 242.30 mg
final weight: 242.10 mg
purge gas: Argon
gas flowrate: 50 ml/min
operator: James Day

Sample Crucible
initial weight: 243.20 mg
final weight: 243.70 mg

heating program: * note values in red must be entered

	start (°C)	setpoint (°C)	rate (°C/min)	duration (min)	cumulative (min)
room temp:	25	25	N/A	0	0
start	25	100	10	7.5	7.5
initial stdby:	100	100	isotherm	10	17.5
dynamic:	100	1500	20	70	87.5
hold:	1500	1500	isotherm	10	97.5
dynamic:	1500	400	20	55	152.5
hold:	400	400	isotherm	5	157.5
dynamic:	400	1500	20	55	212.5
hold:	1500	1500	isotherm	10	222.5
dynamic:	1500	400	20	55	277.5
emergency T:	1520

start standby:

start measurement:

log book ref.

file

95%IN738+5%T-800 Powder

Netzsch 404.C DSC

date:	26-Jun-03	Sample	95IN738+5T-800	Sample Crucible	
run #:	2	initial weight:	36.70 mg	initial weight:	246.50 mg
sample:	95IN738+5T-800	final weight:	36.20 mg	final weight:	246.40 mg
condition:	POWDER	Reference	Al ₂ O ₃		
1st vacuum:	1.10E-04 mbar	initial weight:	242.20 mg		
2nd vacuum:	- mbar	final weight:	242.20 mg		
		purge gas:	Argon		
		gas flowrate:	50 ml/min		
		operator:	James Day		

heating program: * note values in red must be entered

	start (°C)	setpoint (°C)	rate (°C/min)	duration (min)	cumulative (min)
room temp:	25	25	N/A	0	0
start	25	100	10	7.5	7.5
initial stdby:	100	100	isotherm	10	17.5
dynamic:	100	1500	20	70	87.5
hold:	1500	1500	isotherm	10	97.5
dynamic:	1500	400	20	55	152.5
hold:	400	400	isotherm	5	157.5
dynamic:	400	1500	20	55	212.5
hold:	1500	1500	isotherm	10	222.5
dynamic:	1500	400	20	55	277.5
emergency T:	1520

start standby:

start measurement:

log book ref.

file

75%IN738+25%Stellite 694 Powder

Netzsch 404.C DSC

date:	23-May-03	Sample	75IN738+25Stellite 694	Sample Crucible	
run #:	7	initial weight:	39.00 mg	initial weight:	240.60 mg
sample:	75IN738+25Stellite 694	final weight:	37.40 mg	final weight:	241.60 mg
condition:	POWDER	Reference	Al2O3		
1st vacuum:	8.70E-05 mbar	initial weight:	242.30 mg		
2nd vacuum:	- mbar	final weight:	242.30 mg		
		purge gas:	Argon		
		gas flowrate:	50 ml/min		
		operator:	James Day		

heating program: * note values in red must be entered

	start (°C)	setpoint (°C)	rate (°C/min)	duration (min)	cumulative (min)
room temp:	25	25	N/A	0	0
start	25	100	10	7.5	7.5
initial stdby:	100	100	isotherm	10	17.5
dynamic:	100	1500	20	70	87.5
hold:	1500	1500	isotherm	10	97.5
dynamic:	1500	400	20	55	152.5
hold:	400	400	isotherm	5	157.5
dynamic:	400	1500	20	55	212.5
hold:	1500	1500	isotherm	10	222.5
dynamic:	1500	400	20	55	277.5
emergency T:	1520

start standby:

start measurement:

log book ref.

file

75%IN738+25%T-800 Powder

Netzsch 404.C DSC

date:	26-Jun-03	Sample	75IN738+25T-800	Sample Crucible	
run #:	3	initial weight:	37.20 mg	initial weight:	246.30 mg
sample:	75IN738+25T-800	final weight:	37.10 mg	final weight:	246.80 mg
condition:	POWDER	Reference	Al ₂ O ₃		
1st vacuum:	9.20E-05 mbar	initial weight:	242.20 mg		
2nd vacuum:	- mbar	final weight:	242.20 mg		
		purge gas:	Argon		
		gas flowrate:	50 ml/min		
		operator:	James Day		

heating program: * note values in red must be entered

	start (°C)	setpoint (°C)	rate (°C/min)	duration (min)	cumulative (min)
room temp:	25	25	N/A	0	0
start	25	100	10	7.5	7.5
initial stdby:	100	100	isotherm	10	17.5
dynamic:	100	1500	20	70	87.5
hold:	1500	1500	isotherm	10	97.5
dynamic:	1500	400	20	55	152.5
hold:	400	400	isotherm	5	157.5
dynamic:	400	1500	20	55	212.5
hold:	1500	1500	isotherm	10	222.5
dynamic:	1500	400	20	55	277.5
emergency T:	1520

start standby:

start measurement:

log book ref.

file

50%IN738+50%Stellite 694 Powder

Netzsch 404.C DSC

date:	20-May-03	Sample	50%IN738+50%Stellite 694	Sample Crucible	
run #:	4	initial weight:	33.90 mg	initial weight:	243.70 mg
sample:	50%IN738+50%Stellite 694	final weight:	32.30 mg	final weight:	244.50 mg
condition:	POWDER	Reference	Al ₂ O ₃		
1st vacuum:	6.30E-05 mbar	initial weight:	242.10 mg		
2nd vacuum:	- mbar	final weight:	242.20 mg		
		purge gas:	Argon		
		gas flowrate:	50 ml/min		
		operator:	James Day		

heating program: * note values in red must be entered

	start (°C)	setpoint (°C)	rate (°C/min)	duration (min)	cumulative (min)
room temp:	25	25	N/A	0	0
start	25	100	10	7.5	7.5
initial stdby:	100	100	isotherm	10	17.5
dynamic:	100	1500	20	70	87.5
hold:	1500	1500	isotherm	10	97.5
dynamic:	1500	400	20	55	152.5
hold:	400	400	isotherm	5	157.5
dynamic:	400	1500	20	55	212.5
hold:	1500	1500	isotherm	10	222.5
dynamic:	1500	400	20	55	277.5
emergency T:	1520

start standby:

start measurement:

log book ref.

file

50%IN738+50%T-800 Powder

Netzsch 404.C DSC

date: 27-Jun-03
run #: 4
sample: 50IN738+50T-800
condition: POWDER
1st vacuum: 1.00E-04 mbar
2nd vacuum: - mbar

Sample 50IN738+50T-800
initial weight: 36.80 mg
final weight: 36.60 mg
Reference Al2O3
initial weight: 242.20 mg
final weight: 242.10 mg
purge gas: Argon
gas flowrate: 50 ml/min
operator: James Day

Sample Crucible
initial weight: 246.40 mg
final weight: 246.90 mg

heating program: * note values in red must be entered

	start (°C)	setpoint (°C)	rate (°C/min)	duration (min)	cumulative (min)
room temp:	25	25	N/A	0	0
start	25	100	10	7.5	7.5
initial stdby:	100	100	isotherm	10	17.5
dynamic:	100	1500	20	70	87.5
hold:	1500	1500	isotherm	10	97.5
dynamic:	1500	400	20	55	152.5
hold:	400	400	isotherm	5	157.5
dynamic:	400	1500	20	55	212.5
hold:	1500	1500	isotherm	10	222.5
dynamic:	1500	400	20	55	277.5
emergency T:	1520

start standby:

start measurement:

log book ref.

file

25%IN738+75%Stellite 694 Powder

Netzsch 404.C DSC

date:	21-May-03	Sample	25%IN738+75%Stellite 694	Sample Crucible	
run #:	5	initial weight:	39.50 mg	initial weight:	244.20 mg
sample:	25%IN738+75%Stellite 694	final weight:	38.90 mg	final weight:	244.10 mg
condition:	POWDER	Reference	Al ₂ O ₃		
1st vacuum:	5.40E-05 mbar	initial weight:	242.20 mg		
2nd vacuum:	- mbar	final weight:	242.10 mg		
		purge gas:	Argon		
		gas flowrate:	50 ml/min		
		operator:	James Day		

heating program: * note values in red must be entered

	start (°C)	setpoint (°C)	rate (°C/min)	duration (min)	cumulative (min)
room temp:	25	25	N/A	0	0
start	25	100	10	7.5	7.5
initial stdby:	100	100	isotherm	10	17.5
dynamic:	100	1500	20	70	87.5
hold:	1500	1500	isotherm	10	97.5
dynamic:	1500	400	20	55	152.5
hold:	400	400	isotherm	5	157.5
dynamic:	400	1500	20	55	212.5
hold:	1500	1500	isotherm	10	222.5
dynamic:	1500	400	20	55	277.5
emergency T:	1520

start standby:

start measurement:

log book ref.

file

25%IN738+75%T-800 Powder

Netzsch 404.C DSC

date:	27-Jun-03	Sample	25IN738+75T-800	Sample Crucible	
run #:	5	initial weight:	37.40 mg	initial weight:	246.60 mg
sample:	25IN738+75T-800	final weight:	36.90 mg	final weight:	246.70 mg
condition:	POWDER	Reference	Al2O3		
1st vacuum:	8.60E-05 mbar	initial weight:	242.10 mg		
2nd vacuum:	- mbar	final weight:	242.10 mg		
		purge gas:	Argon		
		gas flowrate:	50 ml/min		
		operator:	James Day		

heating program: * note values in red must be entered

	start (°C)	setpoint (°C)	rate (°C/min)	duration (min)	cumulative (min)
room temp:	25	25	N/A	0	0
start	25	100	10	7.5	7.5
initial stdby:	100	100	isotherm	10	17.5
dynamic:	100	1500	20	70	87.5
hold:	1500	1500	isotherm	10	97.5
dynamic:	1500	400	20	55	152.5
hold:	400	400	isotherm	5	157.5
dynamic:	400	1500	20	55	212.5
hold:	1500	1500	isotherm	10	222.5
dynamic:	1500	400	20	55	277.5
emergency T:	1520

start standby:

start measurement:

Appendix D: PHACOMP Algorithm Implementation and Results

The PHACOMP algorithm was incorporated into an Excel spreadsheet requiring only the weight percentage of the alloy composition to be entered, a mean electron hole number for the particular alloy was then automatically calculated. The PHACOMP Calculator is documented below with portions of a sample calculation spreadsheet pasted in and descriptions for each numbered column (or set of columns).

1	2	3	4	5	6	7
Composition from Testing			Calculated			
			Entered			
		atomic mass				
Element	wt %	g/mol	mol	total mol	at %	
C	0.48	12.011	0.039963	1.687237	2.369	C
Si	0.5	28.09	0.0178	1.687237	1.055	Si
Mn	0.5	54.94	0.009101	1.687237	0.539	Mn
P	0	30.974	0	1.687237	0.000	P
S	0	32.064	0	1.687237	0.000	S
Cr	22	52	0.423077	1.687237	25.075	Cr
Ni	33	58.71	0.562085	1.687237	33.314	Ni
Mo	0.85	95.94	0.00886	1.687237	0.525	Mo
W	11.05	183.85	0.060103	1.687237	3.562	W
V	0.5	50.94	0.009815	1.687237	0.582	V
Co	24.525	58.93	0.416172	1.687237	24.666	Co
Cu	0	63.54	0	1.687237	0.000	Cu
Nb	0.45	92.91	0.004843	1.687237	0.287	Nb
Ta	0.85	180.95	0.004697	1.687237	0.278	Ta
Ti	1.7	47.9	0.035491	1.687237	2.103	Ti
Al	1.7	26.98	0.06301	1.687237	3.734	Al
Fe	1.5	55.85	0.026858	1.687237	1.592	Fe
B	0.055	10.81	0.005088	1.687237	0.302	B
Zr	0.025	91.22	0.000274	1.687237	0.016	Zr
1.687237						

Step 1: Convert alloy to atomic percent composition.

Column 1: Element labels
Column 2: Entered values of weight percent composition.
Column 3: Accepted atomic mass of each element from column 1.
Column 4: Molarity = wt% / atomic mass
Column 5: Total mols = Σ mols/element
Column 6: atomic % = (mol/total mol)*100
Column 7: Element labels

7	8	9	10	11	12	13
	TaC	NbC	ZrC	TiC	VC	remaining
C	1.1843	0.9059	0.6188	0.6026	0.0000	1.1843
Si						1.0550
Mn						0.5394
P						0.0000
S						0.0000
Cr						25.0751
Ni						33.3139
Mo						0.5251
W						3.5622
V					0.0000	0.5817
Co						24.6659
Cu						0.0000
Nb		0.2871				0.0000
Ta	0.2784					0.0000
Ti				0.6026		1.5009
Al						3.7345
Fe						1.5918
B						0.3016
Zr			0.0162			0.0000

Step 2: Calculate composition of secondary phases (numbers in brackets denote column number)

a) $\frac{1}{2}$ of carbon forms mono-carbides in a preferred order.

Column 7: Element labels

Columns 8-12: at% available for C and respective carbide formers (in preferential order)

Column 8: TaC

$$C(8) = (\text{total C}) / 2$$

Ta(8): IF(C(8) > Ta(6)), Ta(8) = Ta(6); ELSE, Ta(8) = C(8)

Column 9: NbC

$$C(9) = C(8) - \text{Ta}(8)$$

Nb(9): IF(C(9) > Nb(6)), Nb(9) = Nb(6); ELSE, Nb(9) = C(9)

Columns 10-12: Same as for column 9 for ZrC, TiC, and VC

Column 13: at% remaining after mono-carbide formation.

7	13	14	15
	remaining	Cr ₂₁ (Mo,W) ₂ C ₆	(NiCo ₂ Mo ₃)C
C	1.1843	0.0000	1.1843
Si	1.0550		
Mn	0.5394		
P	0.0000		
S	0.0000		
Cr	25.0751	0.0000	
Ni	33.3139		1.1843
Mo	0.5251	0.0000	0.5251
W	3.5622	0.0000	3.0278
V	0.5817		
Co	24.6659		2.3686
Cu	0.0000		
Nb	0.0000		
Ta	0.0000		
Ti	1.5009		
Al	3.7345		
Fe	1.5918		
B	0.3016		
Zr	0.0000		

- b) Remaining carbon forms an M₂₃C₆ carbide, either Cr₂₃C₆ or Cr₂₁(Mo, W)₂C₆; or M₆C carbide in the form of (NiCo₂Mo₃)C. The M₆C forms only when Mo + ½ W ≥ 6 wt% is satisfied.

Column 13: at% remaining after mono-carbide formation.

Column 14: Cr₂₁(Mo, W)₂C₆

C(14): IF(Mo(2) + W(2) ≥ 6), C(14) = 0; ELSE, C(14) = C(13)

Cr(14): IF(C(14) = 0), Cr(14) = 0; ELSE, Cr(14) = (C(13)/6)*21

Mo(14): IF(C(14) = 0), Mo(14) = 0; ELSE, Mo(14) = C(13)/6

W(14): IF(C(14) = 0), W(14) = 0; ELSE, W(14) = C(13)/6

Column 15: (NiCo₂Mo₃)C

C(15): IF(C(14) = 0), C(15) = C(13); ELSE, C(15) = 0

Ni(15): IF(C(15) = 0), Ni(15) = 0; ELSE, Ni(15) = C(15)

Mo(15): IF(C(15) = 0), Mo(15) = 0; ELSE, IF(3*C(15) > Mo(13)), Mo(13); ELSE, Mo(15) = 3*C(15)

W(15): IF(Mo(15) < 3*C(15)), W(15) = 3*C(15) - Mo(15); ELSE, W(15) = 0

Co(15): IF(C(15) = 0), Co(15) = 0; ELSE, Co(15) = 2*C(15)

7	16	17
	remaining	(MoTiCrNi) ₃ B ₂
C	0.0000	
Si	1.0550	
Mn	0.5394	
P	0.0000	
S	0.0000	
Cr	25.0751	0.1131
Ni	32.1296	0.0452
Mo	0.0000	0.0000
W	0.5345	0.2262
V	0.5817	
Co	22.2973	
Cu	0.0000	
Nb	0.0000	
Ta	0.0000	
Ti	1.5009	0.0678
Al	3.7345	
Fe	1.5918	
B	0.3016	0.3016
Zr	0.0000	

- c) Ni, Cr, Ti, Mo, form a boride as (Mo_{0.5}Ti_{0.15}Cr_{0.25}Ni_{0.10})₃B₂. An assumption is made that W will replace Mo if this element has been exhausted by previously formed phases.

Column 16: at% remaining after carbide formation

Column 17: (Mo_{0.5}Ti_{0.15}Cr_{0.25}Ni_{0.10})₃B₂

$$B(17) = B(16)$$

$$Cr(17) = (3/2) * B(17) * 0.25$$

$$Ni(17) = (3/2) * B(17) * 0.10$$

$$Mo(17): \text{IF}((3/2) * B(17) * 0.5 < Mo(16)), Mo(17) = (3/2) * B(17) * 0.5; \text{ELSE}, Mo(17) = Mo(16)$$

$$W(17): \text{IF}(Mo(17) < (3/2) * B(17) * 0.5), W(17) = (3/2) * B(17) * 0.5 - Mo(17); \text{ELSE}, W(17) = 0$$

$$Ti(17) = (3/2) * B(17) * 0.15$$

7	18	19
	remaining	Ni ₃ (Al,Ti,Nb,Cr)
C	0.0000	
Si	1.0550	
Mn	0.5394	
P	0.0000	
S	0.0000	
Cr	24.9621	0.7523
Ni	32.0844	17.7594
Mo	0.0000	
W	0.3083	
V	0.5817	
Co	22.2973	
Cu	0.0000	
Nb	0.0000	0.0000
Ta	0.0000	
Ti	1.4331	1.4331
Al	3.7345	3.7345
Fe	1.5918	
B	0.0000	
Zr	0.0000	
	TOTAL	5.9198

- d) The total amounts of Al, Ti, Nb, plus 3% of original Cr combine with 3X as much Ni to form Ni₃(Al, Ti, Nb, Cr) γ'.

Column 18: at% remaining after boride formation
Column 19: Cr(19) = 0.03*Cr(6)
Nb(19) = Nb(18)
Ti(19) = Ti(18)
Al(19) = Al(18)
TOTAL = ΣCr(19), Nb(19), Ti(19), Al(19)
Ni(19) = 3*TOTAL(19)

20	21		22	23	24
remaining	Austenite composition		m	Nv	Nbar=m*Nv
0.0000	0.0000	C			
1.0550	1.6253	Si			
0.5394	0.8310	Mn	0.00831	3.66	0.0304
0.0000	0.0000	P			
0.0000	0.0000	S			
24.2098	37.2984	Cr	0.372984	4.66	1.7381
14.3250	22.0696	Ni	0.220696	0.61	0.1346
0.0000	0.0000	Mo	0	4.66	0.0000
0.3083	0.4750	W	0.00475	4.66	0.0221
0.5817	0.8963	V			
22.2973	34.3520	Co	0.34352	1.71	0.5874
0.0000	0.0000	Cu			
0.0000	0.0000	Nb			
0.0000	0.0000	Ta			
0.0000	0.0000	Ti			
0.0000	0.0000	Al			
1.5918	2.4524	Fe	0.024524	2.22	0.0544
0.0000	0.0000	B			
0.0000	0.0000	Zr			
64.9084					
SUM					2.57

Step 3: Phase amounts from Step 2 are summed and subtracted from the original composition to give a remaining composition after all secondary phases have formed. The residual composition is scaled to 100 to give the austenite composition.

Column 20: at% remaining after all secondary phases have formed, a sum of this content (TOTAL(20)) is included at the bottom of the column.

Column 21: austenite% = 100*(value(20) / TOTAL(20))

Step 4: Calculate the mean electron hole number by summation

Column 22: atomic fraction for austenite composition

Column 23: given electron hole numbers for the elements

Column 24: mean electron hole numbers = $m \cdot N_v$

SUM is the mean electron hole number for the alloy (main output)

$$\tilde{N}_v = \sum m_i (N_v)_i$$

PHACOMP Results: